TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC

NORTHEAST SUPPLY ENHANCEMENT PROJECT

ENVIRONMENTAL REPORT

APPENDIX J. OFFSHORE ENVIRONMENTAL SAMPLING REPORT, ADDENDA AND ERRATA

NOVEMBER 2026

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TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC

NORTHEAST SUPPLY ENHANCEMENT PROJECT

ENVIRONMENTAL REPORT

J-1. OFFSHORE ENVIRONMENTAL SAMPLING REPORT

NOVEMBER 2026

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Fall/Winter 2016 Offshore Environmental Sampling Report for the Northeast Supply Enhancement Project New Jersey, New York

June 2017

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List of Abbreviations and Acronyms

BTEX	Benzene, Toluene, Ethylbenzene, Xylene
CFR	Code of Federal Regulations
COC	Chemicals of Concern
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethylene
DDT	dichlorodiphenyltrichloroethane
DO	dissolved oxygen
ER-L	Effects Range Low (ER-L)
ER-M	Effects Range Median (ER-M)
°F	degrees Fahrenheit
E & E	Ecology and Environment, Inc.
EPA	U.S. Environmental Protection Agency
ESC	environmental screening criteria
FERC	Federal Energy Regulatory Commission
H'	Shannon-Weaver's Diversity Index
HDD	horizontal directional drill
MDS	multidimensional scaling
µg/kg	micrograms per kilogram
mg/L	milligrams per liter
mm	millimeters
mS/cm	millliSiemens per centimeter
nm	nautical miles
NOAA	National Oceanographic and Atmospheric Administration
NPL	National Priorities List
NTU	Nephelometric Turbidity Units
NJDEP	New Jersey Department of Environmental Protection
NYCDEP	New York City Department of Environmental Protection

List of Abbreviations and Acronyms (cont.)

NYSDEC	New York State Department of Environmental Conservation
PAHs	polycyclic aromatic hydrocarbons
PCBs	polychlorinated biphenyls
Pg/g	picograms per gram
Project	Northeast Supply Enhancement Project
PSU	practical salinity units
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan
SGVs	sediment guidance values
SVOC	semi-volatile organic compound
TOC	total organic carbon
TOGS	Technical and Operational Guidance Series
Transco	Transcontinental Gas Pipe Line Company, LLC
TSS	total suspended solids
USACE	U.S. Army Corps of Engineers
USGS	U.S. Geological Survey
VOC	volatile organic compound

1 Introduction

1.1 Project Description

Transcontinental Gas Pipe Line Company, LLC (Transco), has filed an application with the Federal Energy Regulatory Commission (FERC) for a Certificate of Public Convenience and Necessity (Certificate) for the Northeast Supply Enhancement Project (Project) (Docket No. CP17-101-000). This Offshore Environmental Sampling Report was prepared to facilitate FERC's review and development of an environmental impact statement for the Project and to support the corresponding state-level evaluation of the Project.

The Project supports National Grid's long-term growth, reliability, and flexibility beginning in the 2019/2020 heating season. Transco is proposing to expand its existing interstate natural gas pipeline system in Pennsylvania and New Jersey and its existing offshore natural gas pipeline system in New Jersey and New York waters (see Figure A-1). The Project's 400,000 dekatherms per day capacity is fully subscribed by two entities of National Grid: Brooklyn Union Gas Company (d/b/a [doing business as] National Grid NY) and KeySpan Gas East Corporation (d/b/a National Grid), collectively referred to herein as "National Grid."

To provide the incremental capacity, Transco plans to expand portions of its system from the existing Compressor Station 195 in York County, Pennsylvania, to the Rockaway Transfer Point, in New York State waters. The Rockaway Transfer Point is the interconnection point between the Project and Transco's existing Rockaway Delivery Lateral.

The offshore portion of the Project includes a new 26-inch-diameter pipeline that will extend approximately 23.33 miles east-northeast across Raritan Bay and Lower New York Bay from the Borough of Sayreville shoreline in Middlesex County, New Jersey, to the Rockaway Transfer Point, located in the Atlantic Ocean approximately 3 miles seaward of the Rockaway Peninsula in Queens County, New York (see Figure A-2).

From the Rockaway Transfer Point, the Rockaway Delivery Lateral supplies gas to an existing onshore meter and regulating station located in Kings County, New York. Approximately 5.95 miles of the Raritan Bay Loop route cross New Jersey state waters, and approximately 17.38 miles cross New York state waters. The route passes offshore of Middlesex County and Monmouth County, New Jersey, and Richmond County and Queens County, New York. Approximately 0.16 mile of the route crosses onshore in Middlesex County, New Jersey.

The proposed Raritan Bay Loop route crosses a continuous expanse of open marine and estuarine waters, which comprise three major waterbodies—Raritan Bay, Lower New York Bay, and the Atlantic Ocean. The Project area is located within the Monmouth watershed management area and the Atlantic Ocean/Long Island Sound watershed, which drains most of the surrounding metropolitan areas and encompasses all marine waters in New York Harbor, Long Island Sound, Block Island Sound, Lower New York Bay/Raritan Bay, Sandy Hook Bay, and the waters that drain into them. The route crosses three federally authorized navigation channels, including the Ambrose Reach B (Ambrose Channel), the Chapel Hill North (Chapel Hill Channel), and the Raritan Bay West Reach (Raritan Bay Channel) (National Oceanic and Atmospheric Administration [NOAA] 2015). The Ambrose Channel was dredged within the last 10 years as part of the U.S. Army Corps of Engineers' (USACE) overall New York/New Jersey Harbor deepening project. Based on a Freedom of Information Act request submitted to the USACE, Transco determined the Chapel Hill Channel was last dredged in March 2012. Transco has not yet determined when the Raritan Bay Channel was last dredged.

1.2 Scope of Work

Transco conducted a field survey from October through December 2016 to evaluate the physical, chemical, and biological characteristics of sediment along the proposed Raritan Bay Loop route. The survey was conducted to support evaluation of the route for both engineering design and environmental review purposes. Physical properties of the water column were also measured at the sediment sampling sites. Transco contracted Ecology and Environment, Inc. (E & E) to support the environmental permitting effort for the Project. Therefore, E & E developed a sampling and analysis plan/quality assurance project plan (SAP/QAPP)prior to the offshore survey and submitted this plan to regulatory agencies for comment to ensure adequacy of the sediment data for agency review. The SAP/QAPP prepared for the offshore component of the Project is provided in Appendix C. Agency comments on initial versions of the SAP/QAPP are provided in Appendix D. Transco contracted Alpine Ocean (a Gardline company) to conduct the survey and arrange for sediment testing at onshore laboratories.

Figure A-3 shows the locations of the sites sampled along the proposed Raritan Bay Loop route during the 2016 offshore study and the locations of several sites sampled along an alternative route in Raritan Bay south of Staten Island. Transco added these sampling sites for an alternative route in response to comments from the New York State Department of Environmental Conservation (NYSDEC) on the SAP/QAPP.

1.3 Previous Investigations

Several previous investigations were completed within Raritan Bay, the Lower New York Bay, and/or the Atlantic Ocean near the Rockaway Peninsula as part of other projects to identify the physical characteristics of the sediments and the degree to which contaminants may exist (Adams and Benyl 2003; Adams et al.

1998; E & E 2003). In New York waters, water quality in the Raritan and Lower New York Bays has been monitored by the New York City Department of Environmental Protection (NYCDEP) since 1977 (NYCDEP 2012). Water quality in Raritan Bay in New Jersey has been monitored by NJDEP, and monitoring data is available going back to 1989 (NJDEP 2003a, 2003b, and 2003c).

Based on data collected by the U.S. Geological Survey (USGS) and available in USGS Open File Report OFR 03-241, the predominant sediment type in this area appears to be fine-to-coarse sands (Mecray et al. 2003). Greater silt and clay content was generally observed near the mouth of the Raritan River, in the entrance to Upper New York Bay, and in Sandy Hook Bay. However, sediment sample data obtained in 2002 as part of the Neptune Regional Transmission System project indicates that material primarily classified as silt may also extend throughout Raritan Bay (E & E 2003).

The sediment data obtained in 2002 for the Neptune Regional Transmission System project indicated a number of potential chemical contaminants were present in Raritan Bay and the Lower New York Bay (E & E 2003). Inorganic contaminants (i.e., metals) such as mercury, zinc, and arsenic; organic contaminants such as polychlorinated biphenyls (PCBs); and polycyclic aromatic hydrocarbons (PAHs) were identified in the sampling data. These sampling results are generally consistent with earlier sediment testing (Adams and Benyl 2003; Adams et al. 1998), but there was a wide variation in results, depending upon sampling location and depth.

Sediment testing was performed in association with the Raritan Bay Slag site along the shore and in Raritan Bay in Old Bridge Township and Sayreville, New Jersey, as part of the U.S. Environmental Protection Agency's (EPA) Remedial Investigation (NJDEP 2016). The Raritan Bay Slag site is located approximately 0.5 miles south of the proposed Raritan Bay Loop route. The site is included on the New Jersey Department of Environmental Protection's (NJDEP's) Known Contaminated Site list (NJDEP 2016) and is on the EPA National Priorities List (NPL) (EPA ID NJN000206276) (EPA 2016). The Project route crosses designated investigation Areas 7 and 11 (Jetty Sector) of the NPL site. Testing results for EPA sampling locations at the seaward end of Area 7, near the offshore Project route, indicated that elevated levels of some contaminants were present, but lead levels at the offshore sample locations were approximately one to two orders of magnitude lower than levels measured at the jetty or along the shore (CDM 2011).

The NJDEP Division of Water Monitoring and Standards, Bureau of Marine Water Monitoring, monitors water quality in Raritan Bay, generally collecting water samples near the surface of the water column (NJDEP 2003a, 2003b, and 2003c). Data on properties such as temperature, transparency, dissolved oxygen (DO), and fecal coliform were collected between 1989 through 2007 at monitoring sites that are close to the Project route (i.e., Stations 26A, 66, and 918). New York City, which has been responsible for monitoring the quality of offshore waters since

1977, currently monitors the Lower New York Bay and Raritan Bay for several water quality parameters (NYCDEP 2012). The sample results of the most recent "State of the Harbor" report, published in 2012, indicated that water quality has improved overall based on the concentrations of DO, bacterial cell counts, water clarity, chlorophyll α, and nitrogen and total suspended solids in wastewater treatment plant discharge (NYCDEP 2012).

More recent investigations conducted by E & E as part of the Rockaway Delivery Lateral project indicated that substrate immediately seaward of the Rockaway Peninsula was predominantly sandy and had minimal contaminant levels (E & E 2011). The 2009/2010 Rockaway Delivery Lateral sample results were predominantly below the Class A thresholds identified in the NYSDEC Technical and Operational Guidance Series (TOGS) 5.1.9 document, *In-Water and Riparian Management of Sediment and Dredged Material* (NYSDEC 2004), and none exceeded the Class C thresholds (definitions provided in Section 2).

1.4 Fall/Winter 2016 Survey Results

This report presents the results of sample collection and analyses supporting the sediment chemistry, benthic community, and water quality evaluations for the Project, including:

- Sediment chemical contaminants;
- Sediment grain size;
- Physical water quality parameters; and
- Benthic infauna characteristics.

Field data collected as part of the Fall/Winter 2016 survey are included in the appendices. Appendix A presents figures referenced in the body of this report; Appendix B presents tables that are referenced in the body of this report; Appendix C presents the SAP/QAPP developed in 2016 for the data collection effort; Appendix D presents comments received from agencies on the initial versions of the SAP/QAPP; Appendix E presents the tabularized laboratory results for all chemical parameters analyzed and grain size statistics; Appendix F presents the sediment chemistry laboratory reports; Appendix G presents the water quality results for all parameters analyzed; Appendix H presents the Benthic Laboratory Report that discusses the results of the benthic sampling performed at each sample location; and Appendix I presents the Data Usability Summary Reports.

Although sediment samples were analyzed extensively for geotechnical properties as part of the 2016 survey, only a summary of grain size analyses are included in this report. Complete sediment grab sample grain size distribution curves are provided in Appendix 1D to Resource Report 1 (General Project Description) of Transco's application for a FERC Certificate. Geotechnical (deep) core logs are provided as Appendix B to Resource Report 6 (Soils) of Transco's application for a FERC Certificate. Transco submitted these resource reports to FERC on March

27, 2017. Transco anticipates submitting geotechnical (shallow) core logs and complete grain size distribution curves on the FERC Docket by July 2017.

Transco is conducting additional shallow core (vibracore) sampling along the proposed offshore route and will submit the associated core logs following the completion of the laboratory analyses. Based on the results of these analyses, Transco will propose appropriate sediment handling and disposal methods during construction, in coordination with the applicable agencies.

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2 Sediment Sampling

The SAP/QAPP developed to evaluate the site-specific sediment conditions along the proposed pipeline route was designed specifically to address chemicals of concern (COCs) identified by NYSDEC and the NJDEP. These chemicals would be of concern if found above threshold levels. The threshold values utilized reflect where there could be harmful effects to aquatic life.

2.1 Thresholds for Chemicals of Concern

Thresholds for COCs in sediments sampled in New York State waters were taken from the TOGS 5.1.9 document, *In-Water and Riparian Management of Sediment and Dredged Material* (NYSDEC 2004) and the *Screening and Assessment of Contaminated Sediment* (NYSDEC 2014). Thresholds for COCs in sediments sampled in New Jersey waters were taken from *The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters* (NJDEP 1997). Additional guidance on COCs within the offshore Project area was taken from the USACE/EPA's *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal* (2016). These agency documents were developed to provide guidance on the statutory and regulatory requirements for dredging activities and to promote uniformity in the certification and/or permitting of dredging projects.

In New York, in TOGS 5.1.9, the NYSDEC Division of Water established three classes of sediment quality thresholds for dredged material (sediment guidance values [SGVs]). This system is based upon the concentrations of contaminants within the dredged sediments, in contrast to the EPA's system, which is based upon bioaccumulation and biotoxicity. The three classes of dredged sediment provided in TOGS 5.1.9 are summarized here as follows:

- Class A – No appreciable contamination, no toxicity to aquatic life. Sediment chemistry is at or below the chemical concentrations that define this class. Dredging and in-water placement at approved locations can generally proceed.
- Class B – Moderate contamination, chronic toxicity to aquatic life. Several restrictions on dredging, applied based upon site-specific concerns and knowledge along with evaluation of sediment.
- Class C – High contamination, acute toxicity to aquatic life. This material may have stringent requirements for dredging and disposal. If contaminant levels exceed Class C, it is the responsibility of the applicant to ensure the material is not regulated as hazardous, as defined in 6 New York Code of Rules

and Regulations (NYCRR) Part 371. TOGS 5.1.9 does not cover hazardous materials/waste.

The discussion of sediment sampling results in Section 2.3 evaluates sediment contamination in terms of Class A and Class C thresholds only. If it is stated that a sediment sample exceeded the Class A threshold, it is understood that the sediment is a Class B. If the sediment sample exceeds the Class C threshold, it is understood that the sediment has exceeded both Class A and Class B thresholds and is considered a Class C sediment.

In New Jersey, the NJDEP's Site Remediation Program (SRP), Environmental Toxicology and Risk Assessment (ETRA) Unit, developed ecological screening criteria (ESC) for surface water, sediment, and soil. These ESCs were developed by the ERTA Unit using multiple sources and the most conservative criterion for each contaminant was used. The ESC are presented in terms of Effects Range Low (ER-L) and Effects Range Median (ER-M) levels for saline waters. The sediment and soil standards presented are not promulgated, and are intended as screening values for ecological assessments.

The EPA's Marine Protection, Research, and Sanctuaries Act provides the criteria for disposal of dredged sediments in the ocean. Under 40 Code of Federal Regulations (CFR) 227, Criteria for the Evaluation of Permit Applications for Ocean Dumping of Materials, there are three conditions that qualify dredged materials for ocean dumping without further testing. Two of these conditions are applicable to dredging materials proposed for placement at offshore sites, i.e., the historic area remediation site (HARS):

- Proposed dredged material is composed of predominantly sand, gravel, rock or any other naturally occurring bottom material with particle size larger than silt. Proposed dredge material is found in areas with high current or wave energy.
- Proposed dumping material is substantially the same as the substrate at the proposed dumping site, prior to dumping activities. Proposed dredging site is far removed from known and existing historical pollution sources, providing reasonable assurance that dredged material is not contaminated.

For materials that do not meet the above two conditions, and require further testing, 40 CFR 227 prohibits the ocean dumping of dredged materials containing anything greater than trace amounts of organohalogen compounds; mercury and mercury compounds; cadmium and cadmium compounds; oil of any kind or in any form; or known carcinogens, mutagens, or teratogens or any materials suspected of being such. Materials not excluded by this condition are subject to toxicity and bioaccumulation testing and must meet the conditions determined by the USACE/EPA guidance document. Based on the required testing and review by USACE/EPA, material may approved for disposal at the HARS. A HARS plan must be reviewed and approved for this activity. This option is under considera-

tion based on results from the offshore survey for management of dredged material during construction.

2.2 Sampling Methods

Sediment was sampled by core boring, vibracoring, and surface grab sample collection at a total of 96 sites. The depths at each sample site reflect the maximum anticipated depth of disturbance during Project construction (see Table B-1), accounting for anticipated pipeline burial depths based upon burial guidelines for the Project provided by the USACE New York District as of June 2016 (Handell 2016).

Deep core samples (core boring) extending up to 125 feet below the seafloor were collected from 9 sample sites, all of which are within New Jersey waters. These deep core sites are located near the New Jersey shoreline and across Ambrose Channel where Transco proposes to install the pipeline using horizontal directional drill (HDD). The deep core depths represent the maximum depths for HDD installation. Deep core samples were analyzed for geotechnical properties only to be used in the engineering design of the HDDs and so are not further discussed in this report.

Shallow core samples (vibracores) were collected at 87 sites, 15 of which are in New Jersey waters and 72 of which are in New York waters. Of the 87, 69 sample sites were centered along the proposed route and the remainder, were centered along a route alternative (see Figure A-3 for the location of all shallow core sample sites) developed at the request of NYSDEC. The shallow core sample sites are identified as "VC" in Table B-1, and which were reviewed and approved by the New Jersey State Historic Preservation Office or the New York State Historic Preservation Office, as appropriate prior to the sampling. Sites identified with "-ALT" indicate those sample sites that were shifted from the original designated site to avoid potential cultural resources.

The shallow cores extended at least 12 feet below the seafloor outside navigation channels and up to 30 feet below the seafloor near navigation channels and designated anchorage areas. The shallow core samples were collected in areas where the pipeline would be installed by trenching methods (e.g., a clamshell dredge, a mechanical plough, and/or a jet sled) and where HDD entry/exit pits would be excavated using a clamshell dredge. Dredged material may be side-cast adjacent to the trench and, following pipeline installation, would be used to backfill the trench to the extent practicable. Shallow core samples were collected for testing and analysis of both geotechnical and bulk sediment chemistry properties. Duplicate shallow core samples were collected at three sample sites located within the proposed HDD pits (see Table B-1 for these sample sites). (Note: The geotechnical analysis of the shallow core samples is not addressed in this report.)

Once retrieved, the sediment core soil types were classified according to the United Soil Classification System, and sediment samples were collected from the core and shipped to a laboratory for chemical analysis. Each core was separated into

3-foot sections for shallow core samples. For shallow cores, distinct layers more than 1-foot thick were separated as unique sub-samples subject to sediment chemistry analyses if they appeared contaminated, based on the field geologist's evaluation of appearance and/or odor. The minimum 1-foot thickness provided sufficient sample volume for all sediment chemistry analyses. A total of 398 samples were collected, as identified in Table B-2. The tests performed, method, and quantities of samples collected at sample sites in New Jersey and New York waters are summarized in Table B-3, and a complete list of chemical analyses performed and the laboratory standard operating procedures are provided in Appendix B. The list of chemical analyses performed is based upon the NYSDEC TOGS 5.1.9 and the NJDEP Site Remediation Program ER-L and Effects Range Median ER-M ecological screening criteria. A summary of the results are discussed below, and the complete analytical results are provided in Appendix E.

Surface sediment grab samples were collected for analysis of benthic invertebrate populations and supplemental physical (grain size and total organic carbon [TOC]) analyses. The results of the analysis of benthic invertebrate populations are addressed in Section 4. Grab samples were collected separately from the vibracore samples and/or at a sufficient distance from the specific vibracore sample locations to ensure that the surface samples were undisturbed before collection.

Quality assurance/quality control (QA/QC) samples were collected in accordance with the SAP/QAPP (Appendix C). Analytical data have been validated for samples collected as part of this sampling effort. Field QC samples included one sediment duplicate per matrix per 20 samples for each analysis (excluding sediment samples where analysis for dioxins and furans would not occur[1]). Trip blanks were collected to establish that the transport of sample containers to and from the field did not result in sample contamination from external sources. One trip blank was included in each cooler containing samples for volatiles analysis. Field equipment blanks were used at a rate of one per equipment set per day. Equipment blanks were only used on equipment sets that were subject to decontamination. Dedicated or disposable equipment did not require the use of equipment blanks.

The results of the bulk chemistry analysis of vibracore samples, and a summary of grain size analysis for vibracore and benthic grab samples are presented below.

2.3 Sediment Chemistry Results

Table B-3 presents a selection of analytes detected throughout the study corridor and the number of times both the SGVs and ESC were exceeded. Table B-3 also presents the range of concentrations detected at sample sites in each state that exceeded the state ESCs or SGVs. Table B-4 presents the number of analyte group exceedances of thresholds from both New Jersey and New York at each sample site.

[1] Only the upper (0- to 3-foot) sampling interval from sample sites in New York where clamshell dredging will not occur were analyzed for dioxins and furans.

Figures A-4, A-6, A-8, A-10, A-11, A-13, and A-14 show the number of exceedances at each sample site along the preferred route for each group of analytes based upon the lowest available threshold of either New Jersey or New York. Figures A-5, A-7, A-9, A-12, and A-15 show the number of exceedances at each depth sampled along the preferred route between 0 ft and 18 ft for each sample site by analyte group based upon the lowest available threshold of either New Jersey or New York. Figures A-16, A-17, A-18, A-19, and A-20 show the number of exceedances at each depth sampled along the alternative route between 0 ft and 18 ft for each sample site by analyte group based upon the lowest available threshold of either New Jersey or New York. Based on the limits of analysis and the lack of detections in samples, profile figures were not included for PCB Congeners and semi-volatile organic compound (SVOCs) (excluding PAHs).

In the following discussion, sample sites within the offshore study area were organized into four groups based upon the location of the proposed route (Table 2-1). Sample sites located within the alternative route comprise a fifth sampling group discussed in this draft report (Table 2-1).

The methods used to calculate the concentrations of the following analytes discussed below are presented in the SAP/QAPP (see Appendix C): sum of DDD (dichlorodiphenyldichloroethane) + DDE (dichlorodiphenyldichloroethylene) + DDT (dichlorodiphenyltrichloroethane), total PCB (Polycholrinated Biphenyls) Aroclors, total PCB congeners, total xylenes, total BTEX (Benzene, Toluene, Ethylbenzene, Xylene), total PAHs (Polycyclic Aromatic Hydrocarbons), and the total toxicity equivalency factor for dioxins and furans.

Table 2-1 Geographic Sediment Sample Groups

Group ID	Included Sample(s)	State
A	VC1-ALT to VC4	NJ
B	VC5 to VC44	NY
C	VC45-ALT to VC55	NJ
D	VC56 to VC69-ALT	NY
ALT	VC70 to VC87	NY

2.3.1 Group A – New Jersey

Metals

Each sediment sample was analyzed for 27 metals, including mercury (see Appendix C for a complete list of these metals). Of these metals, 16 have available ER-L and/or ER-M thresholds. Within this sample segment, all of the 27 metals tested for were detected in at least one sample in Group A. Table 2-2 lists the sample sites where metals were detected. Fourteen of the detected metals exceeded available NJDEP thresholds. The metals detected within this segment that exceeded ER-L and/or ER-M thresholds are presented in Table B-5.

Table 2-2 Number of Detections at Sample Sites in Group A

	Number of Detections							
Sample Site	VOCs	PAHs	Other SVOCs	Dioxins/ Furans	Pesticides	PCB Congener	PCB Aroclor	Metals
VC1-ALT[1]	5	17	13	17	7	30	2	27
VC1B-ALT[1]	1	17	11	15	6	30	3	27
VC2	0	0	0	2	0	10	0	23
VC3-ALT	0	0	0	2	0	5	0	25
VC4	0	2	3	2	0	11	0	26

Key:
[1] Samples labeled VC1-ALT and VC1B-ALT were collected in the same location.

Cyanide

Samples from all sample sites in New Jersey were tested for cyanide. Cyanide was measured as total and amenable concentrations. No standard or guidance value is available for total or amenable cyanide. Total cyanide was detected within the 0- to 3-foot and 3- to 6-foot intervals at locations VC1-ALT and VC1-ALT-B at concentrations ranging from 0.37 to 1.5 milligrams per kilogram (mg/kg). Cyanide was not detected in any samples collected from VC2, VC3-ALT, and VC4.

Pesticides

Each sediment sample was analyzed for 27 pesticides (see Appendix C for a complete list of these pesticides). A total of seven pesticides (Dieldrin, O,P'-DDD, O,P'-DDE, O,P'-DDT, P,P'-DDD, P,P'-DDE, and P,P'-DDT) were detected in at least one sample in Group A. (Table 2-2 lists the sample sites where pesticides were detected.) Eleven pesticides tested for have available ER-L and/or ER-M thresholds and four of these (including the sum of three pesticides [DDD + DDE + DDT) exceeded only the ER-L thresholds.

P,P'-DDD exceeded the ER-L threshold at the 0- to 3-foot and 3- to 6-foot depth intervals at VC1-ALT and VC1-ALT-B and at the 6- to 9-foot depth interval at VC1-ALT-B, with concentrations ranging from 4.48 to 14.7 micrograms per kilogram (µg/kg). P,P'-DDE exceeded the ER-L threshold at the 0- to 3-foot and 3- to 6-foot depth intervals at VC1-ALT and VC1-ALT-B and at the 6- to 9-foot depth interval at VC1-ALT-B with concentrations ranging from 5.5 to 56.5 µg/kg. P,P'-DDT exceeded the ER-L threshold at the 0- to 3-foot and 3- to 6 foot depth intervals at VC1-ALT and VC1-ALT-B with concentrations ranging from 2.39 to 5.37 µg/kg.

The sum of DDD + DDE + DDT exceeded the ER-L threshold value within the 0-3 ft and 3-6 ft depth intervals at VC1-ALT and VC1-ALT-B and the 6-9 ft depth interval at VC1-ALT-B with concentrations ranging from 14.21 to 92.57 µg/kg.

There were no pesticide detections in samples from VC2, VC3-ALT, and VC4.

Polychlorinated Biphenyls (PCBs)

Each sediment sample was analyzed for PCBs expressed as seven Aroclor compounds. Testing for 31 PCB congeners was also performed for samples from all sample sites in New Jersey. A complete list of the Aroclor compounds and PCB congeners included in the analysis is provided in Appendix C. Two PCB Aroclors, Aroclor 1254 and Aroclor 1260, and all 31 PCB congeners were detected in at least one sample in Group A. (Table 2-2 lists the sample sites where PCBs were detected.) ER-L thresholds are available for four Aroclor compounds and for the sum of Aroclor compounds. An ER-M threshold is also available for the sum of Aroclor compounds. While there are no available thresholds for individual PCB congeners, there are ER-L and ER-M thresholds for total PCB congeners. Of these, two Aroclor compounds, the sum of Aroclor compounds, and total PCB congeners exceeded ER-L and/or ER-M thresholds.

Aroclor 1254 and Aroclor 1260 exceeded the ER-L in the following samples: 0- to 3 feet and 3- to 6 feet at VC1-ALT; and 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC1B-ALT. The concentrations of Aroclor 1254 that exceeded the ER-L threshold ranged from 47.6 µg/kg to 714.0 µg/kg. The concentrations of Aroclor 1254 that exceeded the ER-L threshold ranged from 149 µg/kg to 340 µg/kg. The sum of Aroclors exceeded the ER-M threshold in the following samples: 0- to 3 feet and 3- to 6 feet at VC1-ALT; and 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC1B-ALT. The concentrations of the sum of Aroclors that exceeded the ER-M threshold ranged from 291.6 µg/kg to 1,140.0 µg/kg.

The sum of PCB congeners exceeded the ER-L threshold in the 6- to 9 feet sample at sample site VC1B-ALT, with a concentration of 44,100 picograms per gram (pg/g). The sum of PCB congeners also exceeded the ER-M threshold in the 0-3 ft and 3-6 ft samples at sample sites VC1-ALT and VC1B-ALT. Concentrations that exceeded the ER-M threshold ranged from 318,000 pg/g to 1,600,000 pg/g.

The sum of Aroclors and the sum of PCB congeners did not exceed the ER-L threshold in any samples from sample sites VC2, VC3-ALT, or VC4.

Volatile Organic Compounds (VOCs)

Each sediment sample was analyzed for the BTEX analytes, including all three isomers of xylene (m-, p-, and o-) and total xylenes. Benzene, ethylbenzene, o-xylene, and toluene were detected in at least one sample in Group A at sample sites VC1-ALT and VC1B-ALT. Only benzene, ethylbenzene, total xylene, and toluene have available ER-L thresholds. No VOCs have available ER-M thresholds. No VOCs were detected in exceedance of ER-L thresholds. No VOCs were detected at samples sites VC2, VC3-ALT, and VC4.

Polycyclic Aromatic Hydrocarbons (PAHs)

Each sediment sample was analyzed for 18 PAH compounds. Of the samples tested, 17 PAHs were detected (see Table 2-3 for a list of detected PAHs and Table 2-2 for the sample sites where they were detected). Seventeen PAHs and total

PAHs have available ER-L and/or ER-M thresholds. The PAHs that exceeded the ER-L and/or ER-M thresholds are presented in Table B-6.

No PAHs were detected at sample sites VC2 and VC3-ALT.

Table 2-3 PAHs Detected in Sample Group A

PAH	Range (µg/kg)	PAH	Range (µg/kg)
2-Methylnaphthalene	7.83-144	Chrysene	4.87-600
Acenaphthene	9.21-61.2	Dibenz(A,H)Anthracene	7.75-116
Acenaphthylene	8.02-83.1	Fluoranthene	10.2-903
Anthracene	16.1-156	Fluorene	8.11-138
Benzo(A)Anthracene	6.46-643	Indeno(1,2,3-C,D)Pyrene	55.1-385
Benzo(A)Pyrene	39.1-567	Naphthalene	4.51-174
Benzo(B)Fluoranthene	7.92-717	Phenanthrene	6.7-510
Benzo(G,H,I)Perylene	34-477	Pyrene	11-1110
Benzo(K)Fluoranthene	19.1-209		

Semi-Volatile Organic Compounds (SVOCs)

An additional 46 SVOCs were tested in samples from all sample sites in New Jersey. (See Appendix C for a complete list of these SVOCs.) Of those tested for, 14 SVOCs were detected. (see Table 2.4 for a list of detected SVOCs and Table 2.2 for the sample sites where they were detected.) Sixteen SVOCs have available ER-L and/or ER-M thresholds. Four of the SVOCs were detected at concentrations that exceeded the ER-L and/or ER-M thresholds.

Table 2-4 SVOCs Detected in Sample Group A

SVOC	Range (µg/kg)	SVOC	Range (µg/kg)
1,2,4-Trichlorobenzene	5.23-45.9	Benzyl Butyl Phthalate	39.1-117
1,2-Dichlorobenzene	21.5-21.5	Bis(2-Ethylhexyl) Phthalate	34.6-37300
1,3-Dichlorobenzene	15.2-33.6	Carbazole	3.06-46.9
1,4-Dichlorobenzene	58.9-84.9	Dibenzofuan	5.32-64
2,4-Dimethylphenol	29.5-60.6	Di-N-Butyl Phthalate	32.7-32.7
2-Chlorophenol	5.62-5.62	Di-N-Octylphthalate	124-124
4-Methylphenol	24.8-56.1	Phenol	8.72-16.9

1,2,4-Trichlorobenzene exceeded the ER-M threshold in the following samples: 0- to 3 feet and 3- to 6 feet at VC1-ALT; and 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC1B-ALT. The concentrations that exceeded the ER-M threshold ranged from 5.23 µg/kg to 45.9 µg/kg.

At 21.5 µg/kg, 1,2-dichlorobenzene exceeded the ER-M threshold in the 3- to 6-feet sample from sample site VC1-ALT.

Benzyl butyl phthalate exceeded the ER-M threshold in the following samples: 0- to 3 feet at VC1-ALT and 3- to 6 feet at VC1B-ALT. The concentrations that exceeded the ER-M threshold were 117 µg/kg and 69.3 µg/kg, respectively.

Bis(2-ethylhexyl) phthalate exceeded the ER-L threshold in the 3- to 6- feet samples from sample sites VC1-ALT and VC1B-ALT. The concentrations that exceeded the ER-L threshold were 2,300 µg/kg and 2,360 µg/kg, respectively. Bis(2-ethylhexyl) phthalate also exceeded the ER-M threshold in the 0- to 3-foot samples from sample sites VC1-ALT and VC1B-ALT. The concentrations that exceeded the ER-M threshold were 18,300 µg/kg and 37,300 µg/kg, respectively.

No SVOCs were detected at sample sites VC2 and VC3-ALT.

Dioxins/Furans

Sediment samples from each sample site were analyzed for dioxins and furans. (See Appendix C for a complete list of these dioxins and furans.) All dioxins and furans were detected in at least one sample from Group A (see Table 2.2 for the sample sites where they were detected). Of the dioxins and furans tested only the total toxicity equivalence factor of all dioxins and furans has an available ER-M.

For these sample sites, the total toxicity equivalency factor exceeded the ER-M threshold in the following samples: the 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet depth intervals at VC1-ALT and VC1B-ALT; the 12- to 15 foot depth interval at VC1-ALT; the 6-to 9 foot depth interval at VC2; and the 0-to 3 feet, 3- to 6 feet, and 6- to 9 feet depth intervals at VC4. Concentrations in these samples ranged from 3.81 pg/g to 81.5 pg/g.

2.3.2 Group B – New York

Metals

Each sediment sample was analyzed for 27 metals, including mercury (see Appendix C for a complete list of these metals). Each of the 27 metals was detected in at least one sample in Group B (see Table B-7 for the sample sites where they were detected; due to the number of sample sites in Group B, this table is included in Appendix B). Seven of the detected metals exceeded Class A thresholds. Of these metals, nine have available Class A and/or Class C thresholds and all nine of these metals exceeded available NYSDEC thresholds. The metals detected within this segment that exceeded Class A and/or Class C thresholds are presented in Table B-8.

Cyanide

Samples from sample sites in New York where clamshell dredging may occur were tested for cyanide (see Table B-1 for these locations). This decision to expand the analysis of samples from these sites was based upon the anticipated need

for further testing to determine if these sediments were HARS compatible. Cyanide was measured as total and amenable concentrations. No standard or guidance value is available for total or amenable cyanide.

Total cyanide was detected in the following samples: 0- to 3 feet at VC7 and 9- to 12 feet at VC16 and VC17; 3- to 6 feet at VC3; and 3- to 6 feet and 9- to 12 feet at VC36. Detected concentrations ranged from 0.25 mg/kg to 0.97 mg/kg.

Pesticides

Each sediment sample was analyzed for 11 pesticides (see Appendix C for a complete list of these pesticides). Sample sites in New York where clamshell dredging may occur were sampled for more analytes than required by NYSDEC. See above for discussion of analytes included in the analysis of samples at clamshell sites. An additional 16 pesticides were analyzed for in samples from sample sites in New York where clamshell dredging may occur (see Table B-1 for these locations).

Non-Clamshell Dredge Sample Sites

Of the 11 pesticides tested for in samples from sample sites where clamshell dredging would not occur, Class A and Class C thresholds are available for three pesticides and the sum of DDT+DDE+DDD. No pesticides tested for were detected in samples from these sample sites.

Possible Clamshell Dredge Sample Sites

Of the pesticides tested for in samples from sample sites where clamshell dredging may occur, 12 have available Class A and/or Class C thresholds. A total of 8 pesticides (aldrin, endosulfan sulfate, endrin, O,P'-DDD, O,P'-DDE, P,P'-DDD, P,P'-DDE, and P,P'-DDT) were detected in at least one sample in Group B. (See Table B-7 for a list of the sample sites where pesticides were detected.) Of the detected pesticides, only endrin and the sum of DDT + DDE + DDD have available Class A and Class C thresholds, and only the sum of DDT + DDE + DDD exceeded the Class A threshold. The sum of DDT + DDE + DDD exceeded the Class A threshold in the 0- to 3-foot samples at VC6 and VC7; all samples from VC16 and VC17; and 0- to 3 foot sample at VC41-ALT. Concentrations that exceeded the Class A threshold ranged from 8.6 µg/kg to 99.0 µg/kg.

Polychlorinated Biphenyls (PCBs)

Each sediment sample was analyzed for PCBs expressed as seven Aroclor compounds. Samples from sample sites in New York where clamshell dredging may occur were tested for 31 PCB congeners (see Table B-1 for these locations). See above for discussion of analytes included in the analysis of samples at clamshell sites. A complete list of the Aroclor compounds and PCB congeners included in the analysis is provided in Appendix C. Class A and Class C thresholds are available for the sum of Aroclor compounds and total PCB congeners.

Non-Clamshell Dredge Sample Sites

No PCB Aroclors were detected in samples from sample sites where clamshell dredging would not occur.

Possible Clamshell Dredge Sample Sites

Two Aroclor compounds and all 31 PCB congeners were detected in at least one sample in Group B (see Table B-7 for the sample sites where they were detected). Class A thresholds were exceeded for both the sum of Aroclor compounds and total PCB congeners. The Class C threshold for total PCB congeners was also exceeded.

PCBs, expressed as the sum of Aroclor compounds, were detected in concentrations that exceeded the Class A threshold in the 0- to 3 feet samples at VC6 and VC7 and all samples from VC16 and VC17. Concentrations that exceeded the Class A threshold ranged from 161.6 µg/kg to 945.5 µg/kg .

PCBs expressed as the sum of congeners exceeded the Class A threshold in the following samples: 0- to 3 feet at VC6 and VC7; 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, and 12- to 15 feet at VC16; and 0- to 3 feet, 3- to 6 feet, 6- to 12 feet, and 12- to 15 feet at VC17. The concentrations that exceeded the Class A threshold ranged from 149,000 pg/g to 727,000 pg/g. PCBs expressed as the sum of congeners also exceeded the Class C threshold in the 9- to 12-foot sample from VC16 with a concentration of 2,000,000 pg/g.

Volatile Organic Compounds (VOCs)

Each sediment sample was analyzed for the BTEX analytes, including all three isomers of xylene (m-, p-, and o-) and total xylenes. Benzene, ethylbenzene, total xylene, toluene, and total BTEX have available Class A and Class C thresholds. All VOC analytes were detected in at least one sample within Group B. (See Table B-7 for the sample sites where they were detected.) No detected VOCs exceeded the Class A criteria.

Polycyclic Aromatic Hydrocarbons (PAHs)

Each sediment sample was analyzed for 18 PAH compounds. All 18 PAHs were detected in at least one sample in Group B(see Table B-7 for the sample sites where PAHs were detected). Only total PAHs has available Class A and Class C thresholds.

Total PAHs detected only exceed the Class A criteria in the following samples: 0-3 ft, 3- to 6 feet, 6- to 9 feet, 9-to 12 feet at VC16 and VC17; and 0- to 3feet at VC37 and VC38. The concentrations that exceeded the Class A threshold ranged from 4,540.0 µg/kg to 21,860.5 µg/kg.

Semi-Volatile Organic Compounds (SVOCs)

An additional 46 SVOCs were tested in samples from sample sites in New York where clamshell dredging may occur (see Table B-1 for these locations). See above for discussion of analytes included in the analysis of samples at clamshell sites. See Appendix C for a complete list of these SVOCs. Of those tested for, 11 SVOCs were detected (see Table 2-5 for a list of detected SVOCs and Table B-7 for the sample sites where SVOCs were detected). Class A and/or Class C thresholds are available for six SVOCs. None exceeded Class A thresholds.

Table 2-5 SVOCs Detected in Sample Group B

SVOC	Range (µg/kg)	SVOC	Range (µg/kg)
1,2,4-Trichlorobenzene	2.68-32.8	Dibenzofuan	7.34-85.4
1,4-Dichlorobenzene	48.4-48.4	Diethyl Phthalate	2.52-13.6
4-Methylphenol	17.2-108	Di-N-Butyl Phthalate	7.81-93.1
Benzyl Butyl Phthalate	23.9-386	Di-N-Octylphthalate	21.8-109
Bis(2-Ethylhexyl) Phthalate	19.1-18900	Phenol	6.81-17.7
Carbazole	3.63-39.8		

Dioxins/Furans

Sediment samples from each sample site were analyzed for dioxins and furans, but only the upper (0- to 3-foot) sampling interval was tested for samples from sample sites in New York where clamshell dredging will not occur (see Appendix C for a complete list of these dioxins and furans). See above for discussion of analytes included in the analysis of samples at clamshell sites. All dioxins and furans were detected in at least one sample in Group B (see Table B-7 for the sample sites where dioxins/furans were detected). Of the dioxins and furans tested for, Class A and Class C thresholds for only one dioxin (2,3,7,8-tetrachlorodibenzo-P-Dioxin) and the total toxicity equivalence factor of all dioxins and furans are available.

Non-Clamshell Dredge Sample Sites

There were no exceedances detected in samples analyzed from sample sites where clamshell dredging would not occur.

Possible Clamshell Dredge Sample Sites

The total toxicity equivalency factor only exceeded the Class A threshold in samples from sites where clamshell dredging may occur. 2,3,7,8-tetrachlorodibenzo-P-Dioxin did not exceed the Class A threshold. The samples where the total toxicity equivalency factor exceeded the Class A threshold were 3- to 6 feet at VC6; 3- to 6 feet at VC7; 9- to12 feet at VC8; 0- to 3 feet and 6- to 9 feet at VC15; 0- to 3 feet and 6- to 9 feet at VC18; and 3- to 6 feet at VC39. The concentrations that exceeded the Class A threshold ranged from 4.6 pg/g to 8.01 pg/g.

2.3.3 Group C – New Jersey

Metals

Each sediment sample was analyzed for 27 metals, including mercury (see Appendix C for a complete list of these metals). Of these metals, 16 ER-L and/or ER-M thresholds are available. Within this sample segment, 25 metals tested for were detected in at least one sample in Group C (see Table 2-6 for the sample sites where metals were detected). The only two metals not detected were antimony and molybdenum. Three of the metals detected exceeded available NJDEP thresholds.

Table 2-6 Number of Detections at Sample Sites in Group C

Sample Site	Number of Detections							
	VOCs	PAHs	Other SVOCs	Dioxins/ Furans	Pesticides	PCB Congener	PCB Aroclor	Metals
VC45-ALT	0	11	0	1	0	2	0	21
VC46	0	17	2	1	0	0	0	23
VC47	0	17	2	1	0	0	0	23
VC48-ALT	0	3	1	1	0	18	0	17
VC49	1	12	1	3	0	15	0	20
VC50	0	0	0	1	0	1	0	21
VC51	0	0	1	1	0	0	0	22
VC52	0	0	0	1	0	0	0	22
VC53	0	0	1	1	0	2	0	20
VC54[1]	0	17	5	15	0	3	0	24
VC54B[1]	0	17	5	3	0	1	0	24
VC55[1]	0	2	0	1	0	3	0	21
VC55B[1]	0	13	0	1	0	2	0	20

Key:
[1] - Samples labeled "VC#" and "VC#B" were collected in the same location.

Nickel exceeded only the ER-L threshold at the 9- to 12-foot sample at sample site VC47, with a concentration of 23 mg/kg.

Selenium exceeded the ER-M threshold in the following samples: 0- to 3 feet at VC46; 9- to 12 feet at VC47; 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, 9- to 12 feet at VC50, 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, 9- to 12 feet at VC51; 3- to 6 feet, and 6- to 9 feet at VC52; 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, 9- to 12 feet at VC53; 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, 9- to 12 feet, 12- to 15 feet, and 15- to 17.63 feet at VC54; 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, and 9- to 12 feet at VC54B; and 9- to 12 feet at VC55. Concentrations that exceeded the ER-M threshold ranged from 1.00 mg/kg to 4.13 mg/kg.

Mercury exceeded only the ER-L threshold in the following samples: 0- to 3 feet at VC47; 3- to 6 feet and 6- to 9 feet at VC54 and VC54-B, with concentrations ranged from 0.195 to 0.506 mg/kg.

Manganese exceed the ER-M threshold in the following samples: 0- to 3 feet and 3- to 6 feet at VC48-ALT and 0- to 3 feet at VC49. Concentrations that exceeded the ER-M threshold ranged from 422 mg/kg to 724 mg/kg.

Cyanide

Samples from all sample sites in New Jersey were tested for cyanide. Cyanide was measured as total and amenable concentrations. There are no standard or guidance values available for total or amenable cyanide. Total cyanide was detected in three of the sample sites. Detections occurred at the 6- to 9.7-foot interval at location VC48-ALT, the 9- to 12-foot interval at location VC51, and the 3-

to 6-foot and 6- to 99 foot intervals at location VC55 with concentrations ranging from 0.21 to 0.62 mg/kg.

Pesticides

Each sediment sample was analyzed for 27 pesticides (see Appendix C for a complete list of these pesticides). ER-L and/or ER-M thresholds were available for 11 pesticides. Pesticides were not detected in any sediment samples.

Polychlorinated Biphenyls (PCBs)

Each sediment sample was analyzed for PCBs expressed as seven Aroclor compounds. Testing for 22 PCB congeners was also performed for samples from all sample sites in New Jersey. A complete list of the Aroclor compounds and PCB congeners included in the analysis is provided in Appendix C. ER-L thresholds are available for four Aroclor compounds and for the sum of Aroclor compounds, and an ER-M threshold is also available for the sum of Aroclor compounds. While there are no available thresholds for individual PCB congeners, there are ER-L and ER-M thresholds for total PCB congeners.

PCBs, expressed as the sum of Aroclor compounds, were not detected in any sediment samples collected from these sample sites. Eighteen PCB congeners were detected in samples from these samples sites (see Table 2-7 for a list of these PCB congeners and Table 2-6 for the sample sites where they were detected). PCBs expressed as the sum of congeners were detected at locations VC45-ALT, VC48-ALT, VC49, VC50, VC53, VC54-B, VC54, VC55, and VC55-B and did not exceed the ER-L thresholds.

Table 2-7 PCB Congeners Detected in Sample Group C

PCB Congener	Range (µg/kg)	PCB Congener	Range (µg/kg)
PCB-8	14.2-19.4	PCB-52	6.2-42
PCB-170	8.52-24.7	PCB-66	7.42-73.1
PCB-193/180	20.7-74.1	PCB-69/49	30.6-46.1
PCB-183/185	29.7-29.7	PCB-77	7.97-12.9
PCB-187	13.8-67.8	PCB-105	8.45-14.9
PCB-138/163/129	31.4-127	PCB-113/90/101	30.4-54.3
PCB-153/168	32.1-134	PCB-118	34.8-53.8
PCB-20/28	67.4-84.4	PCB-206	7.78-12.1
PCB-44/65/47	39.5-60.3	PCB-209	8.34-25.1

Volatile Organic Compounds (VOCs)

Each sediment sample was analyzed for the BTEX analytes, including all three isomers of xylene (m-, p-, and o-) and total xylenes. Only benzene, ethylbenzene, total xylene, and toluene have available ER-L thresholds. ER-M thresholds are not available for VOCs.

Benzene was detected in only one sample at VC-49. This single detection was below the ER-L threshold. No other VOC detections occurred.

Polycyclic Aromatic Hydrocarbons (PAHs)

Each sediment sample was analyzed for 18 PAH compounds and 17 PAHs were detected (see Table 2-8 for a list of detected SVOCs and Table 2-6 for the sample sites where they were detected). The total toxicity equivalency factor exceeded the ER-M threshold in the 6-to 9 foot sample from sample site VC54 with a concentration of 5.83 pg/g.

Table 2-8 PAHs Detected in Sample Group C

PAH	Range (µg/kg)	PAH	Range (µg/kg)
2-Methylnaphthalene	5.25-41.5	Chrysene	3.57-1160
Acenaphthene	4.18-172	Dibenz(A,H)Anthracene	5.68-211
Acenaphthylene	4.41-131	Fluoranthene	3.24-2490
Anthracene	4.02-989	Fluorene	3.2-263
Benzo(A)Anthracene	3.75-1670	Indeno(1,2,3-C,D)Pyrene	3.76-753
Benzo(A)Pyrene	4.85-1330	Naphthalene	3.15-125
Benzo(B)Fluoranthene	7.37-1450	Phenanthrene	4.44-1570
Benzo(G,H,I)Perylene	4.59-708	Pyrene	3.09-2270
Benzo(K)Fluoranthene	3.29-488		

Semi-Volatile Organic Compounds (SVOCs)

An additional 46 SVOCs were tested in samples from all sample sites in New Jersey (see Appendix C for a complete list of these SVOCs). Five of the 46 SVOCs were detected (4-chloroaniline, 4-methylphenol, benzyl butyl phthalate, dibenzofuan, and phenol). ER-L and/or ER-M thresholds for 16 SVOCs are available. No SVOCs exceeded the ER-L and/or ER-M thresholds.

Dioxins/Furans

Sediment samples from each sample site were analyzed for dioxins and furans (see Appendix C for a complete list of these dioxins and furans). Of these, only the total toxicity equivalence factor of all dioxins and furans has an available ER-M. Dioxins and furans that were detected are listed in Table 2-9 See Table 2-6 for the sample sites where they were detected.

The total toxicity equivalency factor exceeded the ER-M threshold in the 6-to 9 foot sample from sample site VC54 with a concentration of 5.83 pg/g.

Table 2-9 Dioxins and Furans Detected in Sample Group C

SVOC	Range (µg/kg)	SVOC	Range (µg/kg)
1,2,3,4,6,7,8-Heptachlorodibenzofuan	22.6-22.6	1,2,3,7,8,9-Hexachlorodibenzo-P-Dioxin	3.07-3.07
1,2,3,4,6,7,8-Heptachlorodibenzo-P-Dioxin	1.78-14.7	1,2,3,7,8-Pentachlorodibenzofuan	1.69-1.69
1,2,3,4,7,8,9-Heptachlorodibenzofuan	3.72-3.72	2,3,4,6,7,8-Hexachlorodibenzofuan	5.9-5.9
1,2,3,4,7,8-Hexachlorodibenzofuan	5.52-5.52	2,3,4,7,8-Pentachlorodibenzofuan	3.02-3.02
1,2,3,4,7,8-Hexachlorodibenzo-P-Dioxin	1.98-1.98	2,3,7,8-Tetrachlorodibenzofuan	0.349-0.654
1,2,3,6,7,8-Hexachlorodibenzofuan	5.49-5.49	Octachlorodibenzofuan	11-11
1,2,3,6,7,8-Hexachlorodibenzo-P-Dioxin	1.95-1.95	Octachlorodibenzo-P-Dioxin	2.92-171
1,2,3,7,8,9-Hexachlorodibenzofuan	2.22-2.22		

2.3.4 Group D – New York

Metals

Each sediment sample was analyzed for 27 metals, including mercury (see Appendix C for a complete list of these metals). Twenty-six of the 27 metals were detected in at least one sample in Group D (see Table 2-10 for the sample sites where metals were detected). The only metal not detected was antimony. Of these metals, nine have available Class A and/or Class C thresholds and sampling results for four of these metals exceeded available NYSDEC thresholds.

Arsenic exceeded only the Class A threshold in the 0- to 3-foot sample at sample site VC60 and the 3- to 5-foot sample site at VC62. The concentrations that exceeded the Class A threshold were 8.27mg/kg and 10.3 mg/kg, respectively.

Copper exceeded only the Class A threshold in the 0- to 3-foot sample at sample site VC60 with a concentration of 43.1 mg/kg.

Lead exceeded only the Class A threshold in the 0- to 3-foot sample at sample site VC60 and the 3- to 5-foot sample at sample site VC62. The concentrations that exceeded the Class A threshold were 86.3 mg/kg and 77.6 mg/kg, respectively.

Mercury exceeded the Class A threshold in the following samples: 0- to 3 feet at VC60 and VC61-ALT; 3- to 6 feet at VC64; and 3- to 4.8 feet at VC65. Concentrations that exceeded the Class A threshold ranged from 0.169 mg/kg to 0.354 mg/kg. Mercury also exceeded the Class C threshold in the 6- to 6.1-foot sample from sample site VC64 with a concentration of 4.55 mg/kg.

Table 2-10 Number of Detections at Sample Sites in Group D

Sample Site[1]	Number of Detections							
	VOCs	PAHs	Other SVOCs	Dioxins/ Furans	Pesticides	PCB Congener	PCB Aroclor	Metals
VC56	0	0	0	1	0	0	0	17
VC57	1	0	0	0	0	0	0	22
VC58	0	1	0	1	0	0	0	22
VC59	0	3	0	0	0	0	0	19

Table 2-10 Number of Detections at Sample Sites in Group D

Sample Site[1]	Number of Detections							
	VOCs	PAHs	Other SVOCs	Dioxins/ Furans	Pesticides	PCB Congener	PCB Aroclor	Metals
VC60	0	17	0	1	0	0	0	24
VC61-ALT	0	17	0	1	0	0	0	24
VC62	0	18	0	1	0	0	0	22
VC63	0	18	0	5	6	0	2	24
VC64	0	17	0	3	0	0	0	22
VC65	0	17	0	1	0	0	0	22
VC66	0	10	0	1	0	0	0	20
VC67	0	2	0	1	0	0	0	20
VC68	0	0	0	0	0	0	0	19
VC69-ALT	1	1	0	1	0	0	0	20

Key:
[1] Dioxins and furans were analyzed only in the samples taken from 0- to 3 feet at sites in New York where clamshell dredging is not anticipated to occur. Dioxin/furan samples for additional depths at these sites were archived. PCB congeners were not tested at sites in New York where clamshell dredging is not anticipated to occur.

Cyanide

Samples from sampling sites in New York where clamshell dredging may occur were tested for cyanide (see Table B-1 for these locations). See above for discussion of analytes included in the analysis of samples at clamshell sites. However, because sample sites in Group D are not located in areas of planned clamshell dredging, no sample sites within these locations were tested for cyanide.

Pesticides

Each sediment sample was analyzed for 11 pesticides (see Appendix C for a complete list of these pesticides). Samples from sample sites in New York where clamshell dredging may occur were analyzed for an additional 16 pesticides (see Table B-1 for these locations). See above for discussion of analytes included in the analysis of samples at clamshell sites.

Class A and Class C thresholds and the sum of DDT+DDE+DDD were available for 3 of the 11 pesticides. A total of six pesticides (O,P'-DDD, O,P'-DDE, P,P'-DDD, P,P'-DDE, P,P'-DDT, and trans-chlordane) were detected in at least one sample in Group D. Pesticides were only detected at sample site VC63.

A standard or guidance value is not available for any of the detected pesticides. The sum of DDT + DDE + DDD in the 0- to 3 foot sample at sample site VC63 did not exceed the Class A threshold. The concentration of the sum of DDT + DDE + DDD detected was 3.56 µg/kg.

No sample sites in Group D were at locations where clamshell dredging might occur.

Polychlorinated Biphenyls (PCBs)

Each sediment sample was analyzed for PCBs expressed as seven Aroclor compounds. Samples from sample sites in New York where clamshell dredging may occur were tested for 31 PCB congeners (see Table B-1 for these locations). See above for discussion of analytes included in the analysis of samples at clamshell sites. A complete list of the Aroclor compounds and PCB congeners included in the analysis is provided in Appendix C. Class A and Class C thresholds are available for the sum of Aroclor compounds and total PCB congeners.

Non-Clamshell Dredge Sample Sites

Two Aroclor compounds, Aroclor 1254 and 1260, were detected in at least one sample from Group D, from sample site VC63. Neither Aroclor compound exceeded the Class A threshold. The sum of PCB Aroclors did not exceed the Class A threshold in any samples from these sample sites.

No sample sites in Group D were at locations where clamshell dredging might occur.

Volatile Organic Compounds (VOCs)

Each sediment sample was analyzed for the BTEX analytes, including all three isomers of xylene (m-, p-, and o-) and total xylenes. Class A and Class C thresholds are available for benzene, ethylbenzene, total xylene, toluene, and total BTEX. One VOC (toluene) was detected in samples from these sample sites (see Table 2-10 for the sample sites where it was detected). Concentrations of toluene (0.14 µg/kg and 0.16 µg/kg) did not exceed the Class A criteria.

Polycyclic Aromatic Hydrocarbons (PAHs)

Each sediment sample was analyzed for 18 PAH compounds. All 18 PAHs were detected in at least one sample in Group D (see Table 2-10 for the sample sites where they were detected. Class A and Class C thresholds are available only for total PAHs.

Total PAHs exceeded only the Class A threshold in the following samples: 3- to 5 feet at VC62; 6- to 7 feet at VC63; 3- to 6 feet, and 6- to 6.1 foot at VC64. Concentrations that exceeded the Class A threshold ranged from 4,813 µg/kg to 18,500 µg/kg.

Semi-Volatile Organic Compounds (SVOCs)

An additional 46 SVOCs were tested in samples from sample sites in New York where clamshell dredging may occur (see Table B-1 for these locations). See above for discussion of analytes included in the analysis of samples at clamshell sites. See Appendix C for a complete list of these SVOCs. No sample sites in Group D were at locations where clamshell dredging might occur.

Dioxins/Furans

Sediment samples from each sample site were analyzed for dioxins and furans, but only the upper (0- to 3-foot) sampling interval was tested for samples from sample sites in New York where clamshell dredging will not occur. See above for

discussion of analytes included in the analysis of samples at clamshell sites. See Appendix C for a complete list of these dioxins and furans. Of the dioxins and furans tested, only one dioxin (2,3,7,8-tetrachlorodibenzo-p-dioxin) and the total toxicity equivalence factor of all dioxins and furans have available Class A and Class C thresholds.

Three dioxins (1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin, 2,3,7,8-tetrachlorodibenzo-p-dioxin, and octachlorodibenzo-p-dioxin) and two furans (1,2,3,4,6,7,8-heptachlorodibenzofuan and 2,3,7,8-tetrachlorodibenzofuan) were detected in at least one sample in Group D (see Table 2.10 for the sample sites where they were detected). No detected dioxins or furans exceeded the Class A thresholds. The total toxicity equivalency factor did not exceed the Class A threshold.

No sample sites in Group D were at locations where clamshell dredging might occur.

2.3.5 Alternative Route

Metals

Each sediment sample was analyzed for 27 metals, including mercury (see Appendix C for a complete list of these metals). Each of the 27 metals was detected in at least one sample in the alternative route (see Table 2-11 for the sample sites where metals were detected). Class A and/or Class C thresholds for nine of these metals are available, and all nine exceeded the thresholds. The metals detected within this segment that exceeded Class A and/or Class C thresholds are presented in Table B-10.

Table 2-11 Number of Detections at Sample Sites Along Alternative Route

	Number of Detections							
Sample Site[1]	VOCs	PAHs	Other SVOCs	Dioxins/ Furans	Pesticides	PCB Congener	PCB Aroclor	Metals
VC70	0	17	0	17	5	0	2	27
VC71	0	8	0	2	0	0	0	26
VC72	0	12	0	3	0	0	0	23
VC73-ALT	0	16	0	2	4	0	3	24
VC74	0	14	0	2	0	0	1	22
VC75	0	17	0	2	1	0	0	26
VC76	5	17	0	7	5	0	3	26
VC77	0	17	1	7	4	29	3	26
VC78	0	17	5	14	2	28	0	27
VC79-ALT	0	16	2	15	2	27	0	26
VC80	0	16	0	16	11	30	2	27
VC81	0	17	0	17	5	31	2	26
VC82	0	17	0	2	0	0	1	25
VC83-ALT	0	0	0	2	0	0	0	24
VC84-ALT	0	0	0	2	0	0	0	24

Table 2-11 Number of Detections at Sample Sites Along Alternative Route

	Number of Detections							
Sample Site[1]	VOCs	PAHs	Other SVOCs	Dioxins/ Furans	Pesticides	PCB Congener	PCB Aroclor	Metals
VC85	0	11	0	2	0	0	0	24
VC86-ALT	0	11	0	1	0	0	0	24
VC87	0	2	0	2	0	0	0	24

Key:

[1] Dioxins and furans were analyzed only in the samples taken from 0- to 3 feet at sites in New York where clamshell dredging is not anticipated to occur. Dioxin/furan samples for additional depths at these sites were archived. PCB congeners were not tested at sites in New York where clamshell dredging is not anticipated to occur.

Cyanide

Samples from sampling sites in New York where clamshell dredging may occur were tested for cyanide (see Table B-1 for these locations). See above for discussion of analytes included in the analysis of samples at clamshell sites. Cyanide was measured as total and amenable concentrations. No standard or guidance values are available for total or amenable cyanide. Total cyanide was detected in three of the sample sites. Total cyanide was detected in the following samples: 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC80; and 0- to 3 feet, 6-to 9 feet, 9- to 12 feet, and 12- to 14.75 feet at VC81. Concentrations in these samples ranged from 0.38 mg/kg to 0.66 mg/kg.

Pesticides

Each sediment sample was analyzed for 11 pesticides (see Appendix C for a complete list of these pesticides). In samples from sites in New York where clamshell dredging may occur an additional 16 pesticides were analyzed (see Table B-1 for these locations). See above for discussion of analytes included in the analysis of samples at clamshell sites.

Non-Clamshell Dredge Sample Sites

In samples from sample sites where clamshell dredging would not occur three of the 11 pesticides and the sum of DDT+DDE+DDD have available Class A and Class C thresholds. Five pesticides were detected (O,P'-DDD, O,P'-DDE, P,P'-DDD, P,P'-DDE, and P,P'-DDT) in at least one sample in the alternative route, none of which have available Class A thresholds (see Table 2.11 for the locations where these pesticides were detected). The sum of DDT+DDE+DDD exceeded only the Class A threshold in the 0- to 3-foot sample at sample site VC70 with a concentration of 52.2 µg/kg.

Possible Clamshell Dredge Sample Sites

Twelve off the 27 pesticides analyzed were detected in at least one sample in the alternative route (see Table 2-12 for the list of detected pesticides and Table 2-11 for the sample sites where pesticides were detected. Of these 12 pesticides, one (Mirex) has an available Class A threshold, which was not exceeded. The Class A threshold of the sum of DDT+DDE+DDD was exceeded in the 6- to 9-foot sample at sample site VC80 and the 9- to 12-foot sample at sample site VC81.

Concentrations that exceeded the Class A threshold were 63.1 µg/kg and 63.0 µg/kg, respectively.

Table 2-12 Pesticides Detected in the Alternative Route at Possible Clamshell Sites

Pesticide	Range (µg/kg)	Pesticide	Range (µg/kg)
Alpha BHC	0.742-0.967	P,P'-DDD	1.27-11.1
Endosulfan Sulfate	1.92-1.92	P,P'-DDE	0.399-32.3
Endrin Aldehyde	4.19-4.19	P,P'-DDT	0.574-1.96
Mirex	0.866-0.866	Trans-Chlordane	1.07-1.53
O,P'-DDD	0.719-21.4	Trans-Nonachlor	0.849-1.24
O,P'-DDE	0.388-15.8		

Polychlorinated Biphenyls (PCBs)

Each sediment sample was analyzed for PCBs expressed as seven Aroclor compounds. Samples from sample sites in New York where clamshell dredging may occur were tested for 31 PCB congeners (see Table B-1 for these locations). See above for discussion of analytes included in the analysis of samples at clamshell sites. A complete list of the Aroclor compounds and PCB congeners included in the analysis is provided in Appendix C.

Non-Clamshell Dredge Sample Sites

Three Aroclor compounds (Aroclor 1016, Aroclor 1254, and Aroclor 1260) were detected in at least one sample from the alternative route (see Table 2-11 for the sample sites where PCBs were detected). Class A thresholds were exceeded only for the sum of Aroclor compounds in the following samples: 0- to 3 feet at VC70 and VC76 and 9- to 12 feet at VC73 and VC74. Concentrations that exceeded the Class A threshold ranged from 300 µg/kg to 554 µg/kg.

Possible Clamshell Dredge Sample Sites

Three Aroclor compounds (Aroclor 1016, Aroclor 1254, and Aroclor 1260) and all 31 PCB congeners were detected in at least one sample in the alternative route (see Table 2.11 for the sample sites where they were detected). Class A thresholds were exceeded for both the sum of Aroclor compounds and total PCB congeners. The Class C threshold for total PCB congeners was also exceeded.

PCBs, expressed as the sum of Aroclor compounds, were detected in concentrations that exceeded the Class A threshold in the following samples: 0- to 3 feet at VC77; 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC80, and all samples from VC81. Concentrations that exceeded the Class A threshold ranged from 108.5 µg/kg to 725.5 µg/kg.

The total of PCB congeners exceeded the Class A threshold in the following samples: 0- to 3 feet at VC77, VC78, and VC79-ALT; 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC80 and VC81. Concentrations that exceeded the Class A threshold ranged from 112,000 pg/g to 652,000 pg/g. Total PCB congeners also exceeded

the Class C threshold in the 9- to 12-foot sample at sample site VC81, with a concentration of 1,200,000 pg/g.

Volatile Organic Compounds (VOCs)

Each sediment sample was analyzed for the BTEX analytes, including all three isomers of xylene (m-, p-, and o-) and total xylenes. Class A and Class C thresholds are available for benzene, ethylbenzene, total xylene, toluene, and total BTEX. Four VOCs (benzene, ethylbenzene, m,p-xylene, and toluene) were detected in at least one sample in the alternative route. VOCs were only detected at sample site VC76 and none of the detected VOCs, total xylenes, nor total BTEX exceeded Class A thresholds.

Polycyclic Aromatic Hydrocarbons (PAHs)

Each sediment sample was analyzed for 18 PAH compounds. All 18 PAHs were detected in at least one sample in the alternative route (see Table 2-11 for the sample sites where PAHs were detected). Class A and Class C thresholds are available only for total PAHs.

Total PAHs exceeded only the Class A threshold in the following samples: 0- to 3 feet at VC70, VC76, VC77, and VC78; 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC80; and 0- to 3 feet, 6- to 9 feet, and 9- to 12 feet at VC81. Concentrations that exceeded the Class A threshold ranged from 4,020 µg/kg to 11,600 µg/kg.

Semi-Volatile Organic Compounds (SVOCs)

Samples from sample sites in New York where clamshell dredging may occur (see Table B-1 for these locations) were tested for an additional 46 SVOCs. See above for discussion of analytes included in the analysis of samples at clamshell sites. See Appendix C for a complete list of these SVOCs. Six SVOCs have available Class A and/or Class C thresholds. Of those samples tested, five SVOCs were detected (4-methylphenol, bis(2-ethylhexyl) phthalate, carbazole, hexachlorocyclopentadiene, and phenol) in at least one sample in the alternative route (see Table 2-11 for the sample sites where SVOCs were detected). None exceeded Class A thresholds.

Dioxins/Furans

Sediment samples from each sample site were analyzed for dioxins and furans, but only the upper (0- to 3-foot) sampling interval was tested for samples from sample sites in New York where clamshell dredging will not occur. See above for discussion of analytes included in the analysis of samples at clamshell sites. (See Appendix A for a complete list of these dioxins and furans.) All dioxins and furans were detected in at least one sample in the alternative route (see Table 2-11 for the sample sites where dioxons/furans were detected). Of the dioxins and furans tested, only one dioxin (2,3,7,8-tetrachlorodibenzo-p-dioxin) and the total toxicity equivalence factor of all dioxins and furans have Class A and Class C thresholds.

Non-Clamshell Dredge Sample Sites

2,3,7,8-tetrachlorodibenzo-p-dioxin exceeded the Class A threshold in the 0- to 3 foot samples from sample sites VC70 and VC76. Concentrations that exceeded the Class A threshold were 6.04 pg/g and 1.38 pg/g, respectively. The total toxicity equivalency factor exceeded only the Class A threshold in the 0- to 3 foot samples at sample sites VC70, VC72, VC76, and VC85. Concentrations that exceeded the Class A threshold ranged from 4.51 pg/g to 19.56 pg/g.

Possible Clamshell Dredge Sample Sites

2,3,7,8-tetrachlorodibenzo-p-dioxin exceeded only the Class A threshold in the following samples: 0- to 3 feet at VC78 and VC79-ALT; 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC80; and 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, and 9- to 12 feet at VC81. Concentrations that exceeded the Class A threshold ranged from 1.53 pg/g to 28.10 pg/g.

The total toxicity equivalency factor exceeded the Class A threshold in the following samples:12- to 15.6 feet at VC77; 0- to 3 feet, 6- to 9 feet, 12- to15 feet, and 15- to 17 feet at VC78; 0- to 3 feet, 3- to 6 feet, and 12- to 16 feet at VC79-ALT; 0- to 3 feet, 6- to 9 feet, 9- to 12 feet, and 12- to15 feet at VC80; and 0- to 3 feet, 3- to 6 feet, and 9- to12 feet at VC81. The concentrations that exceeded the Class A threshold ranged from 4.59 pg/g to 38.56 pg/g. The total toxicity equivalency factor also exceeded the Class C threshold in the 3- to 6-foot sample from sample site VC80 and the 6- to 9-foot sample from sample site VC81. Concentrations that exceeded the Class C threshold were 53.01 pg/g and 65.72 pg/g, respectively.

2.4 Sediment Grain Size Results

Both vibracores and sediment grab samples were analyzed for grain size. The field geologist determined the number of depth intervals from each vibracore included in this analysis. This determination was based upon the observed uniformity of sediment type within each vibracore. Grain size analysis of these samples was conducted by the geotechnical laboratory using the sieve-based particle size analysis methodology (ASTM D6913) and/or a hydrometer (ASTM D7928). The grain size analysis for sediment grab samples was conducted by the geotechnical laboratory using the sieve-based particle size analysis methodology for unconsolidated material in the grab samples. Of the three sediment grab samples collected at each sample site, only the central sample was analyzed for grain size. Further discussion of grain sizes in the sediment grab samples from the preferred and the alternative routes are provided in Sections 4.1.1 and 4.2.1. Results of the sediment grain-size analysis are presented in Tables B-11 and B-12. Sediment grain size curves for vibracore sample sites are presented in Appendix E.

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3 Physical Water Quality Survey Results

The offshore portion of the Raritan Bay Loop will cross a continuous expanse of open marine and estuarine waters, including three major and contiguous waterbodies: Raritan Bay, Lower New York Bay, and the Atlantic Ocean (see Figure A-21). Raritan Bay covers the shallower portions of the Project area east of the Sandy Hook Peninsula and south of Crookes Point on Staten Island. Raritan Bay and Lower New York Bay are included in the New York – New Jersey Harbor Estuary Program (NYNJHEP). The waters of the Atlantic Ocean are under state jurisdiction (i.e., are state waters) from the shoreline out to 3 nautical miles (nm), which is the boundary where federal jurisdiction begins. The 3 nm limit of state-controlled waters is measured from a line between Sandy Hook and the western tip of the Rockaway Peninsula, as shown on NOAA Chart 12327 for New York Harbor (NOAA Fisheries 2016).

The water quality of the offshore Project area is influenced by many physical factors, including sediment inputs and geographic characteristics. A survey of physical water quality obtained data regarding background conditions in the water column along both the proposed and alternative pipeline routes.

The survey of water quality included the collection of water quality profiles using a Seabird SBE 19 Plus water quality meter. The SBE 19 meter collected a profile of the following physical parameters: DO, temperature, salinity, turbidity, pH, and conductivity. Water quality survey sites were co-located with the 87 vibracore sediment sampling locations (see Figure A-1 and A-2), and collected from October through December 2016. One profile record was conducted at each site, prior to any sediment sampling, in order to ensure the water was not impacted by disturbed sediment. Water quality readings were taken in the field at 2-foot depth intervals with the first reading at 2 feet (see Table B-13 for the depths sampled at each site). The results of the readings from the water quality meter are summarized below. The complete data output from the SBE 19 meter is provided in Appendix G.

Sample collection and laboratory analyses were not proposed for the water samples collected as part of the pre-construction investigations since chemical components in an open water environment are constantly changing. Thus, samples collected on one day may not be representative of conditions during the construction stage.

3.1 Dissolved Oxygen (DO)

Based on the field survey results, the average measured DO value along the entire proposed route was 8.56 mg/L and the concentration ranged from 4.69 mg/L to 11.88 mg/L. The average measured DO value along the alternative route was 8.53 mg/L and the concentration ranged from 6.77 mg/L to 9.84 mg/L. The ranges and averages of concentrations of DO measured at each sampling depth are presented for the proposed route and the alternative route in Table 3-1

Table 3-1 Dissolved Oxygen (mg/L) Measured at Depth

	Proposed Route			Alternative Route		
Depth (ft)	Minimum	Maximum	Average	Minimum	Maximum	Average
2	6.57	11.88	8.60	7.05	9.64	8.05
4	5.06	10.34	8.41	6.77	9.30	8.31
6	5.78	10.46	8.40	7.17	9.84	8.72
8	7.21	9.60	8.67	7.16	9.70	8.77
10	6.93	9.59	8.69	7.19	9.71	8.76
12	6.80	9.58	8.72	7.21	9.71	8.75
14	6.59	9.59	8.70	7.20	9.68	8.74
16	5.95	9.59	8.67	7.25	9.71	8.74
18	4.69	9.61	8.60	7.32	9.04	8.56
20	7.56	9.60	8.62	7.32	9.17	8.51
22	7.89	9.37	8.50	7.26	9.33	8.45
24	7.67	8.90	8.40	7.26	8.98	7.87
26	7.63	8.91	8.39	7.22	8.97	7.83
28	8.06	8.90	8.39	7.21	8.95	7.83
30	8.03	8.89	8.35	7.26	8.93	8.10
32	8.07	8.87	8.32	7.23	8.96	8.10
34	7.94	8.86	8.31	7.19	7.19	7.19
36	7.89	8.77	8.22	7.16	7.16	7.16
38	7.95	8.41	8.17	--	--	--
40	7.92	8.37	8.15	--	--	--

Key:
"—" = Depth was not included in profile

3.2 Temperature

Based on the field survey results, the average measured temperature along the entire proposed route was 48.41°F and temperatures ranged from 40.78°F to 60.07°F. The average measured temperature along the alternative route was 48.66°F and the temperature ranged from 43.29°F to 57.11°F. The ranges and averages of concentrations of temperature measured at each sampling depth for the proposed route and the alternative route are presented in Table 3-2.

Table 3-2 Temperature (°F) Measured at Depth Alternative Route

Depth (ft)	Proposed Route			Alternative Route		
	Minimum	Maximum	Average	Minimum	Maximum	Average
2	40.78	60.07	48.82	45.09	57.11	49.27
4	42.45	54.42	48.25	43.29	53.28	48.14
6	42.51	54.02	48.26	46.07	53.49	48.27
8	42.73	53.94	48.28	46.15	53.49	48.27
10	42.82	53.83	48.31	46.36	53.53	48.28
12	42.81	54.08	48.33	46.39	53.61	48.27
14	42.77	54.49	48.38	46.40	53.66	48.24
16	42.74	54.88	48.43	46.40	53.72	48.19
18	44.67	55.09	48.69	46.42	53.75	48.56
20	46.31	52.76	48.98	46.43	53.76	48.63
22	47.01	52.75	49.47	46.43	53.80	49.13
24	47.01	52.74	49.88	46.84	53.81	51.36
26	47.77	52.82	50.22	46.81	53.80	51.36
28	47.74	52.78	50.44	46.79	53.79	51.36
30	47.73	52.85	50.66	46.72	53.53	50.12
32	47.71	53.06	50.77	46.66	53.56	50.11
34	47.55	53.14	50.90	53.58	53.58	53.58
36	47.57	53.14	51.40	53.60	53.60	53.60
38	50.70	53.17	52.17	–	–	–
40	51.77	53.18	52.54	–	–	–

Key:
"—" = Depth was not included in profile

3.3 Salinity

Based on the field survey results, the average measured salinity along the entire proposed route was 28.72 practical salinity units (PSU) and salinity ranged from 12.33 PSU to 34.73 PSU. The average measured salinity along the alternative route was 27.35 PSU, and the salinity ranged from 22.71 PSU to 30.56 PSU. The ranges and averages of salinity measured at each sampling depth are presented for the proposed route and the alternative route in Table 3-3.

Table 3-3 Salinity (PSU) Measured at Depth

Depth (ft)	Proposed Route			Alternative Route		
	Minimum	Maximum	Average	Minimum	Maximum	Average
2	12.33	34.73	27.06	22.71	28.51	26.74
4	21.40	34.56	28.00	24.95	30.56	27.28
6	21.45	33.59	27.91	25.00	28.13	27.12
8	21.98	33.79	27.95	24.95	28.16	27.18
10	23.08	33.64	28.02	25.20	28.16	27.24
12	23.31	33.65	28.10	25.49	28.16	27.31
14	23.58	33.65	28.27	25.70	28.21	27.37
16	23.59	33.67	28.44	26.13	28.25	27.44

Table 3-3 Salinity (PSU) Measured at Depth

	Proposed Route			Alternative Route		
Depth (ft)	Minimum	Maximum	Average	Minimum	Maximum	Average
18	24.91	33.66	28.82	26.12	28.26	27.46
20	25.03	33.66	29.16	26.86	28.29	27.72
22	25.80	33.65	29.90	26.87	28.32	27.71
24	27.48	33.68	30.86	28.01	28.34	28.16
26	27.50	33.68	31.54	28.08	28.34	28.18
28	27.50	33.69	31.59	28.10	28.34	28.18
30	27.51	33.72	31.81	28.09	28.13	28.11
32	27.53	33.79	31.82	28.10	28.15	28.12
34	27.53	33.76	31.63	28.16	28.16	28.16
36	27.60	33.78	31.94	28.16	28.16	28.16
38	33.00	33.78	33.45	–	–	–
40	33.48	33.79	33.62	–	–	–

Key:
"—" = Depth was not included in profile

Field survey results indicated that salinity values increase from east to west along the pipeline route, with an average value of 23.19 PSU at VC1-ALT and an average value of 33.74 PSU at VC69-ALT.

3.4 Turbidity

Based on the field survey results, the average measured turbidity along the entire proposed route was 9.74 nephelometric turbidity units (NTU) and turbidity ranged from 0.39 NTU to 27.78 NTU. The average turbidity along the alternative route was 9.69 NTU and turbidity ranged from 0.26 NTU to 14.25 NTU. The ranges and averages of turbidity measured at each sampling depth for the proposed route and the alternative route are presented in Table 3-4.

Table 3-4 Turbidity (NTU) Measured at Depth

	Proposed Route			Alternative Route		
Depth (ft)	Minimum	Maximum	Average	Minimum	Maximum	Average
2	0.39	27.78	6.82	0.26	10.84	5.54
4	0.61	17.13	9.48	6.42	14.25	10.31
6	3.97	15.74	10.10	9.44	10.55	10.07
8	6.56	12.04	10.10	9.49	11.23	10.23
10	6.69	11.43	10.06	9.58	10.45	10.13
12	5.95	10.97	10.01	9.20	10.75	10.04
14	6.27	11.03	9.91	9.49	10.67	10.07
16	7.72	11.50	10.14	8.79	11.45	10.07
18	8.59	11.36	10.07	9.74	10.52	10.01
20	9.06	11.60	10.16	9.73	10.27	9.94
22	9.32	10.94	10.15	9.99	10.94	10.33
24	9.50	11.63	10.23	9.90	10.27	10.11

Table 3-4 Turbidity (NTU) Measured at Depth

	Proposed Route			Alternative Route		
Depth (ft)	Minimum	Maximum	Average	Minimum	Maximum	Average
26	9.21	11.09	10.08	9.81	10.04	9.93
28	9.50	10.82	10.06	9.89	10.18	10.05
30	9.37	10.96	10.00	10.19	10.63	10.41
32	8.91	10.27	9.72	10.21	10.24	10.22
34	9.72	10.58	10.17	10.03	10.03	10.03
36	9.68	10.25	9.95	10.21	10.21	10.21
38	9.34	10.53	9.95	–	–	–
40	9.90	10.57	10.14	–	–	–

Key:
"—" = Depth was not included in profile

3.5 pH

Based on the field survey results, the average measured pH along the entire proposed route was 7.98, and pH ranged from 5.87 to 8.30. The average pH along the alternative route was 7.72, and pH ranged from 6.40 to 8.58. The ranges and averages of pH measured at each sampling depth are presented for the proposed route and the alternative route in Table 3-5.

Table 3-5 pH Measured at Depth

	Proposed Route			Alternative Route		
Depth (ft)	Minimum	Maximum	Average	Minimum	Maximum	Average
2	5.87	8.22	7.63	6.40	7.98	7.07
4	6.28	8.25	7.79	6.88	8.58	7.67
6	7.10	8.30	7.98	7.53	8.11	7.81
8	7.11	8.30	7.99	7.52	8.02	7.81
10	7.14	8.28	7.98	7.53	8.02	7.82
12	7.15	8.28	7.99	7.53	8.02	7.82
14	7.17	8.28	7.99	7.54	8.02	7.82
16	7.17	8.28	7.99	7.55	8.02	7.82
18	7.18	8.28	8.03	7.56	7.98	7.80
20	7.65	8.28	8.07	7.57	7.98	7.78
22	7.79	8.28	8.10	7.60	7.98	7.77
24	7.78	8.28	8.13	7.74	7.79	7.77
26	7.86	8.28	8.16	7.75	7.79	7.77
28	7.87	8.29	8.16	7.75	7.80	7.77
30	7.87	8.28	8.17	7.75	7.80	7.77
32	7.87	8.28	8.17	7.75	7.80	7.78
34	7.87	8.29	8.15	7.75	7.75	7.75
36	7.90	8.28	8.17	7.75	7.75	7.75
38	8.15	8.28	8.25	–	–	–
40	8.15	8.28	8.25	–	–	–

Key:
"—" = Depth was not included in profile

3.6 Conductivity

Based on the field survey results, the average measured conductivity along the entire proposed route was 31.37 millliSiemens per centimeter (mS/cm) and conductivity ranged from 12.46 mS/cm to 38.54 mS/cm. The average conductivity along the alternative route was 29.94 mS/cm and conductivity ranged from 27.16 mS/cm to 33.18 mS/cm. The ranges and averages of conductivity measured at each sampling depth are presented for the proposed route and the alternative route in Table 3-6.

Table 3-6 Conductivity (mS/cm) Measured at Depth

	Proposed Route			Alternative Route		
Depth (ft)	Minimum	Maximum	Average	Minimum	Maximum	Average
2	12.46	37.87	29.63	27.16	33.05	29.53
4	23.64	37.90	30.40	27.62	32.52	29.67
6	23.86	38.03	30.31	27.78	32.70	29.55
8	24.01	38.32	30.37	27.74	32.72	29.60
10	24.03	38.14	30.45	27.96	32.76	29.66
12	24.02	38.14	30.55	28.15	32.90	29.73
14	24.02	38.15	30.73	28.27	32.97	29.78
16	24.01	38.18	30.92	28.54	33.04	29.83
18	27.80	38.19	31.41	27.80	33.06	30.01
20	27.81	38.19	31.89	28.71	33.10	30.30
22	28.30	38.18	32.84	28.72	33.16	30.50
24	29.60	38.21	33.98	29.91	33.18	31.92
26	29.74	38.24	34.82	29.89	33.18	31.94
28	29.74	38.23	34.98	29.87	33.17	31.95
30	29.74	38.31	35.30	29.83	32.83	31.33
32	29.74	38.48	35.38	29.81	32.87	31.34
34	29.64	38.50	35.26	32.88	32.88	32.88
36	29.72	38.52	35.81	32.89	32.89	32.89
38	36.48	38.53	37.68	–	–	–
40	37.59	38.54	38.04	–	–	–

Key:
"—" = Depth was not included in profile

4 Benthic Community Analysis

As part of a 2016 offshore sediment sampling effort, Transco conducted a site-specific baseline benthic survey to assess the existing benthic community along the proposed pipeline route. Grab samples of surface sediments were collected at 69 sampling sites along the Raritan Bay Loop route and at 18 stations along the alternative route (for sample site locations see Figures A-1 and A-2). At each sample site, three discrete grab samples were taken: one centered on the proposed corridor and two located approximately 10 meters north (N) and south (S) of the central (C) sample location. A modified Day grab sampler was used to collect the samples from 1 square-foot (0.1 square-meter) sections of the seabed. Samples were transported to an onshore laboratory for specimen identification. Collected benthic infauna were identified to the lowest possible taxonomic category and benthic community parameters, including abundance, richness, dominant taxon, evenness of the distribution, and other community assemblage patterns were calculated.

4.1 Proposed Route

The survey identified 208 taxa that included annelids, crustaceans, round worms, ribbon worms, gastropods, bivalves, and shrimp. Table 4-1 below presents the top 25 taxa identified based on relative abundance. A complete list of identified taxa, along with numerical information on their occurrence is provided in Table B-14. Table B-15 presents a summary of sampling results from each sample site along the proposed route.

Table 4-1 Top 25 Taxa Identified for All Stations by Abundance for the Proposed Route

Rank	Taxon	Common Name	Frequency of Occurrence (N=207)	Total Number of Individuals	Relative Abundance (%)
1	*Mediomastus ambiseta*	polychaete	134	93041.8	56.83
2	*Streblospio benedicti*	polychaete	110	15020.7	9.18
3	*Oligochaeta*	oligochaete	177	8553.2	5.22
4	*Cirratulidae*	polychaete	64	3706.4	2.26
5	*Glycinde solitaria*	polychaete	84	3268.7	2.00
6	*Polygordius jouinae*	polychaete	99	2814.4	1.72
7	*Exogone dispar*	polychaete	61	2565.4	1.57
8	*Ameritella agilis*	mollusk	116	2199.7	1.34
9	*Ampelisca abdita*	amphipod	32	1942.1	1.19
10	*Sabellaria vulgaris*	polychaete	40	1798.7	1.10
11	*Tharyx acutus*	polychaete	60	1330.9	0.81

Table 4-1 Top 25 Taxa Identified for All Stations by Abundance for the Proposed Route

Rank	Taxon	Common Name	Frequency of Occurrence (N=207)	Total Number of Individuals	Relative Abundance (%)
12	*Eumida sanguinea*	polychaete	56	1312.1	0.80
13	*Unciola serrata*	amphipod	47	1147.6	0.70
14	*Maldanidae*	polychaete	47	1128.3	0.69
15	*Protohaustorius cf. wigleyi*	amphipod	26	1126.7	0.69
16	*Parametopella cypris*	amphipod	10	1055.8	0.64
17	*Euclymene collaris*	polychaete	49	955.6	0.58
18	*Stenothoe minuta*	amphipod	6	900.8	0.55
19	*Caulleriella venefica*	polychaete	73	872.9	0.53
20	*Glycera americana*	polychaete	61	758.7	0.46
21	*Tritia trivittata*	gastropod	83	703.5	0.43
22	*Tubulanus pellucidus*	nemertean	49	654.2	0.40
23	*Heteromastus filiformis*	polychaete	36	551.0	0.34
24	*Crepidula fornicata*	gastropod	30	527.7	0.32
25	*Cyathura burbancki*	isopod	37	524.2	0.32

4.1.1 Sediment Grain Size

Sediment grain size was analyzed only for the central (C) grab sample. Sediment grain size generally is partitioned into two distinct groupings: fine-grained versus coarse-grained. The fine-grained fraction of a sediment is defined as silt (particles with diameters less than 62 microns down to 4 microns) plus clay (particles with diameters less than 4 microns, with colloidal clay being less than 0.1 microns) (Poppe et al. 2000). Coarse-grained sediments, not including shell fragments, were those having particle diameter greater than 62 microns. Although there is no specific guidance for labeling sediment types as fine or coarse grained, for this study, and based on information provided by the EPA (2015) for ocean disposal of dredged material, stations that had more than 10% fine-grained material in the composite analysis were considered 'fine-grained' habitat. Those having less than 10% fine-grained material were considered 'sandy' or coarse-grained habitat.

Generally, substrate structure transitioned from fine-grained sediment at western stations to sandy at eastern stations (Figure 4-1). This trend likely reflects depositional conditions to the west associated with Raritan Bay and Raritan River. Steimle et al. (1989) noted an irregular band of silt from the mouth of the Raritan River to Sandy Hook during their benthic survey. This study may have crossed similar habitat within the western portion of the route. In addition, a greater percentage of fine-grained sediments associated with the Chapel Hill South Channel were found between Stations VC37 through VC41-Alt. The presence of these sediments may reflect the depositional nature of the channel. This phenomenon is common, and concerns regarding the association of organic compounds within fine-grained, depositional channel sediments, was noted by Speight (2014).

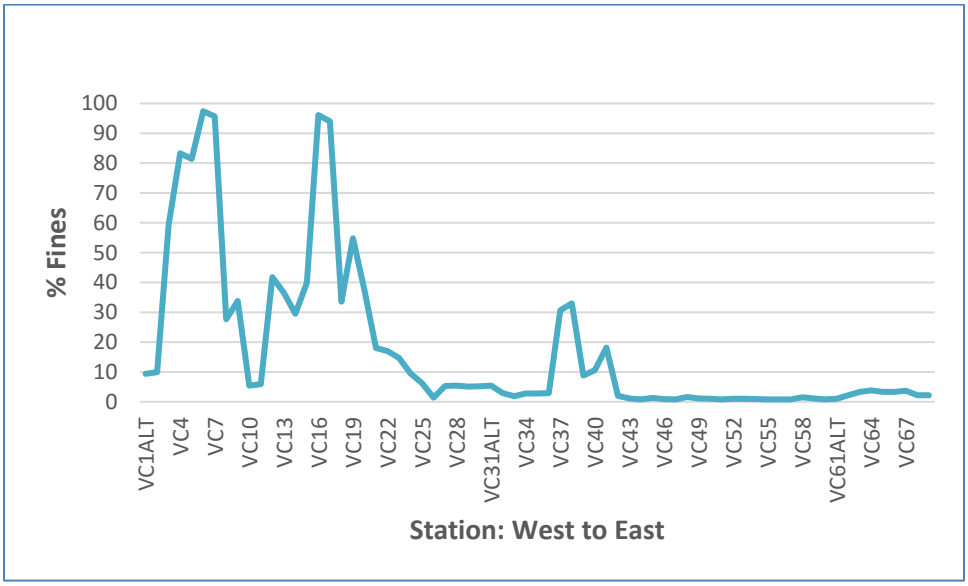


Figure 4-1 Fine-grained (>10%) Sediments at Stations Along the Proposed Route

4.1.2 Macrobenthic Invertebrate Densities

Macrobenthic invertebrate densities ranged from less than 20 to more than 4,000 organisms per 0.1 m^2 (mean = 791). Taxonomic richness ranged from 5 to 28 species (mean = 17.2). These relatively low richness values are typical for dynamic, stressed conditions in nearshore environments. Similar seasonal (fall-winter) richness values (~20 taxa per sample) were found by Reid et al. (1991) at inshore stations in their study of the New York Bight.

Two community parameters, Shannon-Weaver's diversity index and Pielou's evenness index, were calculated for measuring the consistency of taxa and numbers within each sample. Shannon-Weaver's (H′) diversity index increases as the richness, or number of taxa, of the community increases. Pielou's (J′) evenness index measures the relative abundance compared with all taxa that are found within a sample. Pielou's index can range from 0 to 1, with 1 being a community that exhibits a uniform distribution of species. The H′ indices ranged from 0.411 to 2.85, and J′ ranged from 0.0507 to 0.7982.

Generally, higher abundance was noted at stations that had higher percentages of fine-grained sediments. Correlation analysis (Pearson) revealed that although there was a positive (coefficient = 0.3594) relationship between abundance and percent fine-grained material, the relationship was not strong. A linear regression of abundance (base-10) for comparison with percent fine-grained sediment also did not reveal a statistically significant relationship, although a positive slope was observed (Figure 4-2).

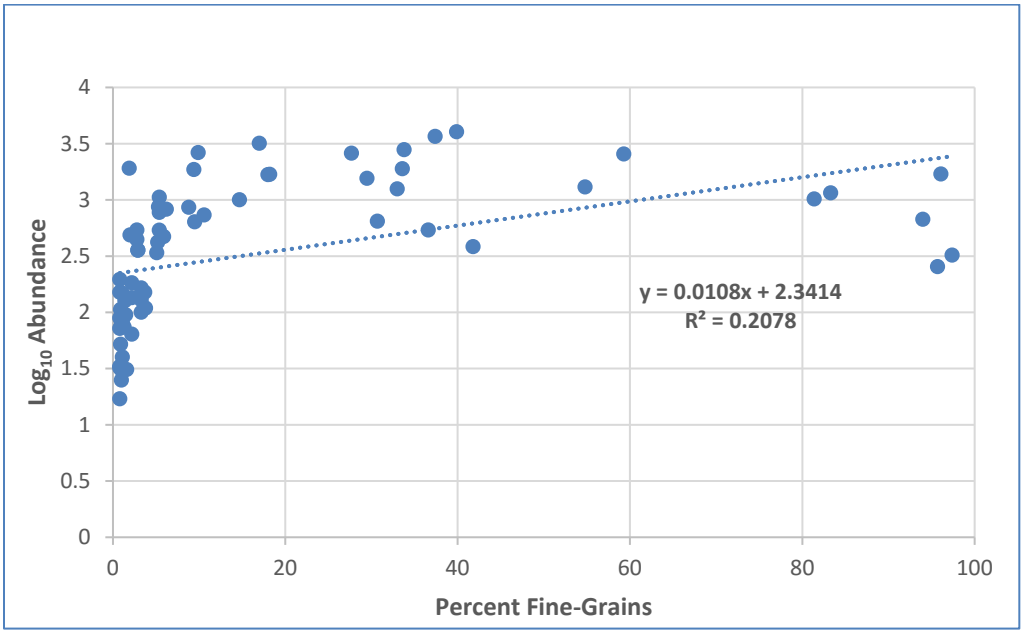


Figure 4-2 Linear Regression of Abundance (log-10) to Percent Fine-grained Sediment

Highest abundance was noted at stations to the west; for the top 20 stations with highest taxa abundance, all but one was west of the Chapel Hill South Channel. Fifteen of the top 20 stations in taxa abundance had fine-grained sediments that averaged 45 percent. Generally, at stations having either high or low percentages of fine-grained sediment, abundance was somewhat reduced, suggesting that mixed sediment types tend to support larger populations. This could likely be related to a more diverse habitat (i.e., variable grain size), greater organic carbon, higher productivity, and/or greater food resources. Higher abundance values were typically associated with an abundance of polychaete taxa and individuals.

It was also noted, generally, that stations having lower percentages of fine-grained sediment, especially those to the east, also had highest evenness values, but Pearson correlation analysis (coefficient = -0.5047) did not reveal a positive relationship between the two variables. Similarly, richness could not be predicted based on sediment type (coefficient = -0.1179). Generally, greater diversity was found at stations to the east and at stations associated with sandy habitat. Amphipods (*Ampelisca* spp., *Unciola* spp., *Protohaustorius wigleyi*, and *Stenothoe minuta*) were almost exclusively found at sandy stations. At these stations, the average number of gammarid amphipods per sample tended to be much greater than what Reid et al. (1991) reported (10 per 0.1 m^2) for fall sampling at inshore New York and New Jersey locations. Greater diversity is likely a result of a more stable environment, compared with depositional, fine-grained sediment habitat that is typically in constant flux and associated with opportunistic, tolerant, early successional (Stage 1) species, such as polychaete genera *Mediomastus*, *Streblospio* and *Exogone* noted by McCall (1977).

4.1.3 Community Analysis

A Bray-Curtis multivariate (resemblance) cluster analysis revealed that there were three major community groups identified across the route (Figure 4-3). Resemblance measures such as cluster analysis have been shown to be one of the most consistent methods for analyzing benthic information (Jackson 1993). The major division was between stations at the western end of the pipeline (VC1-ALT – VC42) and those on the eastern section (VC43 – VC69). The most uniform of these three groups included stations VC2 to VC27. This group had similarities typically in the 60% to 70% range. A second group, related to the first, consisted of stations VC28 to VC40. There were three outliers in this section of the dendrogram. A single station, VC26, was the most distinctive, showing minimal similarity with any other site (<25%). Inspection of the faunal data revealed that the distinction was due to the high numbers of the polychaetes *Polycirrus* spp. and *Exogone dispar*, and the gastropod *Tritia trivittata*. In spite of the fact that sediments were similar to stations either side of VC26, there were very low numbers of the typical dominants for that section of the pipeline, i.e., *Mediomastus ambiseta*, *Streblospio benedicti*, and oligochaetes. The other outliers in this subset, Stations VC33-ALT and VC42, had unusually large numbers of two amphipod species in the samples, *Parametopella cypris* and *Stenothoe minuta*. The final cluster, Stations VC43 and above had lower overall similarity to each other and with several subsets. Within this group Stations VC63 – VC69, dominated by haustoriid amphipods, had the greatest overall similarity.

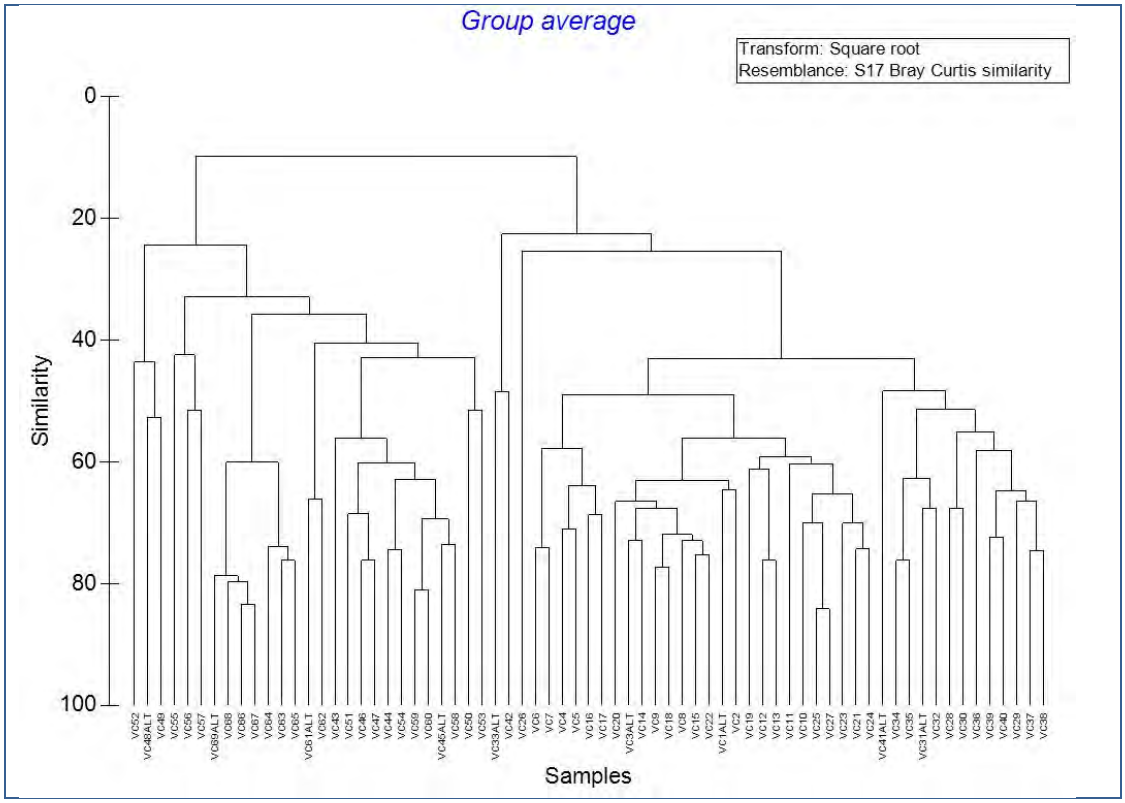


Figure 4-3 Multivariate Cluster Analysis of Pipeline Proposed Route Stations

Faunal data were also subjected to non-metric multidimensional scaling (MDS), an ordination procedure where stations are placed along a set of axes reflecting their similarity. The results (Figure 4-4) show a close grouping of lower numbered stations up to VC42 with some separation of the lowest numbers higher on the y axis. At the left end of the x-axis, and showing lower overall similarity, are the stations in the 50s and 60s. MDS supports the grouping of stations observed by cluster analysis which shows a distinction between low numbered stations found at the western end of the route and those with higher numbers found to the east. Again, the separation of groups is indicative of varying sediment types found along the proposed pipeline route.

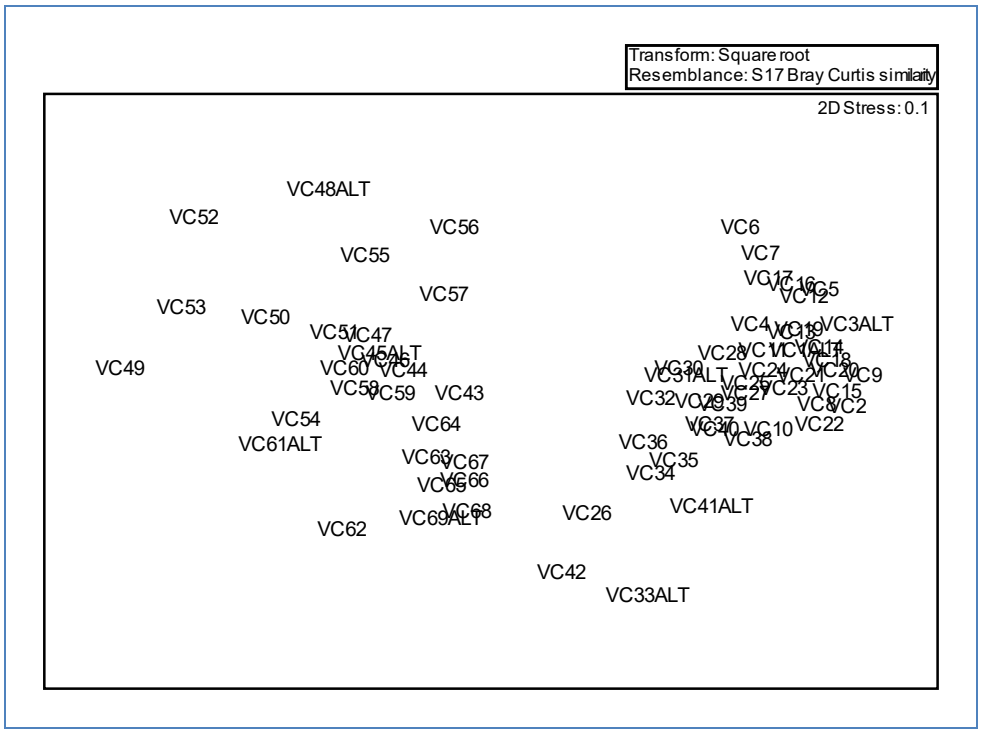


Figure 4-4 MDS for Station Similarity of Benthic Assemblage Along Proposed Route

4.1.4 Occurrence of Hard Clams

As a subset of the 2016 benthic macroinvertebrate survey, Transco performed a stand-alone analysis of hard clam (*Mercenaria mercenaria*) occurrence within a select portion (between MPs 14.40 and 21.60) of the proposed route. Stations selected for analysis were located in NYSDEC Uncertified Shellfish Areas and included those stations that were located within the NYSDEC special permit harvest area associated with a hard clam transplantation program. Data were obtained at designated (north [N], south [S] and center [C]) locations for stations VC7-VC28. For the analyses, the number of hard clam individuals found at all three locations (N, S and C), per station, were combined.

Hard clams were categorized based on size (hard clams are measured from end to end along a straight line that runs parallel to the hinge). Partitioned length groups

included those individuals less than 25 millimeters (mm) (<25 mm) and greater than 25 mm (>25 mm). Of the 213 clams found along the section of the proposed route, 158 (74%) were <25 mm, and 55 (26%) were >25 mm (Table 4-2). Table 4-3 presents the descriptive statistics for the sample pool.

Table 4-2 Hard Clam Counts - Proposed Route Stations

Station	<25 mm	>25 mm	Total
VC7	0	0	0
VC8	32	0	32
VC9	0	0	0
VC10	21.3	3	24.3
VC11	7.1	1	8.1
VC12	0	8	8
VC13	10.9	9	19.9
VC14	0	2	2
VC15	16	3	19
VC16	0	0	0
VC17	0	0	0
VC18	0	8	8
VC19	0	7	7
VC20	0	3	3
VC21	0	3	3
VC22	64	2	66
VC23	6.4	0	6.4
VC24	0	1	1
VC25	0	1	1
VC26	0	1	1
VC27	0	1	1
VC28	0	2	2
Total	**157.7**	**55**	**212.7**
Percent of Total	**74%**	**26%**	

Table 4-3 Hard Clam Descriptive Statistics for Proposed Route Stations

Total	N	Mean	Std Dev.	Size Ratio[1]
212.7	22	9.7	15.4	2.8:1

Note:
[1] Ratio of individuals <25 mm to >25 mm

4.2 Alternative Route

As with the proposed route, sample data from the alternative route determined abundance, richness, dominant taxon, evenness of the distribution, and other community assemblage patterns from 18 stations (Table B-16). For each sample site, three discrete grab samples were taken: one centered on the proposed corridor, and two located approximately 10 meters north (N) and south (S) of the central (C) sample location. Abundance ranged from almost 5,000 individuals at VC72 to less than 100 at VC78. The survey identified 111 taxa that included pol-

ychaetes, annelids, crustaceans, gastropods, and bivalves. The top 5 taxa made up more than 78% of the individuals identified (Table 4-4). A complete list of identified taxa, along with numerical information on their occurrence is provided in Table B-17.

Table 4-4 Top 25 Taxa Identified for all Stations by Abundance for the Alternative Route

Rank	Taxonomic Name	Common Name	Frequency of Occurrence (N=54)	Total Number of Individuals	Relative Abundance (%)
1	*Mediomastus ambiseta*	polychaete	54	28598.7	51.95
2	*Streblospio benedicti*	polychaete	35	5884.9	10.69
3	*Ampelisca abdita*	amphipod	21	3467.5	6.30
4	*Oligochaeta*	oligochaete	39	3373.9	6.13
5	*Exogone dispar*	polychaete	46	1835.1	3.33
6	*Glycinde solitaria*	polychaete	38	1367.4	2.48
7	*Opisthodonta longocirrata*	polychaete	13	759.7	1.38
8	*Ameritella agilis*	mollusk	28	685	1.24
9	*Euclymene collaris*	polychaete	24	679.7	1.23
10	*Caulleriella venefica*	polychaete	13	596.1	1.08
11	*Brania wellfleetensis*	polychaete	10	517.3	0.94
12	*Mercenaria mercenaria*	mollusk	34	487.9	0.89
13	*Eumida sanguinea*	polychaete	20	445.5	0.81
14	*Mulinia lateralis*	mollusk	14	431.6	0.78
15	*Cirratulidae*	polychaete	8	387.6	0.70
16	*Polydora cornuta*	polychaete	12	310.9	0.56
17	*Polycirrus* sp.	polychaete	13	297.2	0.54
18	*Tritia trivittata*	Gastropod	26	281.7	0.51
19	*Sabellaria vulgaris*	polychaete	9	245.5	0.45
20	*Hypereteone* sp.	polychaete	12	239.7	0.44
21	*Maldanidae*	polychaete	13	214.3	0.39
22	*Cyathura burbancki*	isopod	11	205.7	0.37
23	*Oxydromus obscurus*	polychaete	11	200.9	0.37
24	*Glycera americana*	polychaete	25	192.2	0.35
25	*Myrianida prolifera*	polychaete	7	182	0.33

4.2.1 Sediment Grain Size and Macrobenthic Invertebrate Densities

There was no clear spatial trend for substrate structure along the alternative route (Figure 4-5). Most stations (12) contained fine-grained sediment, but stations in the most easterly segment (VC82 – VC86) exhibited coarse or sandy substrates. These coarse sediments at the easterly end were somewhat different from those of the preferred route with a median grain size of approximately 0.4 mm. Stations at the eastern end of the preferred route, although having similar percentages of fines (2% – 3%) had median grain sizes of approximately 0.15 mm. As a result, the fauna were different, with haustoriid amphipods dominant at the proposed route, and virtually absent at the alternate route. Among the dominants in this area were several species that were absent or at low densities for the other segments of the alternative route, including the polychaetes *Opisthodonta longocirrata, Brania wellfleetensis*, *Polycirrus sp., and Polygordius jouinae*.

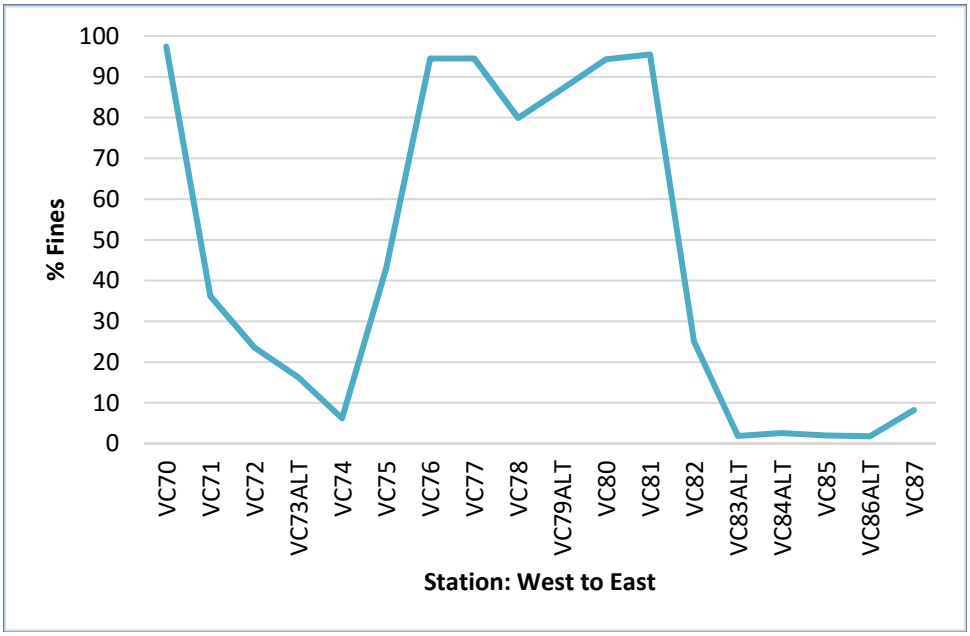


Figure 4-5 Fine-grained (>10%) Sediments at Stations Along the Alternative Route

Multivariate analysis (Figure 4-6) revealed three major clusters that represent three sediment groups. Fine sediments at the western end (VC71 – VC74), coarse sediments at the eastern end (VC83 – VC86), and the remaining group an assortment of intermediate sediments.

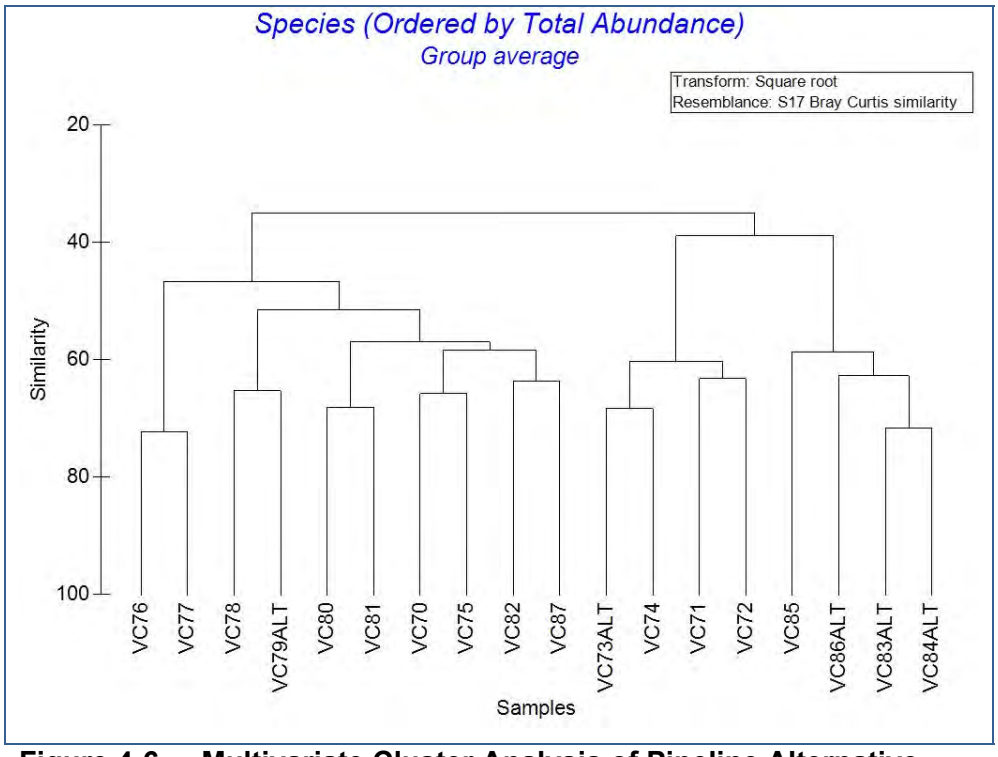


Figure 4-6 Multivariate Cluster Analysis of Pipeline Alternative Route Stations

Non-metric multidimensional scaling produced a similar arrangement of groups with the eastern stations (VC83 – VC86) separated from the remaining sites along the y-axis and the fine sediment stations (VC71 – VC74) removed from the mixed sediment sites along the x-axis (Figure 4-7).

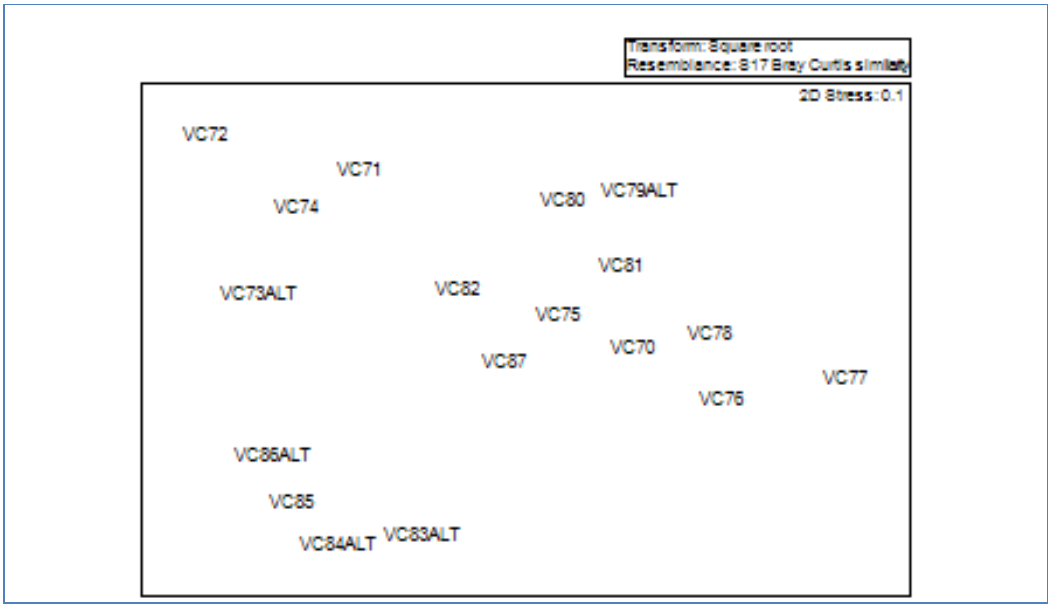


Figure 4-7 MDS for Station Similarity of Benthic Assemblage Along the Alternative Route

Overall, there was a significant overlap in the fauna between the proposed and alternative pipeline routes. This was particularly true at the western end where the polychaetes *Mediomastus ambiseta, Streblospio benedicti* and oligochaetes were numerically dominant in both data sets. In the mixed sediments of the middle sections most species found along the alternative route were also found along the proposed route. The major difference between the two routes was at the eastern end where a slight difference in the sediment composition resulted in a significant difference in the benthic fauna. Haustoriid amphipods such as *Protohaustorius wigleyi* and *Acanthohaustorius similis* were replaced as dominants by several polychaete species.

4.2.2 Occurrence of Hard Clams

A similar assessment of hard clam densities was performed for the alternative route under consideration. Stations selected for analysis were located in NYSDEC Uncertified Shellfish Areas and included those stations that were located within the NYSDEC special permit harvest area associated with a hard clam transplantation program. Data were obtained at designated (north [N], south [S] and center [C]) locations for stations VC70-VC87. As noted for the proposed

route above, the number of hard clam individuals found at all three locations (N, S, and C), per station, were combined.

Hard clams were categorized based on size (hard clams are measured from end to end along a straight line that runs parallel to the hinge). Partitioned length groups included those individuals less than 25 mm (<25 mm) and greater than 25 mm (>25 mm). Of the 488 clams found along the selected route, 423 (86%) were <25 mm, and 65 (14%) were >25 mm (Table 4-5). Table 4-6 presents the descriptive statistics for the sample pool.

Table 4-5 Hard Clam Counts - Alternative Route Stations

Station	<25 mm	>25 mm	Total
VC70	0	0	0
VC71	25.6	3	28.6
VC72	128	1	129
VC73-Alt	114.7	3	117.7
VC74	81.6	1	82.6
VC75	0	8	8
VC76	8	6	14
VC77	31.5	11	42.5
VC78	6.4	4	10.4
VC79-Alt	0	4	4
VC80	0	0	0
VC81	0	1	1
VC82	16	13	29
VC83-Alt	0	0	0
VC84-Alt	0	1	1
VC85	3.6	1	4.6
VC86-Alt	0	2	2
VC87	7.6	6	13.6
Total	**423**	**65**	**488**
Percent of Total	**86%**	**14%**	

Table 4-6 Hard Clam Descriptive Statistics for Alternative Route Stations

Total	N	Mean	Std Dev.	Size Ratio[1]
488	18	27.1	40.7	6.5:1

Note:
[1] Ratio of individuals <25 mm to >25 mm

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5 References

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A Figures

Figure A-1 Project Location Map

Figure A-2 Offshore Location

Figure A-3 Offshore Sample Site Map

Figure A-4 Offshore Sediment Sample contamination Results – Metals

Figure A-5 Metal Exceedances at Depth Along the Preferred Route

Figure A-6 Offshore Sediment Sample contamination Results – Pesticides

Figure A-7 Pesticide Exceedances at Depth Along the Preferred Route

Figure A-8 Offshore Sediment Sample contamination Results – PCB Aroclors

Figure A-9 PCB Aroclor Exceedances at Depth Along the Preferred Route

Figure A-10 Offshore Sediment Sample contamination Results – PCB Congeners

Figure A-11 Offshore Sediment Sample contamination Results – PAHs

Figure A-12 PAH Exceedances at Depth Along the Preferred Route

Figure A-13 Offshore Sediment Sample contamination Results – Other SVOCs

Figure A-14 Offshore Sediment Sample contamination Results – Dioxins and Furans

Figure A-15 Dioxin and Furan Exceedances at Depth Along the Preferred Route

Figure A-16 Metal Exceedances at Depth Along the Preferred Route

Figure A-17 Pesticide Exceedances at Depth Along the Preferred Route

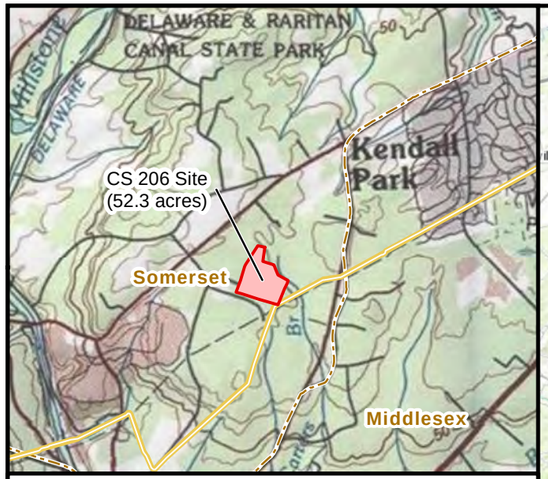
Figure A-18 PCB Aroclor Exceedances at Depth Along the Preferred Route

Figure A-19 PAH Exceedances at Depth Along the Preferred Route

Figure A-20 Dioxin and Furan Exceedances at Depth Along the Preferred Route

Figure A-21 Offshore Waterbodies

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Compressor Station 206 Site

Northampton County
Warren County
Lehigh County
Hunterdon County
Somerset County
Union County
Richmond County
Kings County
Queens County
Rockaway Delivery Lateral
Morgan M&R Station
Raritan Bay
MP 35.49
STA. 207
MP 12.00
MP 8.57
Compressor Station 206 Site
Middlesex County
Raritan Bay Loop (Offshore)
Madison Loop
STA. 205
Bucks County
PA
Mercer County
Monmouth County
Montgomery County
Trenton
State Waters
Federal Waters
Atlantic Ocean
STA. 200
Lancaster County
Chester County
Philadelphia County
Quarryville Loop
MP 1691.17
MP 1681.00
York County
STA. 195
Delaware County
Burlington County
Ocean County
NJ
Camden County
Gloucester County
Salem County
Cumberland County
Atlantic County
DE
MD
PENNSYLVANIA
MARYLAND
New Castle County
Cecil County
Harford County

Legend

- Milepost
- Existing Compressor Station
- Existing Meter & Regulating Station
- Compressor Station 206
- Proposed Pipeline
- Existing Transco Pipeline
- State Boundary
- County Boundary
- State/Federal Offshore Line

N

0 10 20
Miles

SITE LOCATION



DRAWING NO.

REFERENCE TITLE

A-1

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
PROJECT LOCATION MAP
NORTHEAST SUPPLY ENHANCEMENT PROJECT
PENNSYLVANIA, NEW JERSEY, NEW YORK

Williams

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
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B	8/31/2016	AL	ISSUED FOR PLANNING PRESENTATION	1000891	JM	JM
C	11/14/2016	AL	ISSUED FOR FERC DRAFT FILING	1000891	JM	SM
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Data Sources: BOEM 2005; Williams 2017; E&E 2017; ESRI 2012, 2017.

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Bergen Co.
Morris Co.
Essex Co.
Hudson Co.
New York Co.
Queens Co.
Nassau Co.
Union Co.
Somerset Co.
Kings Co.
Richmond Co.
Middlesex Co.
Monmouth Co.
Rockaway Inlet
Raritan Bay Loop (Offshore)
Raritan Bay Loop (Onshore)
Madison Loop (Onshore)
New York
New Jersey
Federal Jurisdiction
Raritan Bay
Atlantic Ocean

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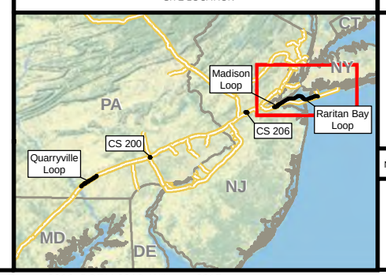
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- Raritan Bay Loop
- Madison Loop
- State Boundary
- State/Federal Offshore Line
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

N

0 2.5 5

Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE
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OFFSHORE LOCATION
NEW JERSEY AND NEW YORK

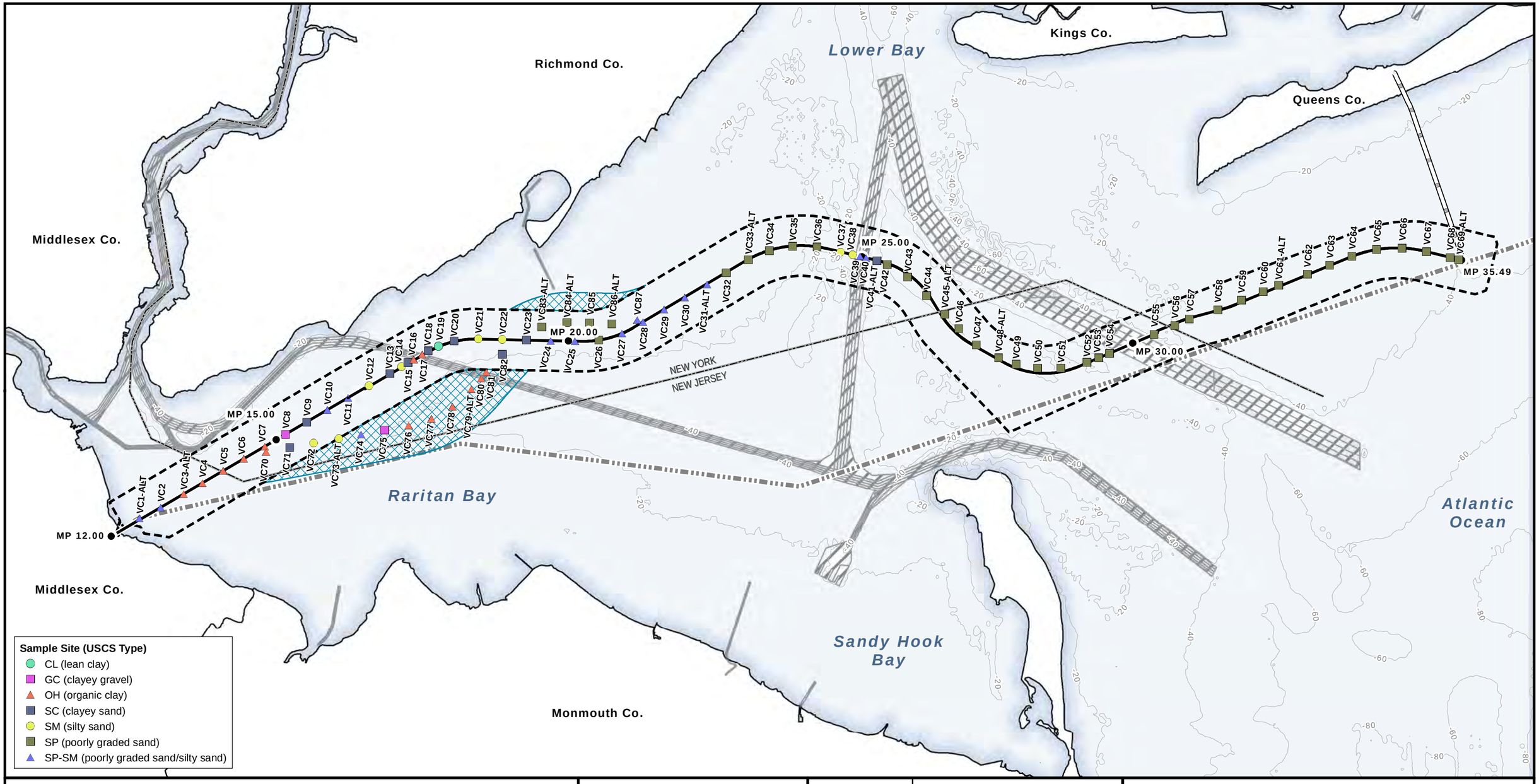
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Data Sources: E&E 2017; ESRI 2012; ESRI Base Map Services 2017; NYS Office of Information Technology Services GPO 2016; New Jersey Office of GIS 2016; U.S. Census Bureau 2016; BOEM 2010; Williams 2017.

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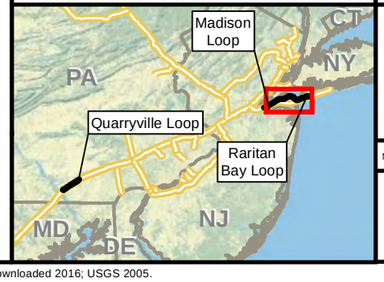
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- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel
- Study Area Extension

N

0 1 2

Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE
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OFFSHORE SAMPLE SITE MAP
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

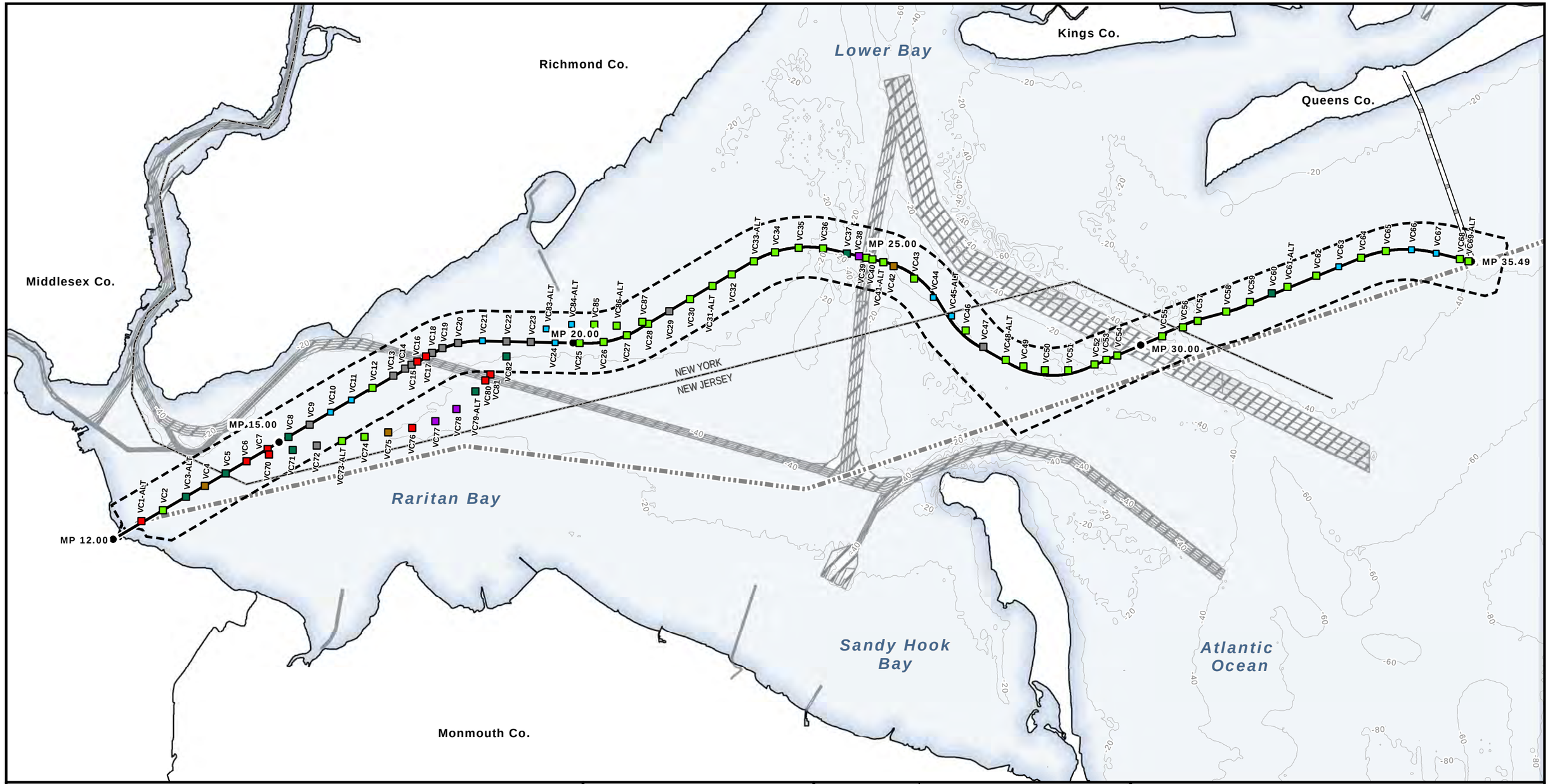
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Data Sources: Williams 2017; E&E 2017; ESRI 2012; ESRI Base Map Services 2017; NJDEP Digital Data Series Bathymetric Data 2006; NOAA ENC, Approach Scale, data downloaded 2016; USGS 2005.

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Legend

Metals Exceedances*

- 0
- 1-2
- 3-4
- 5-6
- 7-8
- 11-12
- 13-15

- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

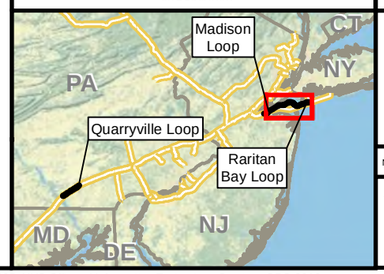
*Number of analyte exceedances for lowest threshold of either the NJDEP Ecological Screening Criteria or NYSDEC Sediment Guidance Values.

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Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE
	A-4

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
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TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE CONTAMINATION RESULTS
METALS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

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Data Sources: Williams 2017; E&E 2017; ESRI 2012, 2017; NJDEP Digital Data Series Bathymetric Data 2006; NOAA ENC, Approach Scale, data downloaded 2016; USGS 2005; NYS Office of Information Technology Services GPO 2016.

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02:1000891.0020.05-Figure A-5 Metal Exceedances at Depth.ai-5/18/17

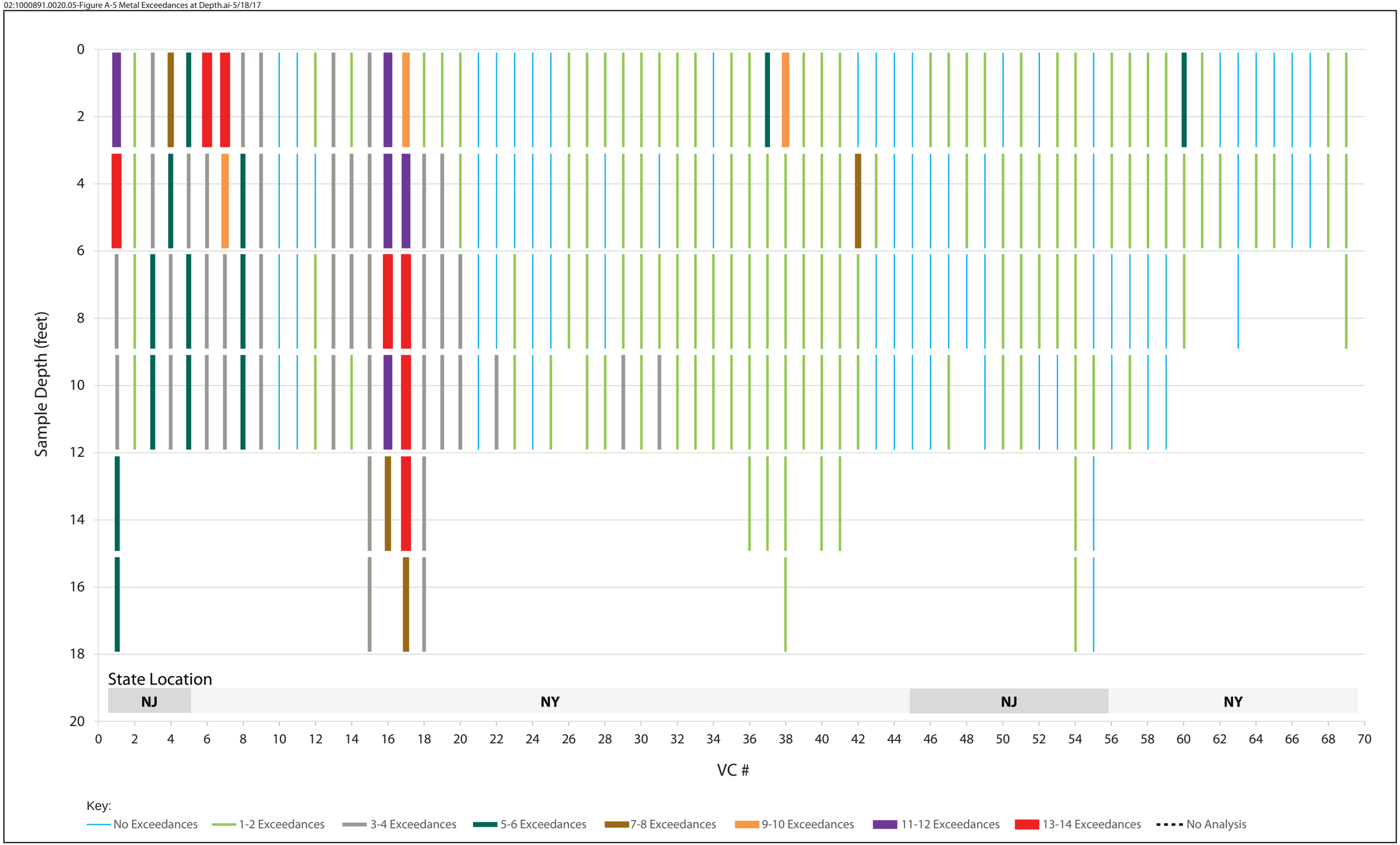
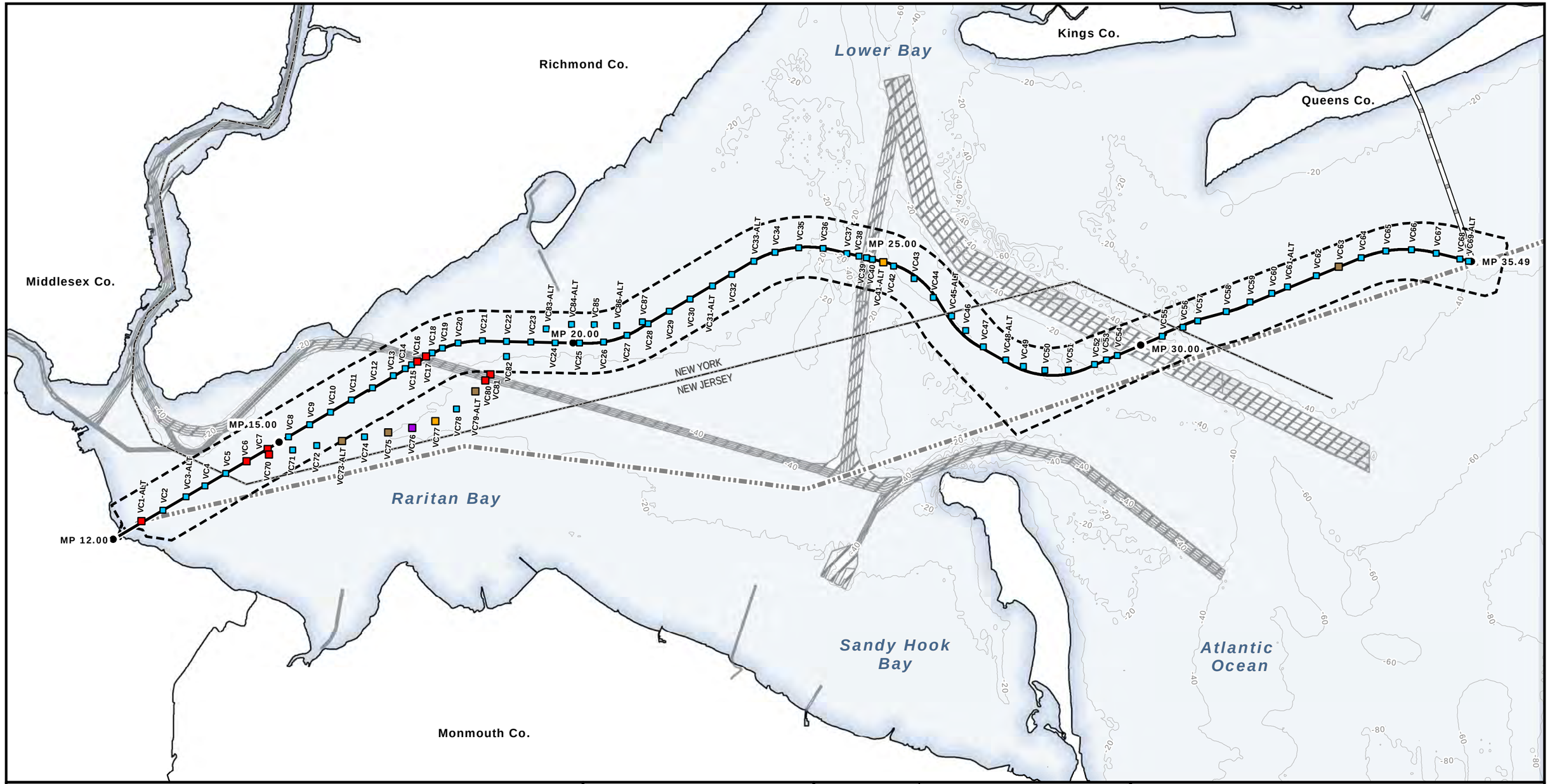


Figure A-5: Metal Exceedances at Depth Along the Preferred Route



Legend

Pesticide Exceedances*

- 0
- 1
- 2
- 3
- 4

- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

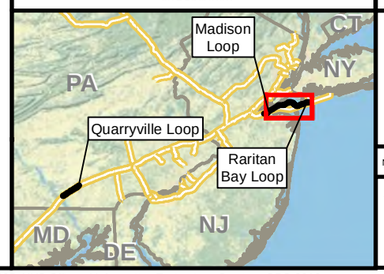
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N

0 1

Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE
	A-6

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE CONTAMINATION RESULTS
PESTICIDES
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

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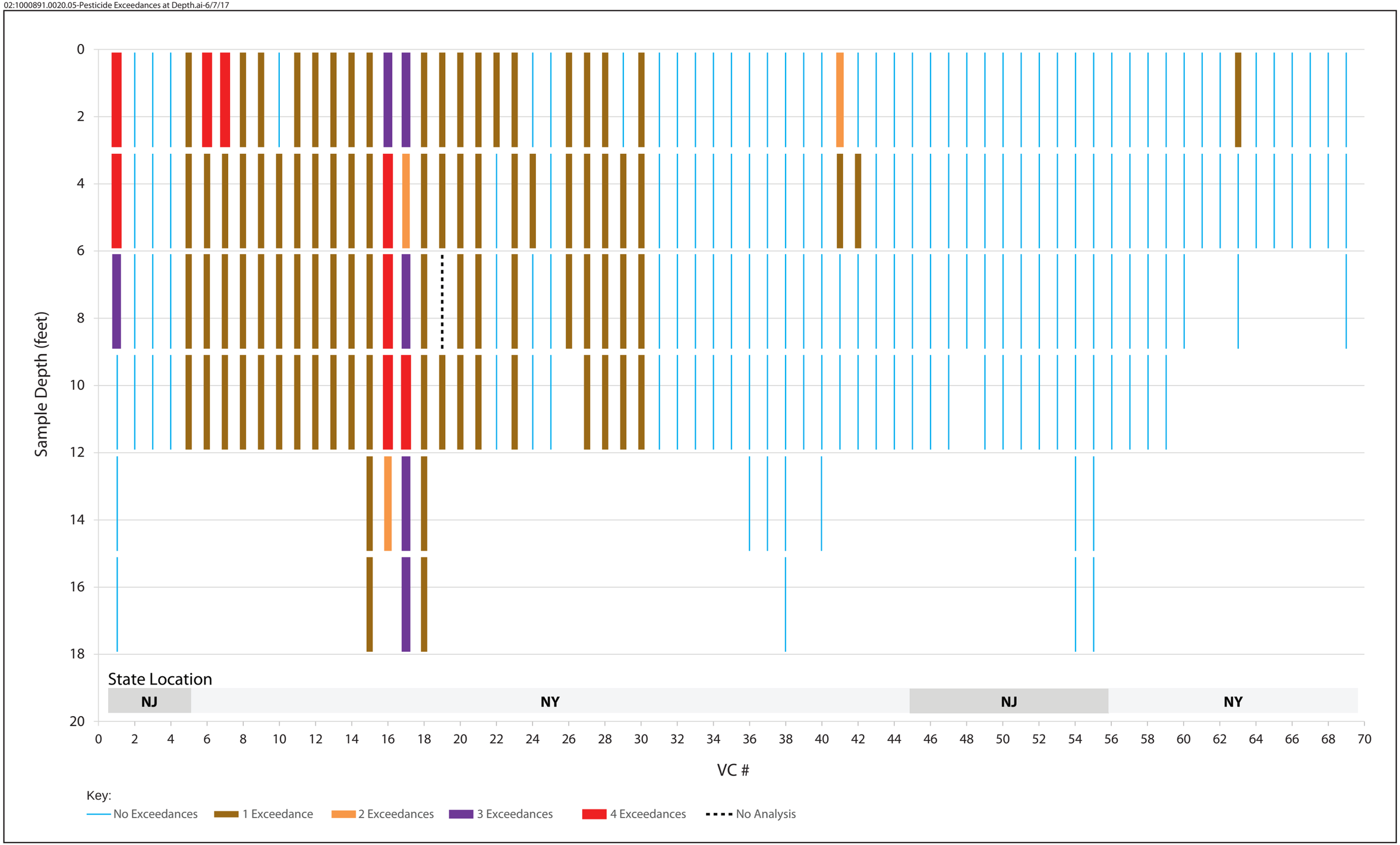
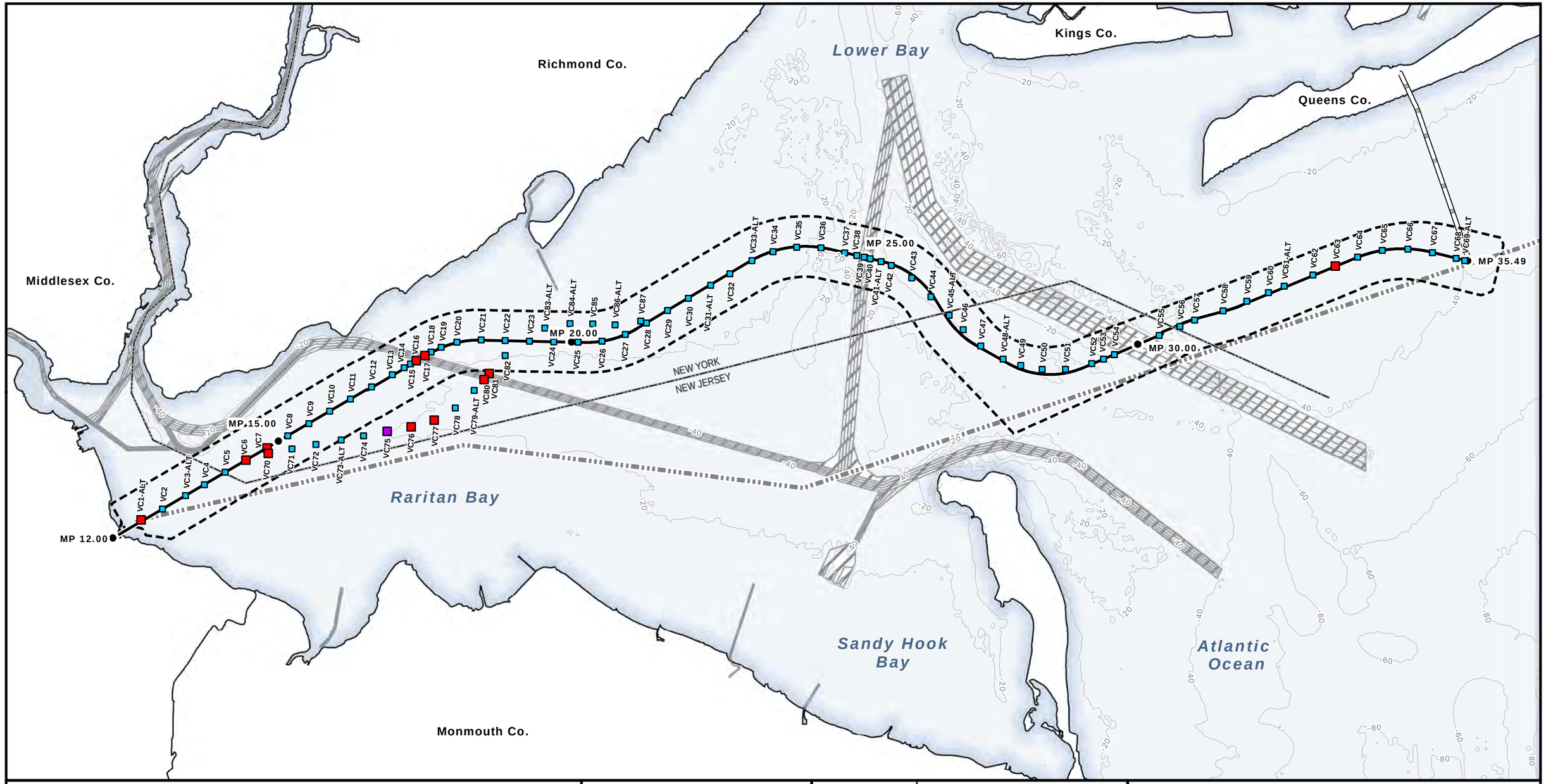


Figure A-7: Pesticide Exceedances at Depth Along the Preferred Route



Legend

PCB Aroclor Exceedances*

- 0
- 1
- 2 - 3

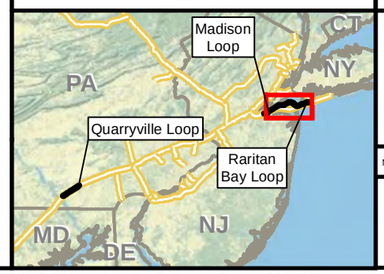
- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

*Number of analyte exceedances for lowest threshold of either the NJDEP Ecological Screening Criteria or NYSDEC Sediment Guidance Values.

N

0 1 Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE
	A-8

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE CONTAMINATION RESULTS
PCB AROCLORS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
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02:1000891.0020.05-Figure A-9 PCB Aroclor Exceedances at Depth.ai-5/18/17

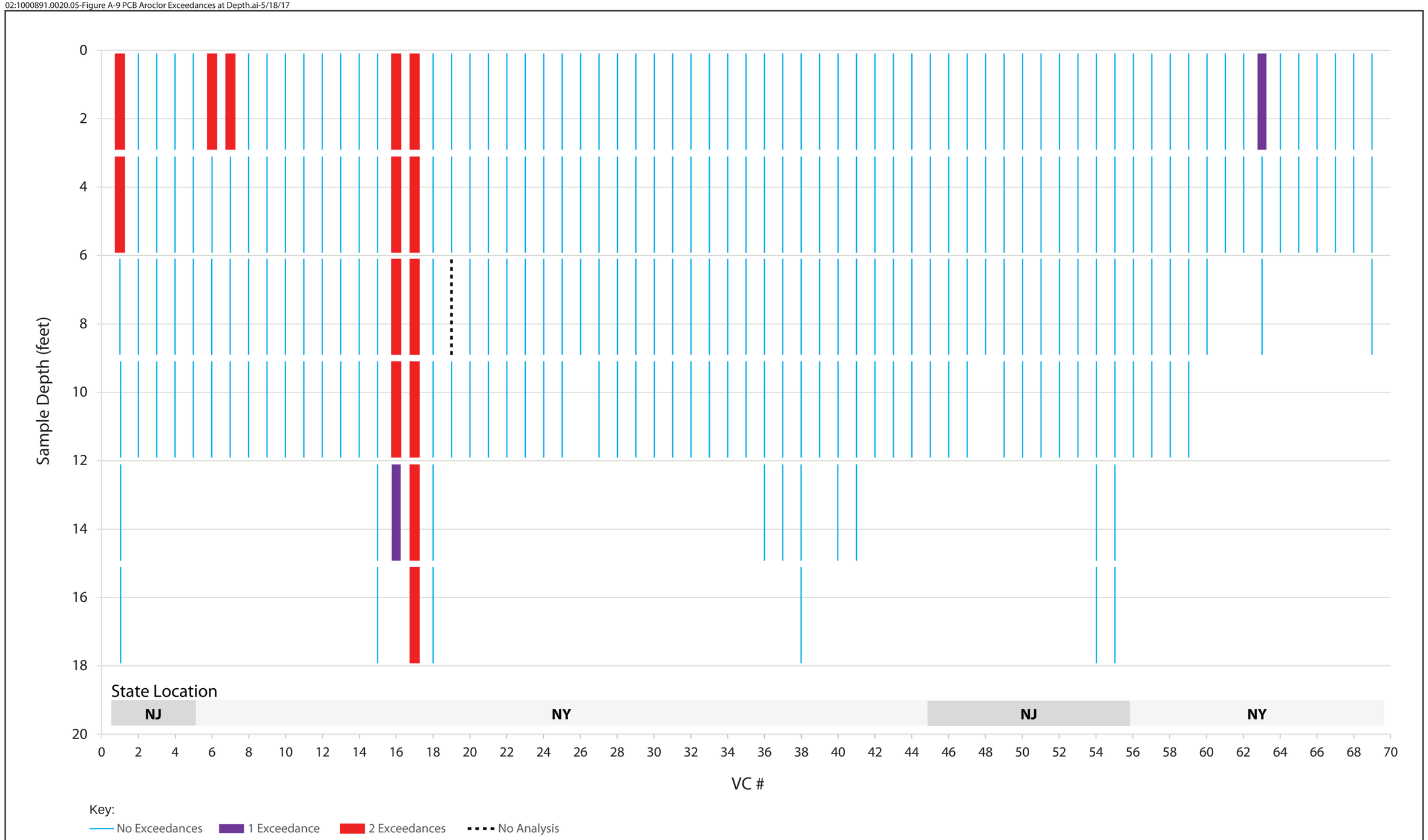
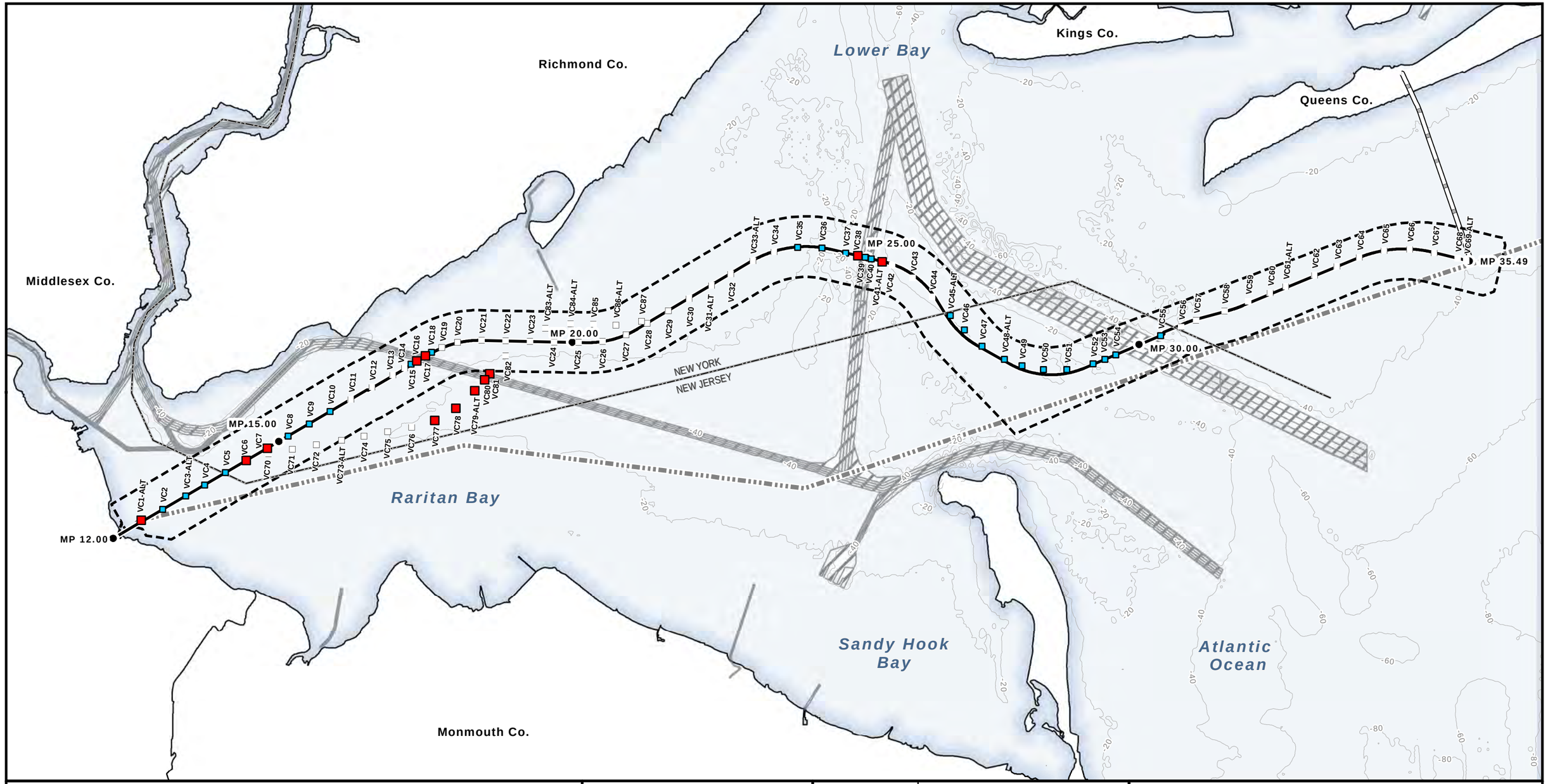


Figure A-9: PCB Aroclor Exceedances at Depth Along the Preferred Route



Legend

PCB Congener Exceedances*

- 0
- 1
- No Analysis

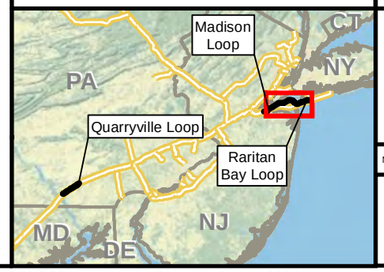
- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

*Number of analyte exceedances for lowest threshold of either the NJDEP Ecological Screening Criteria or NYSDEC Sediment Guidance Values.

N

0 1 Miles

SITE LOCATION



DRAWING NO.

REFERENCE TITLE

A-10

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE CONTAMINATION RESULTS
PCB CONGENERS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

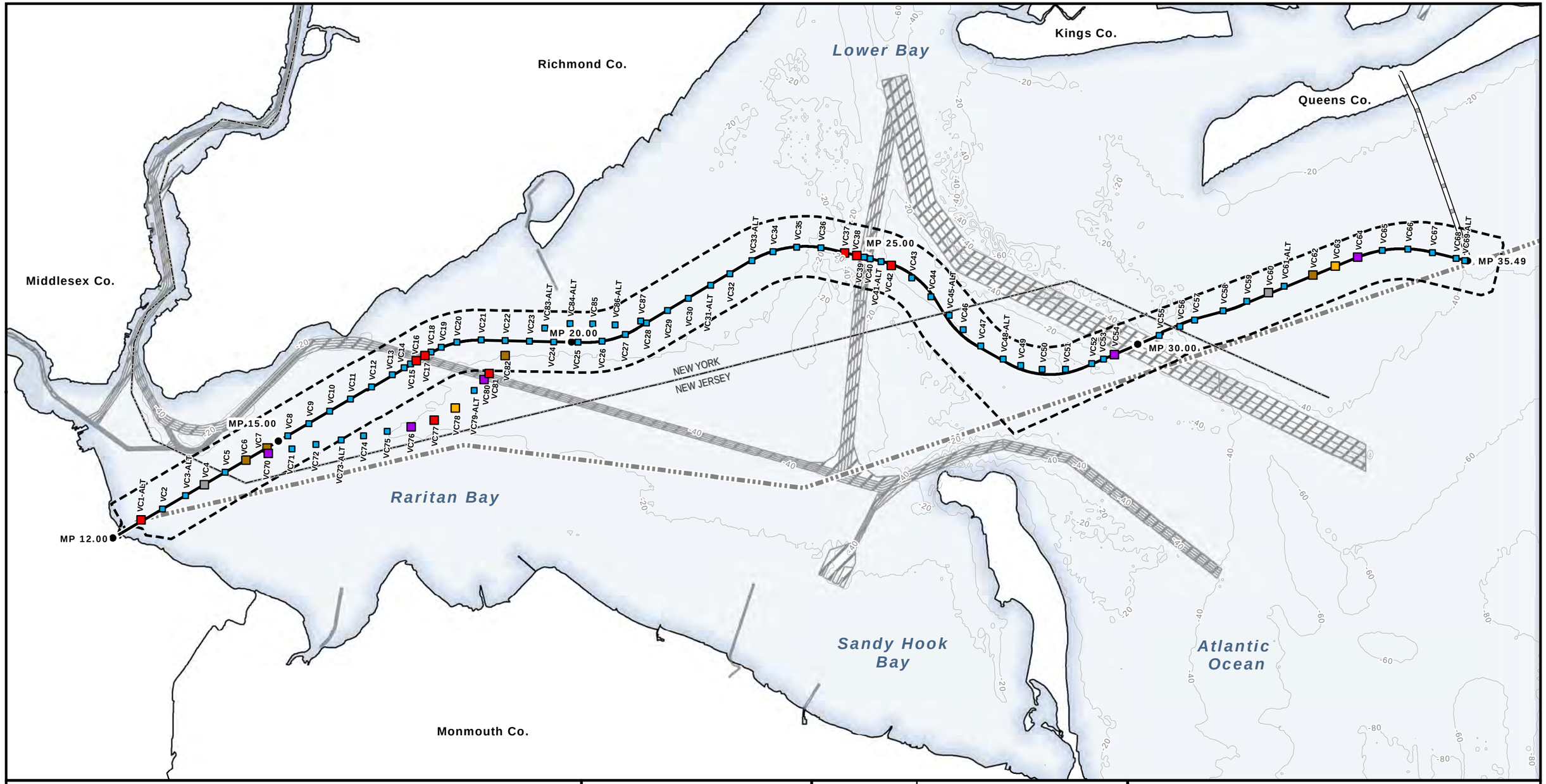
Williams

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A	6/7/2017	MK		1000891		

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CHECKED BY:	DATE:	ISSUE FOR CONSTRUCTION: N/A	Project features ver15
APPROVED BY:	DATE:	DRAWING NUMBER: Figure A-10	SHEET 1 OF 1
WO: 1000891		10:44 PM 6/7/2017	

Data Sources: Williams 2017; E&E 2017; ESRI 2012, 2017; NJDEP Digital Data Series Bathymetric Data 2006; NOAA ENC, Approach Scale, data downloaded 2016; USGS 2005; NYS Office of Information Technology Services GPO 2016.

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Legend

PAH Exceedances*

- 0
- 1 - 3
- 4 - 6
- 7 - 9
- 10 - 12
- 13 - 15
- 16 - 18

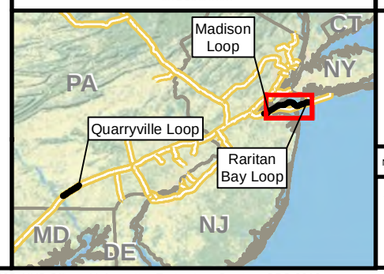
- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

*Number of analyte exceedances for lowest threshold of either the NJDEP Ecological Screening Criteria or NYSDEC Sediment Guidance Values.

N

0 1 Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE
	A-11

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE CONTAMINATION RESULTS
PAHS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
A	6/2/2017	MK		1000891		

DRAWN BY: MK	DATE: 6/2/2017	ISSUE FOR BID: N/A	SCALE: 1:95,000
CHECKED BY:	DATE:	ISSUE FOR CONSTRUCTION: N/A	Project features ver15
APPROVED BY:	DATE:	DRAWING NUMBER: Figure A-11	SHEET 1 OF 1
WO: 1000891		11:03 AM 6/2/2017	

Data Sources: Williams 2017; E&E 2017; ESRI 2012, 2017; NJDEP Digital Data Series Bathymetric Data 2006; NOAA ENC, Approach Scale, data downloaded 2016; USGS 2005; NYS Office of Information Technology Services GPO 2016.

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02:1000891.0020.05-Figure A-13 PAH Exceedances at Depth.ai-5/18/17

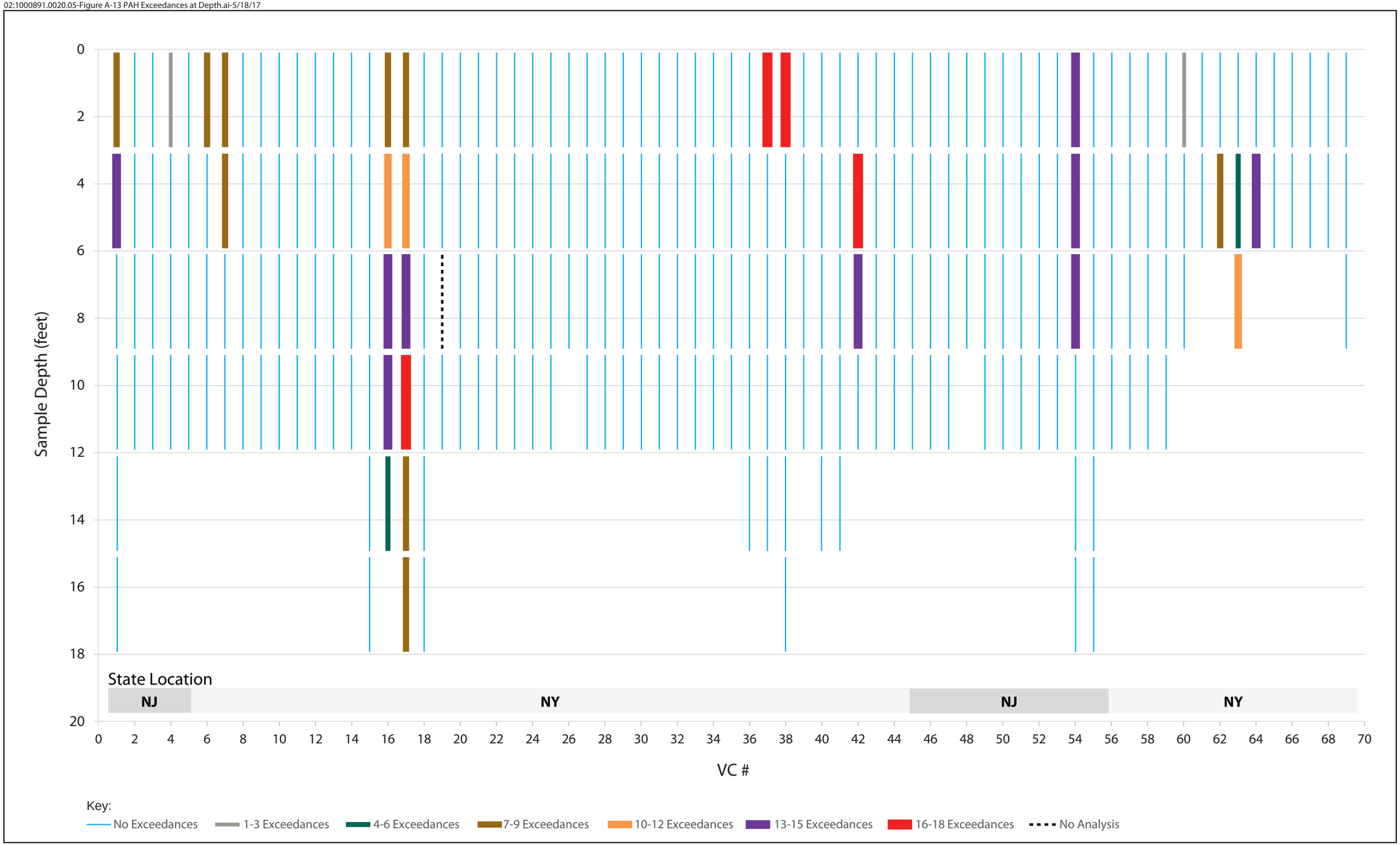
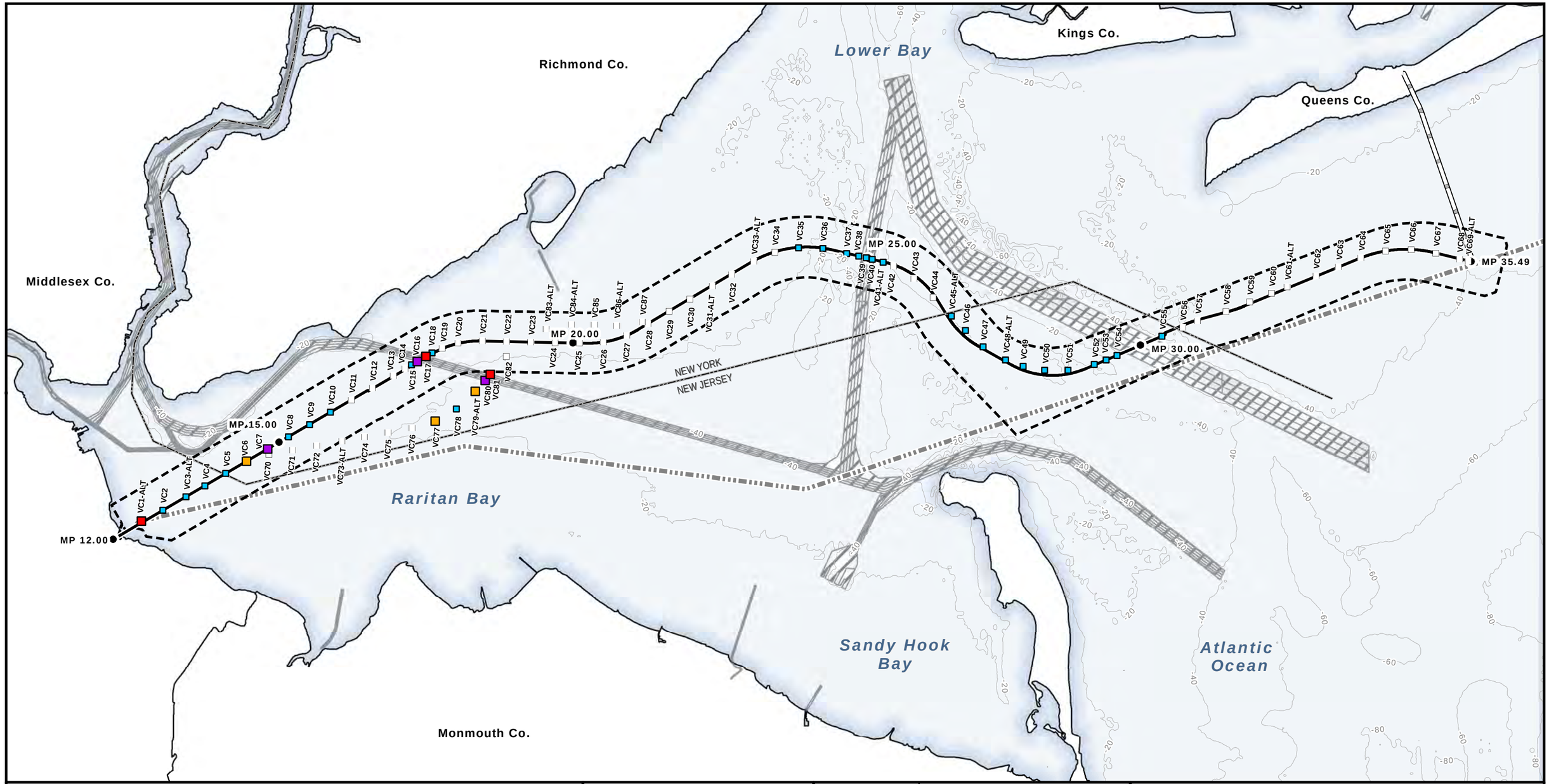


Figure A-12: PAH Exceedances at Depth Along the Preferred Route



Legend

Other SVOC Exceedances*

- 0
- 1
- 2 - 3
- 4
- No Analysis

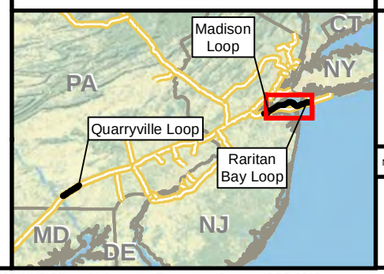
- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

*Number of analyte exceedances for lowest threshold of either the NJDEP Ecological Screening Criteria or NYSDEC Sediment Guidance Values.

N

0 1 Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE
	A-13

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE CONTAMINATION RESULTS
OTHER SVOCS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

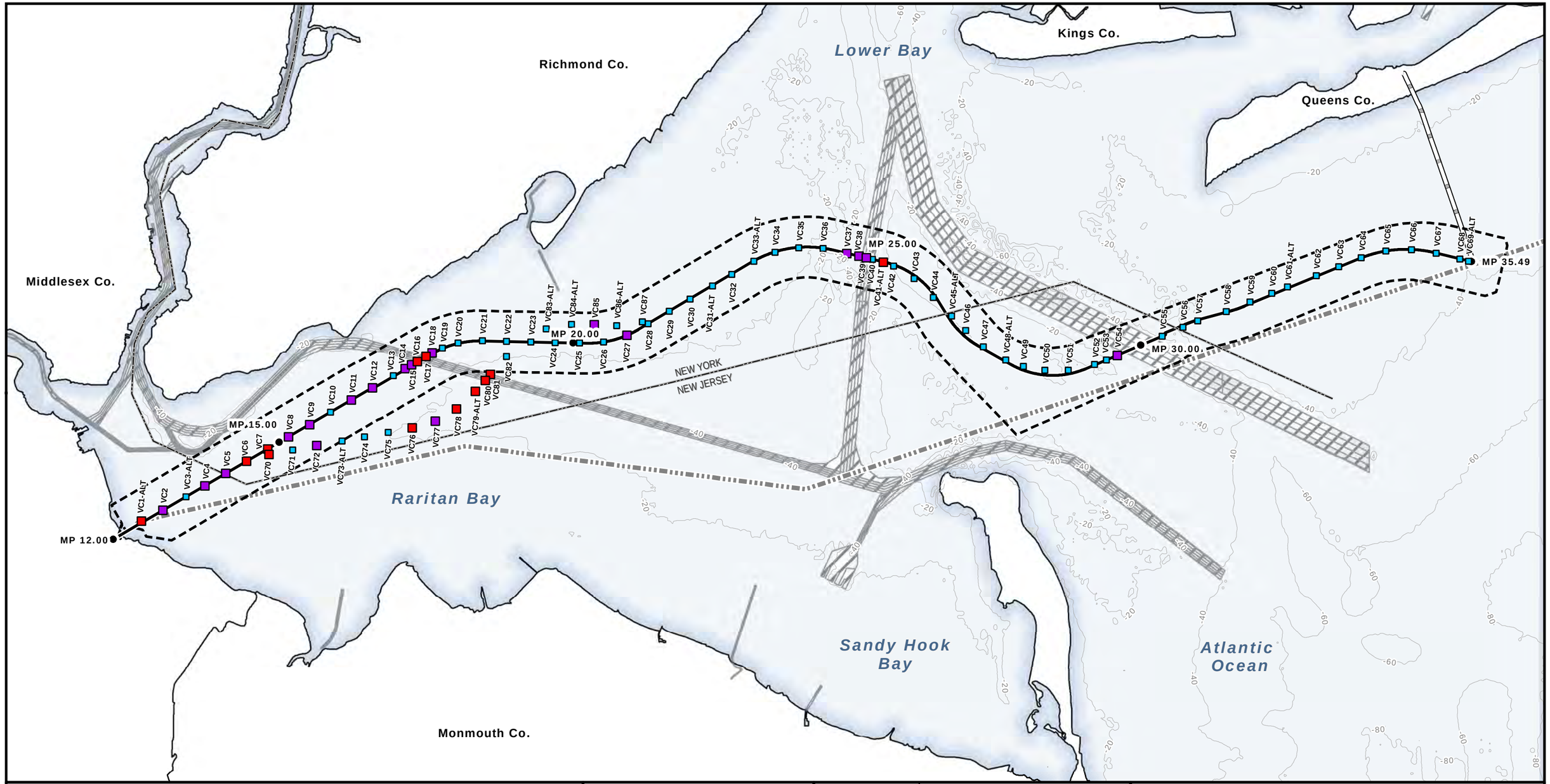
Williams

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
A	6/2/2017	MK		1000891		

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CHECKED BY:	DATE:	ISSUE FOR CONSTRUCTION: N/A	Project features ver15
APPROVED BY:	DATE:	DRAWING NUMBER: Figure A-13	SHEET 1 OF 1
WO: 1000891		11:21 AM 6/2/2017	

Data Sources: Williams 2017; E&E 2017; ESRI 2012, 2017; NJDEP Digital Data Series Bathymetric Data 2006; NOAA ENC, Approach Scale, data downloaded 2016; USGS 2005; NYS Office of Information Technology Services GPO 2016.

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Legend

Dioxin Furan Exceedances*

- 0
- 1
- 2

- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

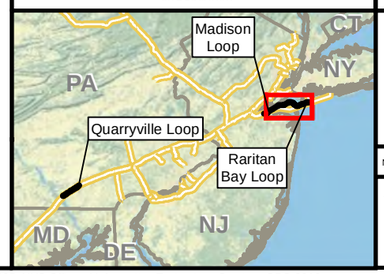
*Number of analyte exceedances for lowest threshold of either the NJDEP Ecological Screening Criteria or NYSDEC Sediment Guidance Values.

N

0 1

Miles

SITE LOCATION



DRAWING NO.

REFERENCE TITLE

A-14

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE CONTAMINATION RESULTS
DIOXINS AND FURANS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
A	6/2/2017	MK		1000891		

DRAWN BY: MK	DATE: 6/2/2017	ISSUE FOR BID: N/A	SCALE: 1:95,000
CHECKED BY:	DATE:	ISSUE FOR CONSTRUCTION: N/A	Project features ver15
APPROVED BY:	DATE:	DRAWING NUMBER: Figure A-14	SHEET 1 OF 1
WO: 1000891		11:17 AM 6/2/2017	

Data Sources: Williams 2017; E&E 2017; ESRI 2012, 2017; NJDEP Digital Data Series Bathymetric Data 2006; NOAA ENC, Approach Scale, data downloaded 2016; USGS 2005; NYS Office of Information Technology Services GPO 2016.

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02:1000891.0020.05-Figure A-16 Dioxin Furan Exceedances at Depth.ai-5/18/17

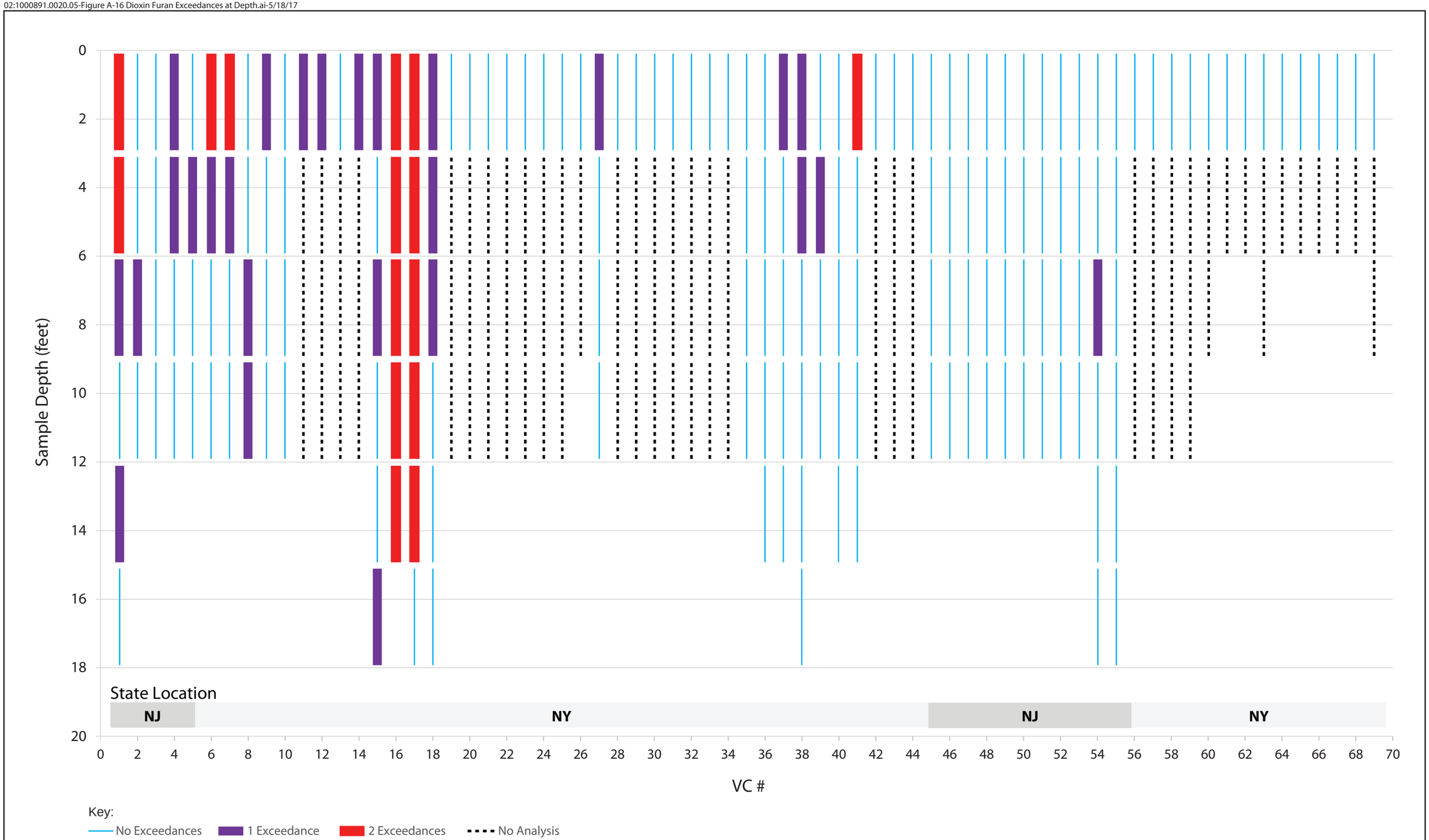


Figure A-15: Dioxin and Furan Exceedances at Depth Along the Preferred Route

02:1000891.0020.05-Metals Exceedances at Depth-Alternative Route.ai-6/7/17

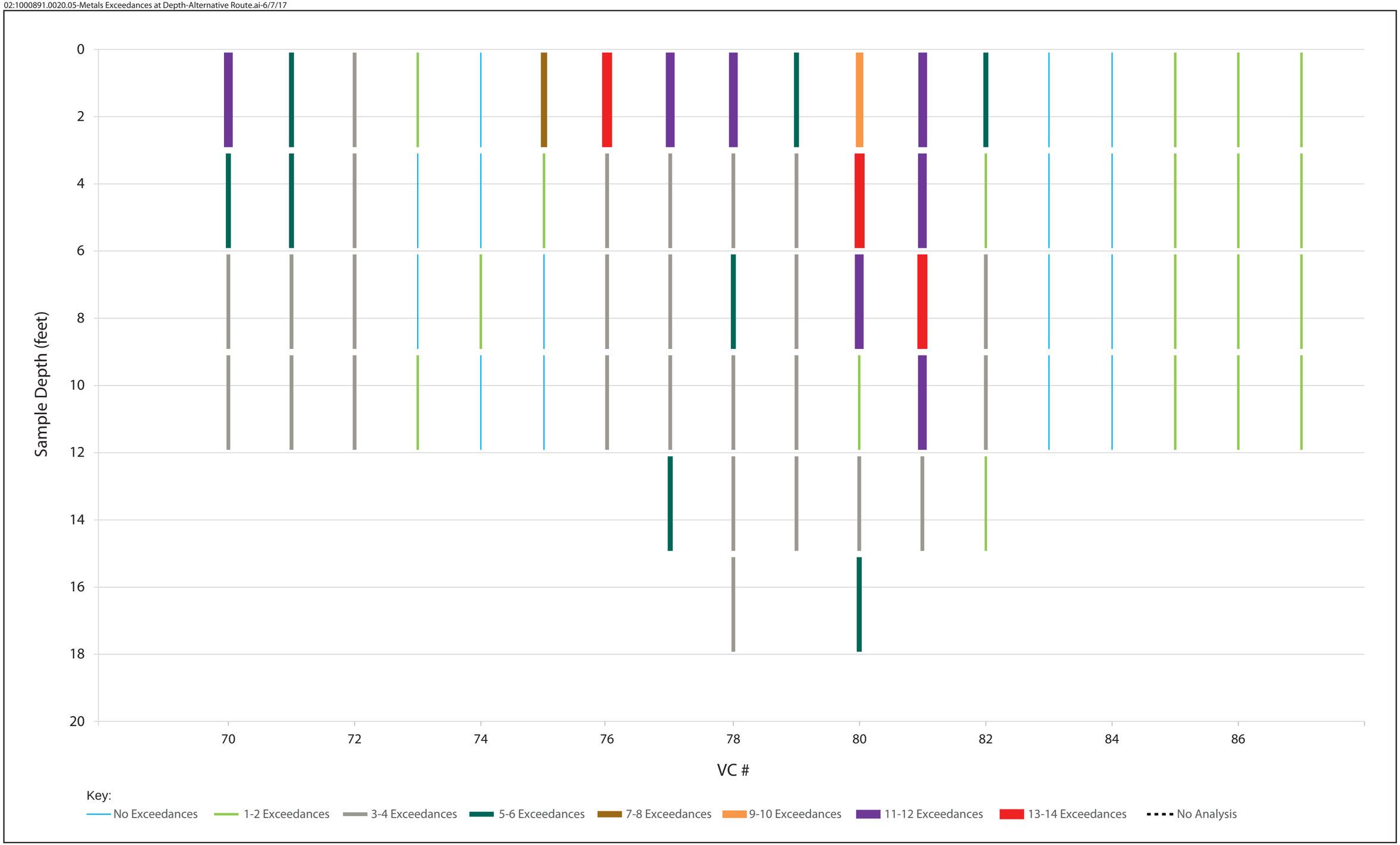


Figure A-16: Metals Exceedances at Depth Along the Alternative Route

02:1000891.0020.05-Pesticide Exceedances at Depth-Alternative Route.ai-6/7/17

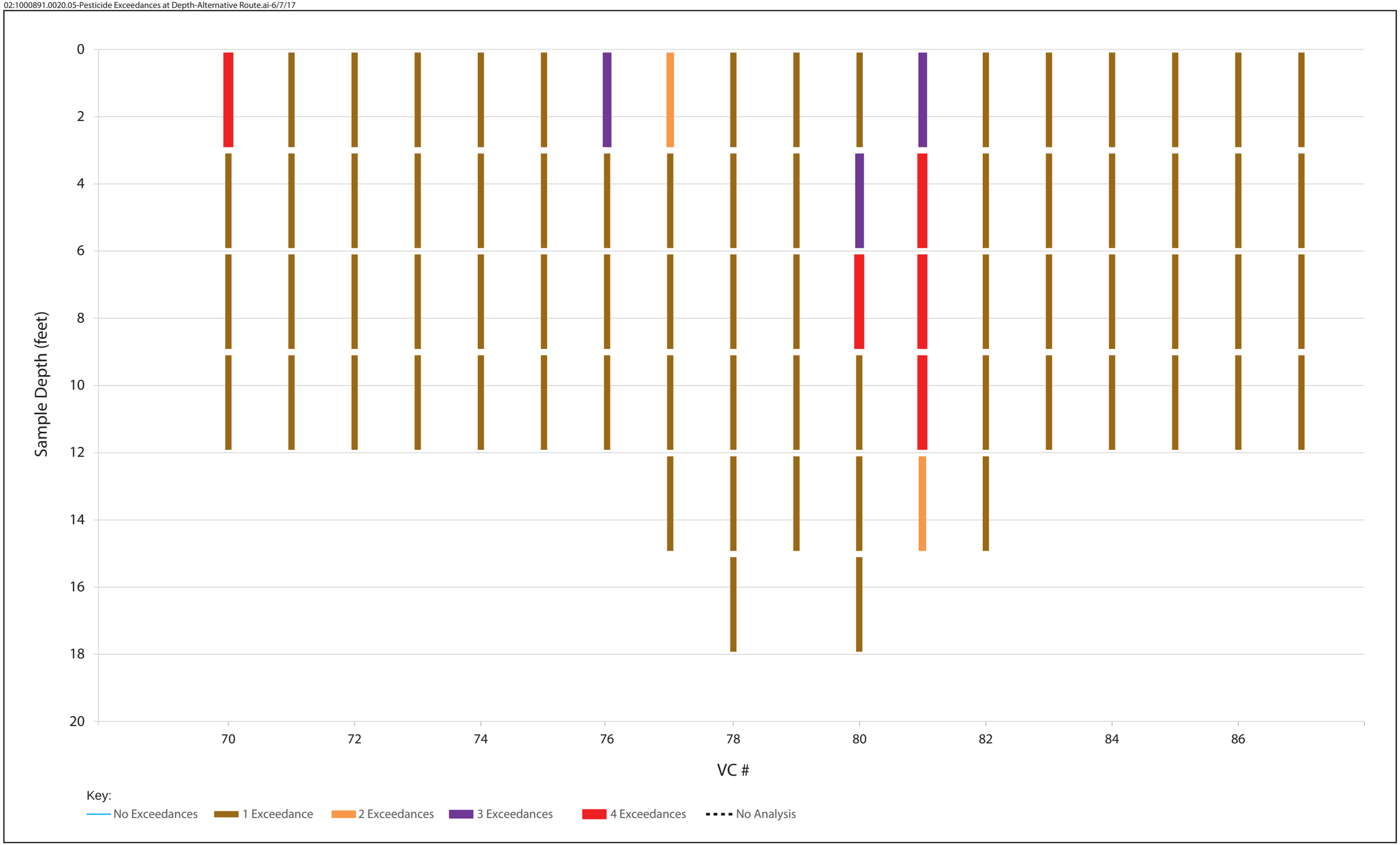


Figure A-17: Pesticide Exceedances at Depth Along the Alternative Route

02:1000891.0020.05-PCB Aroclor Exceedances at Depth-Alternative Route.ai-6/7/17

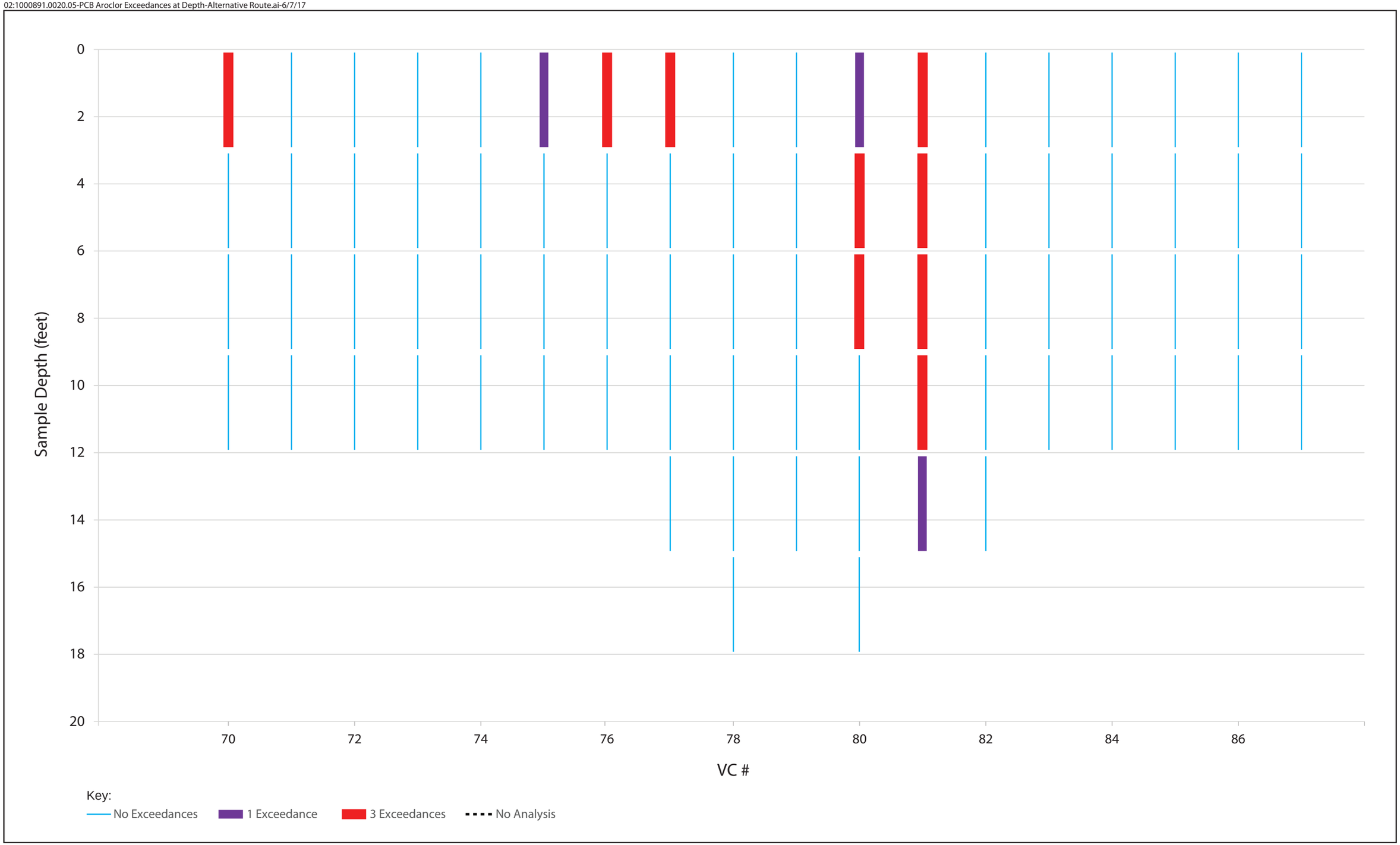


Figure A-18: PCB Aroclor Exceedances at Depth Along the Alternative Route

02:1000891.0020.05-PAH Exceedances at Depth-Alternative Route.ai-6/7/17

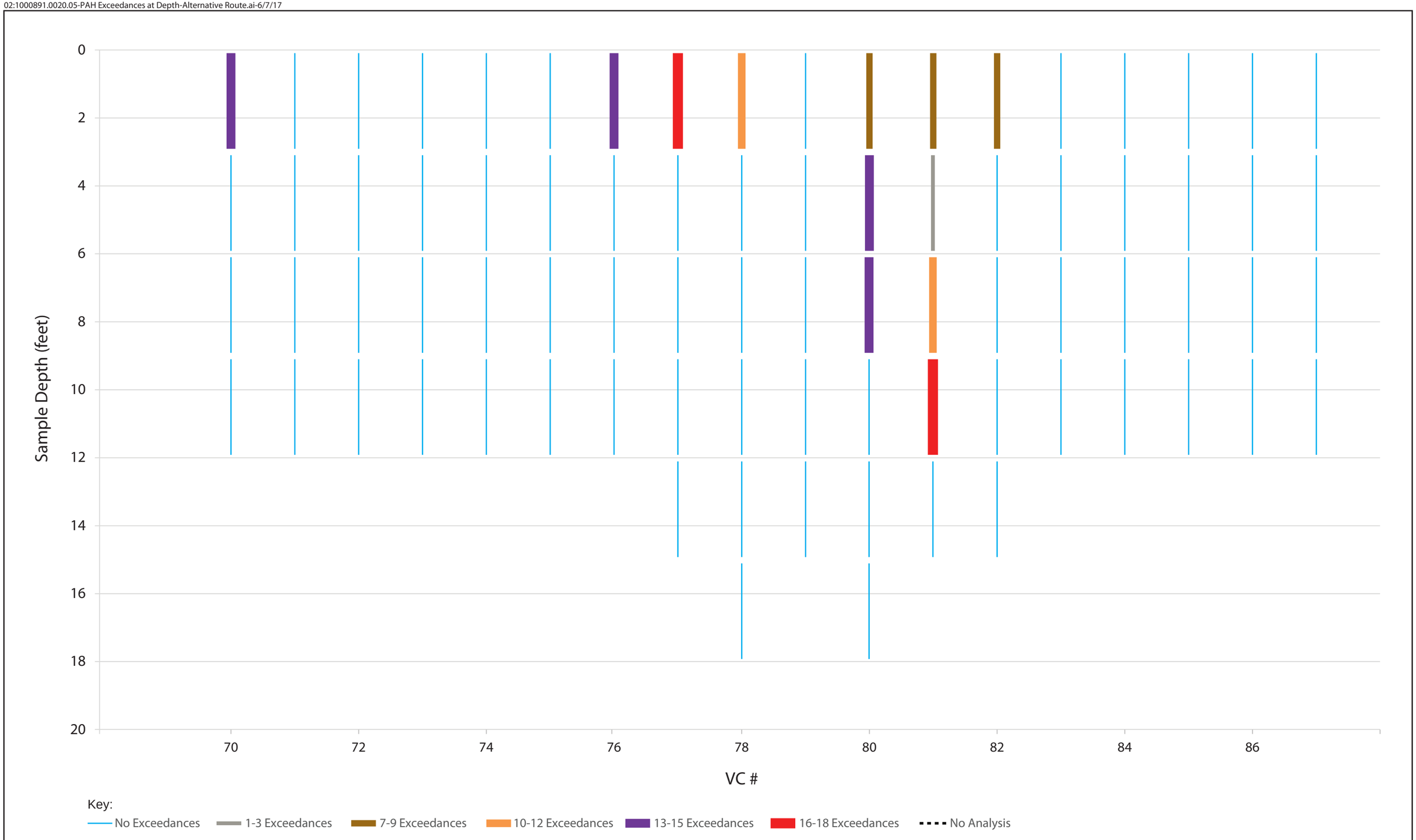


Figure A-19: PAH Exceedances at Depth Along the Alternative Route

02:1000891.0020.05-Dioxin Furan Exceedances at Depth-Alternative Route.ai-6/7/17

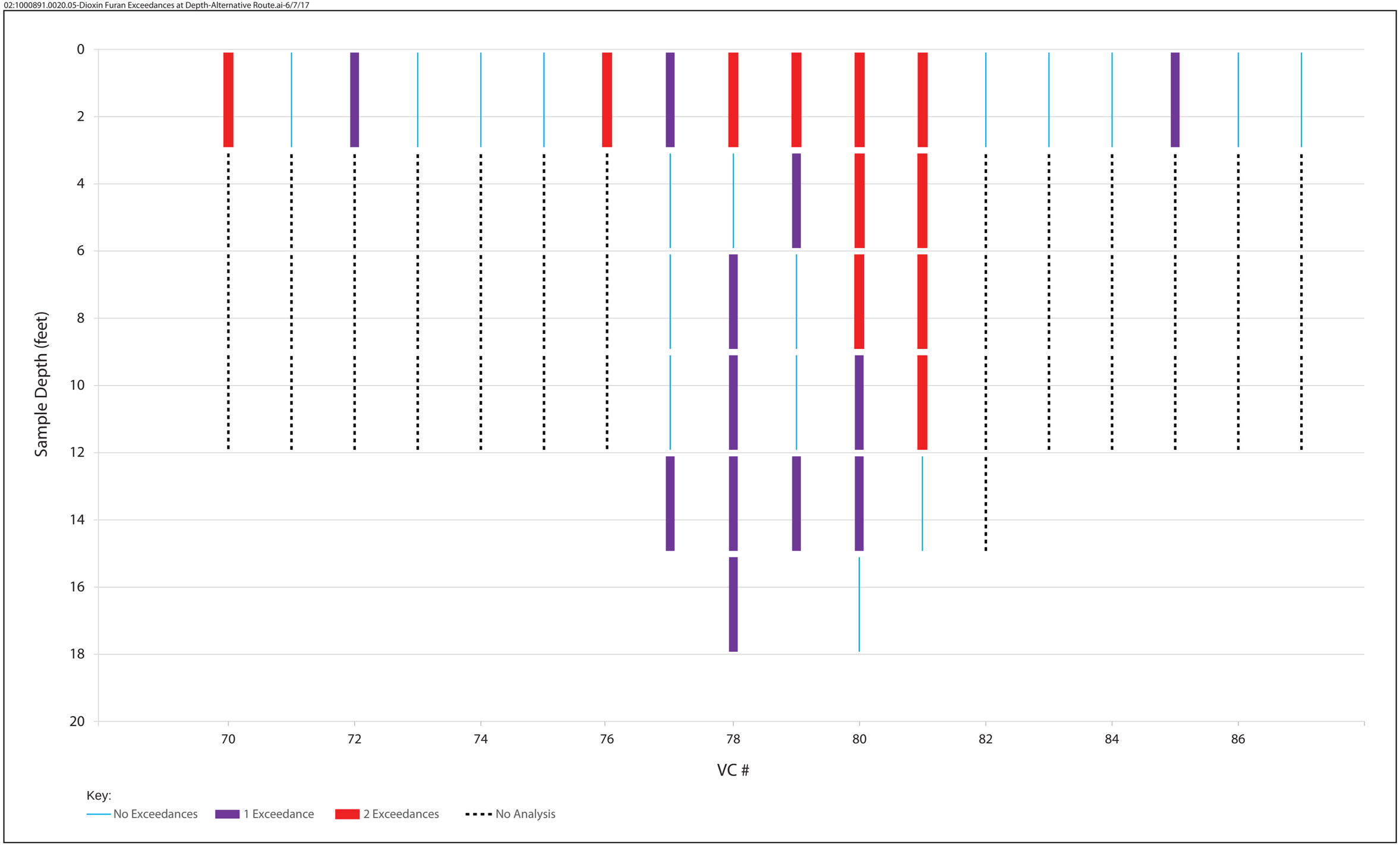
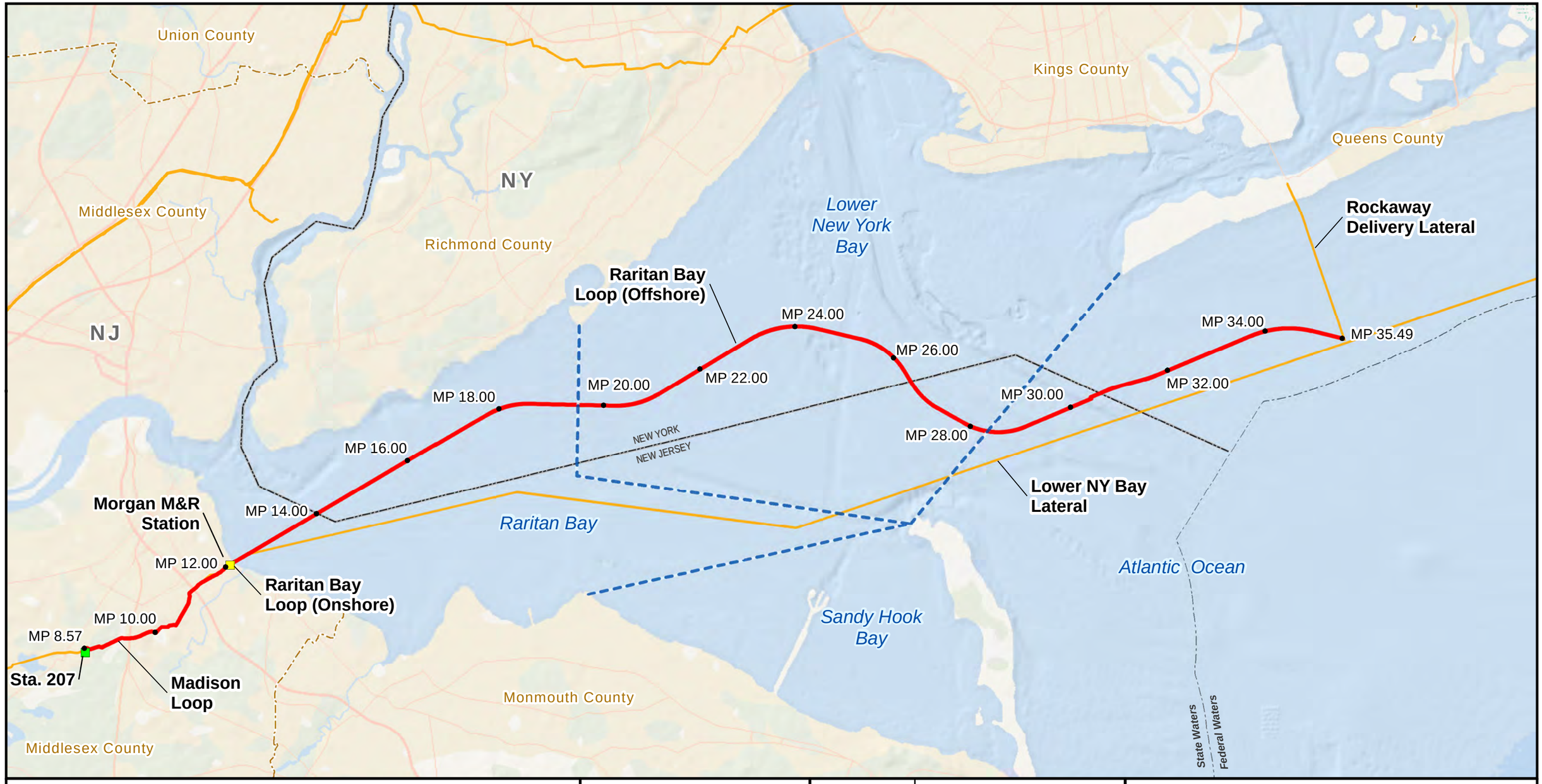


Figure A-20: Dioxin and Furan Exceedances at Depth Along the Alternative Route



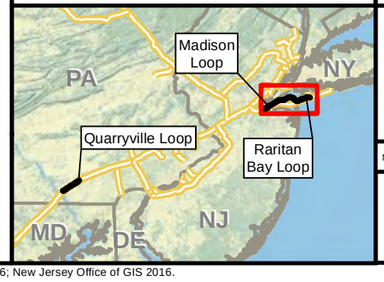
Legend

- Milepost
- Existing Compressor Station
- Existing Meter & Regulating Station
- Proposed Pipeline
- Existing Pipeline
- County Boundary
- NY/NJ State Boundary
- State/Federal Offshore Line
- Approximate Offshore Waterbody Boundary

N

0 1 2 Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE
	A-16

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE WATERBODIES
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
A	3/22/2017	DM	ISSUED FOR FERC FILING	1000891	MK	SM
B	6/1/2017	RH	ISSUED FOR FERC SUPPLEMENTAL FILING	1000891	MK	

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CHECKED BY: MK	DATE: 6/1/2017	ISSUE FOR CONSTRUCTION: N/A	Project features ver15
APPROVED BY:	DATE:	DRAWING NUMBER: FIGURE A-21	SHEET 1 OF 1
WO: 1000891		11:55 AM 6/1/2017	

Data Sources: BOEMRE 2010; BOEM 2005; E&E 2017; ESRI 2012, 2017; NYS Office of Information Technology Services GPO 2016; Williams 2017; U.S. Census Bureau 2016; New Jersey Office of GIS 2016.

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B Tables

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Table B-6	Sample Sites and Samples in Group A Exceeding ER-L and/or ER-M Thresholds for PAHs
Table B-7	Number of Detections at Sample Sites in Group B
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Table B-13	Physical Water Quality Sample Locations
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Table B-16	Benthic Biota Richness, Abundance, Diversity and Evenness at Alternative Route Stations
Table B-17	Benthic Macroinvertebrate Taxonomic List for Alternative Route

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Table B-1 Vibracore and Grab Sample Locations

Sample Site [a]	State	Coring Depth (ft)[d,e]	No. Off Samples Collected	Sample Site [a]	State	Coring Depth (ft)[d,e]	No. Off Samples Collected
VC1-ALT	***NJ***	***16***	***11[b]***	VC45-ALT	NJ	12	4
VC2	***NJ***	***10.5***	***4***	VC46	NJ	12	4
VC3-ALT	***NJ***	***12***	***4***	VC47	NJ	12	4
VC4	***NJ***	***12***	***4***	VC48-ALT	NJ	9.7	6[c]
VC5	***NY***	***12***	***4***	VC49	NJ	12	4
VC6	***NY***	***12***	***4***	VC50	NJ	12	4
VC7	***NY***	***12***	***4***	VC51	NJ	12	4
VC8	***NY***	***12***	***4***	VC52	NJ	12	4
VC9	***NY***	***12***	***4***	***VC53***	***NJ***	***12***	***4***
VC10	***NY***	***10.4***	***4***	***VC54***	***NJ***	***17.6***	***12[b]***
VC11	NY	12	4	***VC55***	***NJ***	***27.9***	***17[b]***
VC12	NY	12	4	VC56	NY	12	4
VC13	NY	12	4	VC57	NY	11.6	4
VC14	NY	12	4	VC58	NY	12	4
VC15	***NY***	***16.5***	***12[c]***	VC59	NY	12	4
VC16	***NY***	***15***	***5***	VC60	NY	8.7	3
VC17	***NY***	***17.5***	***6***	VC61-ALT	NY	5.1	2
VC18	***NY***	***16.2***	***6***	VC62	NY	5	2
VC19	NY	12	4	VC63	NY	7	3
VC20	NY	12	4	VC64	NY	6.1	3
VC21	NY	12	4	VC65	NY	4.8	4[c]
VC22	NY	12	4	VC66	NY	5.1	2
VC23	NY	12	4	VC67	NY	5.7	2
VC24	NY	10.5	4	VC68	NY	6.8	2
VC25	NY	11.5	4	VC69-ALT	NY	8.1	3
VC26	NY	8.7	3	VC70	NY	12	4
VC27	NY	12	8[c]	VC71	NY	12	4
VC28	NY	12	4	VC72	NY	12	4
VC29	NY	12	4	VC73-ALT	NY	12	4
VC30	NY	12	4	VC74	NY	12	4
VC31-ALT	NY	10.6	4	VC75	NY	12	4
VC32	NY	10.1	4	VC76	NY	12	4
VC33-ALT	NY	11.7	4	VC77	NY	15.6	5
VC34	NY	11	4	VC78	NY	17	6
VC35	***NY***	***10.9***	***4***	VC79-ALT	NY	16	5
VC36	***NY***	***15***	***5***	VC80	NY	16.5	6
VC37	***NY***	***14.6***	***5***	VC81	NY	14.75	5
VC38	***NY***	***17.6***	***6***	VC82	NY	16	5
VC39	***NY***	***12***	***4***	VC83-ALT	NY	10	4
VC40	***NY***	***12***	***5***	VC84-ALT	NY	12	4

Table B-1 Vibracore and Grab Sample Locations

Sample Site [a]	State	Coring Depth (ft)[d,e]	No. Off Samples Collected	Sample Site [a]	State	Coring Depth (ft)[d,e]	No. Off Samples Collected
VC41-ALT	***NY***	***14.6***	***5***	VC85	NY	12	8[c]
VC42	NY	12	4	VC86-ALT	NY	11	4
VC43	NY	12	4	VC87	NY	12	4
VC44	NY	12	4				

a = Site in bold and italics indicate locations where clamshell dredging might occur.
b = Two vibracores were collected at this site.
c = Duplicate samples at each depth were collected at this site.
d = Depths provided represent actual recovery at each sample site.
e = If two Vibracores or duplicate samples were taken, this depth represents the greatest recovery depth at which samples were collected.

Key:
ft = feet

Table B-2 Selection of Sediment Chemistry Tests for Analytes Detected that Exceeded State Thresholds

Test Description	EPA Method	Unit	NYSDEC Required Method Detection Limits	NJDEP or USACE Required Reporting Limits[1]	NJDEP Effects Range Low (ER-L)[2]	NJDEP Effects Range-Median (ER-M)[3]	NYSDEC Class A	NYSDEC Class C
Arsenic	EPA 6020A	mg/Kg	3^	1	8.2	70	8.2	70
Barium	EPA 6020A	mg/Kg	--	40	--	48	--	--
Cadmium	EPA 6020A	mg/Kg	1	1	1.2	9.6	1.2	9.6
Chromium	EPA 6020A	mg/Kg	--	1	81	370	81	370
Cobalt	EPA 6020A	mg/Kg	--	10	--	10	--	--
Copper	EPA 6020A	mg/Kg	5^	1	34	270	34	270
Lead	EPA 6020A	mg/Kg	2^	0.6	47	218	47	220
Manganese	EPA 6020A	mg/Kg	--	3	--	260	--	--
Mercury	EPA 7474	mg/Kg	0.2^	0.1	0.15	0.71	0.15	0.71
Nickel	EPA 6020A	mg/Kg	--	1	21	52	21	52
Selenium	EPA 6020A	mg/Kg	--	1	--	1	--	--
Silver	EPA 6020A	mg/Kg	--	1	1	3.7	1	3.7
Zinc	EPA 6020A	mg/Kg	--	4	150	410	150	410
Benzene	EPA 8260C	µg/kg	0.3	10	340	N/A	460	1400
Total PAHs (sum of Target Compound List PAH)	EPA 8270D	µg/kg	330	--	4000	45000	4000	45000
Sum of DDT+DDE+DDD	EPA 8081B	µg/kg	3.3	--	1.6	46	44	5700
PCBs (sum of Aroclors)	EPA 8082A	µg/kg	33	<100	23	180	100	1000
PCBs (sum of congeners)	EPA 1668C	µg/kg	--	--	23	180	100	1000
Dioxins/Furans - Total Toxicity Equivalency Factor	EPA 1613B	pg/g	2000	--	--	3.65	4.5	50

Key:
-- = Guidance value not available
^ = If the value found is less than the method detection limit, the laboratory reporting limit is listed.

Notes:
1. Unshaded cells present reporting limits from USACE/EPA *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal* (2014). Shaded Cells present reporting limits from *The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters* (1997). If both documents provide reporting limits, only the lowest is shown here.
2. NJDEP Ecological Screening Criteria (2009)
3. NYS TOGS 5.1.9 (2004) and NYS *Screening and Assessment of Contaminated Sediment* (2014).

Table B-3 Summary of Sediment Sample Analytical Results that Exceeded Available Thresholds for the Proposed Route

Analyte	No. of Sites (and Samples) Included in Analysis	No. of Sites (and Samples) Where Analyte was Detected	Maximum Result	Location of Maximum Result	No. of Exceedances (and Range of Values in Exceedance)[3]			
					NJDEP ER-L	NJDEP ER-M	NYSDEC Class A	NYSDEC Class C
Total BTEX (µg/kg)	69(314)	6(12)	9.81	VC16-D9-12E	N/A	N/A	0	0
Total PAHs (µg/kg)	69(313)	56(155)	21860.5	VC38-D0-3E	24(4230-21860.5)	0	24(4230-21860.5)	0
Dioxins and Furans Total Toxicity Equivalency Factor[1] (pg/g)	69(219)	66(202)	81.499	VC1-ALT-D0-3E	N/A	45(3.70104-81.499)	26(4.60107-81.499)	1(81.499-81.499)
P,P'-DDE (µg/kg)	69(313)	7(21)	56.5	VC1B-ALT-D3-6E	19(4.1-56.5)	N/A	0	N/A
Sum of DDT + DDE + DDD (µg/kg)	69(313)	7(21)	99.003	VC6-D0-3E	21(3.393-99.003)	N/A	0	0
Total PCB Congeners[2] (pg/g)	37(177)	28(135)	2000000	VC16-D9-12E	19(28800-2000000)	N/A	0	3(1160000-2000000)
Total PCB Aroclors (µg/kg)	69(313)	6(19)	1140	VC1B-ALT-D0-3E	19(93.9-1140)	0	0	1(1140-1140)
Aluminum (mg/kg)	69(314)	69(314)	18000	VC16-D0-3E	N/A	N/A	0	N/A
Antimony (mg/kg)	69(314)	8(23)	15.8	VC6-D0-3E	N/A	4(73.497-99.003)	4(8.672-99.003)	N/A
Arsenic (mg/kg)	69(314)	69(314)	108	VC1-ALT-D3-6E	86(8.27-108)	15(233000-2000000)	16(149000-2000000)	3(87.8-108)
Barium (mg/kg)	69(314)	68(300)	135	VC1B-ALT-D0-3E	N/A	17(239.8-1140)	18(161.6-1140)	N/A
Cadmium (mg/kg)	69(314)	43(146)	3.49	VC16-D6-9E	12(1.3-3.49)	0	N/A	0
Chromium, Total (mg/kg)	69(314)	65(296)	202	VC16-D6-9E	14(86.5-202)	1(15.8-15.8)	N/A	0
Cobalt (mg/kg)	69(314)	69(313)	16.3	VC16-D6-9E	N/A	3(87.8-108)	86(8.27-108)	N/A
Copper (mg/kg)	69(314)	65(281)	504	VC1B-ALT-D0-3E	22(37.3-504)	17(48.8-135)	N/A	2(307-504)
Lead (mg/kg)	69(314)	69(314)	285	VC16-D6-9E	24(47.4-285)	0	12(1.3-3.49)	4(235-285)
Manganese (mg/kg)	69(314)	69(314)	906	VC16-D6-9E	N/A	0	14(86.5-202)	N/A
Nickel (mg/kg)	69(314)	68(301)	53.5	VC16-D6-9E	86(21.4-53.5)	45(10.1-16.3)	N/A	1(53.5-53.5)
Selenium (mg/kg)	69(314)	46(167)	18.6	VC1-ALT-D3-6E	N/A	2(307-504)	22(37.3-504)	N/A
Silver (mg/kg)	69(314)	13(34)	8.52	VC16-D9-12E	18(1.06-8.52)	4(235-285)	24(47.4-285)	8(3.92-8.52)

Table B-3 Summary of Sediment Sample Analytical Results that Exceeded Available Thresholds for the Proposed Route

Analyte	No. of Sites (and Samples) Included in Analysis	No. of Sites (and Samples) Where Analyte was Detected	Maximum Result	Location of Maximum Result	No. of Exceedances (and Range of Values in Exceedance)[3]			
					NJDEP ER-L	NJDEP ER-M	NYSDEC Class A	NYSDEC Class C
Vanadium (mg/kg)	69(314)	64(279)	94.4	VC22-D9-12E	N/A	94(263-906)	N/A	N/A
Zinc (mg/kg)	69(314)	66(270)	494	VC16-D6-9E	18(154-494)	1(53.5-53.5)	86(21.4-53.5)	3(427-494)
Mercury (mg/kg)	69(314)	53(196)	5.28	VC1B-ALT-D0-3E	36(0.169-5.28)	156(1.01-18.6)	N/A	21(0.904-5.28)

Key:
DDD = dichloro-diphenyl-dichloroethane
DDE = dichloro-diphenyl-dichloroethylene
DDT = dichloro-diphenyl-trichloroethane
µg/kg = micrograms per kilogram
mg/kg = milligrams per kilogram
PCBs = polychlorinated biphenyls
PCBs = polychlorinated biphenyls
pg/g = picogram/gram

Notes:

1 Dioxins/Furans only analyzed in top 3 feet in NY non-clamshell.
2 Not analyzed in NY non-clamshell.
3 N/A indicates that there is no guidance value for that analyte, 0 indicates no exceedances of the guidance value. All measured concentrations of analytes detected in samples from New Jersey and New York were compared to both the SGVs and ESC.

Sources: NJDEP 2009, NYSDEC 2004, 2014c.

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Table B-4 Count of Exceedances in New York and New Jersey per Number of Available SGVs and ESCs

Analyte Group	VC1-ALT[1]		VC1B-ALT[1]		VC2		VC3-ALT		VC4		VC5		VC6		VC7		VC8		VC9		VC10		VC11[2]		VC12[2]	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	4/15	0/6	3/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	1/15	0/6	2/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/0	0/15	0/0
PAHs[3]	16/18	1/1	13/18	1/1	0/18	0/1	0/18	0/1	1/18	0/1	0/18	0/1	7/18	0/1	8/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1
Dioxins/ Furans[3]	1/1	2/2	1/1	2/2	1/1	1/2	0/1	0/2	1/1	1/2	1/1	0/2	1/1	2/2	1/1	2/2	1/1	1/2	1/1	0/2	0/1	0/2	1/1	0/2	1/1	0/2
Pesticides[3]	4/11	1/11	4/11	1/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	4/11	1/11	4/11	1/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/4	0/11	0/4
PCB Congener[3]	1/1	1/1	1/1	1/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	1/1	1/1	1/1	1/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/0	0/1	0/0
PCB Aroclor[3]	3/5	1/1	3/5	1/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	3/5	1/1	3/5	1/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	14/16	9/9	14/16	9/9	2/16	1/9	5/16	2/9	7/16	4/9	5/16	2/9	15/16	9/9	13/16	9/9	5/16	2/9	4/16	2/9	0/16	0/9	0/16	0/9	2/16	1/9
Total	**43/71**	**15/36**	**39/71**	**15/36**	**3/71**	**2/36**	**5/71**	**2/36**	**9/71**	**5/36**	**6/71**	**2/36**	**32/71**	**14/36**	**32/71**	**14/36**	**6/71**	**3/36**	**5/71**	**2/36**	**0/71**	**0/36**	**1/71**	**0/22**	**3/71**	**1/22**

Analyte Group	VC13[2]		VC14[2]		VC15		VC16		VC17		VC18		VC19[2]		VC20[2]		VC21[2]		VC22[2]		VC23[2]		VC24[2]		VC25[2]	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	0/15	0/0	0/15	0/0	0/15	0/6	3/15	0/6	4/15	0/6	0/15	0/6	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0
PAHs[3]	0/18	0/1	0/18	0/1	0/18	0/1	17/18	1/1	17/18	1/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1
Dioxins/ Furans[3]	0/1	0/2	1/1	0/2	1/1	1/2	1/1	2/2	1/1	2/2	1/1	1/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2
Pesticides[3]	0/11	0/4	0/11	0/4	0/11	0/11	4/11	1/11	4/11	1/11	0/11	0/11	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4
PCB Congener[3]	0/1	0/0	0/1	0/0	0/1	0/1	1/1	1/1	1/1	1/1	0/1	0/1	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0
PCB Aroclor[3]	0/5	0/1	0/5	0/1	0/5	0/1	3/5	1/1	3/5	1/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	3/16	2/9	3/16	2/9	4/16	2/9	14/16	9/9	13/16	9/9	4/16	2/9	4/16	2/9	4/16	2/9	0/16	0/9	3/16	1/9	3/16	2/9	0/16	0/9	1/16	1/9
Total	**3/71**	**2/22**	**4/71**	**2/22**	**5/71**	**3/36**	**43/71**	**15/36**	**43/71**	**15/36**	**5/71**	**3/36**	**4/71**	**2/22**	**4/71**	**2/22**	**0/71**	**0/22**	**3/71**	**1/22**	**3/71**	**2/22**	**0/71**	**0/22**	**1/71**	**1/22**

Analyte Group	VC26[2]		VC27[2]		VC28[2]		VC29[2]		VC30[2]		VC31-ALT[2]		VC32[2]		VC33-ALT[2]		VC34[2]		VC35		VC36		VC37		VC38	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6
PAHs[3]	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	17/18	1/1	18/18	1/1
Dioxins/ Furans[3]	0/1	0/2	1/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	1/1	1/2	1/1	0/2
Pesticides[3]	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11
PCB Congener[3]	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/1	0/1	0/1	0/1	0/1	1/1	0/1
PCB Aroclor[3]	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	1/16	0/9	2/16	1/9	1/16	0/9	3/16	1/9	1/16	0/9	2/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9	5/16	4/9	11/16	7/9
Total	**1/71**	**0/22**	**3/71**	**1/22**	**1/71**	**0/22**	**3/71**	**1/22**	**1/71**	**0/22**	**2/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/36**	**1/71**	**0/36**	**23/71**	**6/36**	**31/71**	**8/36**

Analyte Group	VC39		VC40		VC41-ALT		VC42[2]		VC43[2]		VC44[2]		VC45-ALT		VC46		VC47		VC48-ALT		VC49		VC50		VC51	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6
PAHs[3]	0/18	0/1	0/18	0/1	0/18	0/1	17/18	1/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1
Dioxins/ Furans[3]	1/1	1/2	0/1	0/2	1/1	1/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2
Pesticides[3]	0/11	0/11	0/11	0/11	2/11	1/11	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11
PCB Congener[3]	0/1	0/1	0/1	0/1	1/1	0/1	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1
PCB Aroclor[3]	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	1/16	0/9	1/16	0/9	2/16	1/9	8/16	6/9	1/16	0/9	0/16	0/9	0/16	0/9	1/16	0/9	3/16	2/9	1/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9
Total	**2/71**	**1/36**	**1/71**	**0/36**	**6/71**	**3/36**	**25/71**	**7/22**	**1/71**	**0/22**	**0/71**	**0/22**	**0/71**	**0/36**	**1/71**	**0/36**	**3/71**	**2/36**	**1/71**	**0/36**	**1/71**	**0/36**	**1/71**	**0/36**	**1/71**	**0/36**

Table B-4 Count of Exceedances in New York and New Jersey per Number of Available SGVs and ESCs

Analyte Group	VC52		VC53		VC54[1]		VC54B[1]		VC55[1]		VC55B[1]		VC56[2]		VC57[2]		VC58[2]		VC59[2]		VC60[2]		VC61-ALT[2]		VC62[2]	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0
PAHs[3]	0/18	0/1	0/18	0/1	15/18	1/1	15/18	1/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	1/18	1/1	0/18	0/1	9/18	1/1
Dioxins/ Furans[3]	0/1	0/2	0/1	0/2	1/1	1/2	1/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2
Pesticides[3]	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4
PCB Congener[3]	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0
PCB Aroclor[3]	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	1/16	0/9	1/16	0/9	2/16	1/9	2/16	1/9	1/16	0/9	0/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9	5/16	4/9	2/16	1/9	2/16	2/9
Total	**1/71**	**0/36**	**1/71**	**0/36**	**18/71**	**3/36**	**18/71**	**2/36**	**1/71**	**0/36**	**0/71**	**0/36**	**1/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/22**	**6/71**	**5/22**	**2/71**	**1/22**	**11/71**	**3/22**

Analyte Group	VC63[2]		VC64[2]		VC65[2]		VC66[2]		VC67[2]		VC68[2]		VC69-ALT[2]	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0
PAHs[3]	11/18	1/1	15/18	1/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1
Dioxins/ Furans[3]	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2
Pesticides[3]	1/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4
PCB Congener[3]	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0
PCB Aroclor[3]	2/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	0/16	0/9	1/16	1/9	1/16	1/9	0/16	0/9	0/16	0/9	1/16	0/9	1/16	0/9
Total	**14/71**	**1/22**	**16/71**	**2/22**	**1/71**	**1/22**	**0/71**	**0/22**	**0/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/22**

Key

[1] Both samples of each set (VC1-ALT/VC1B-ALT, VC54/VC54B, and VC55/VC55B) were collected in the same location.

[2] Dioxins and furans were tested for only in the samples taken from 0-3 feet at sites in New York where clamshell dredging is not anticipated to occur. Dioxin/furan samples for additional depths at these sites were archived. PCB Congeners were not tested for at sites in New York where clamshell dredging is not anticipated to occur.

[3] Highlighted cells indicate that ER-M or Class C thresholds were also exceeded.

Table B-5 Sample Sites and Samples in Group A Exceeding ER-L and/or ER-M Thresholds for Metals

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded ER-L (mg/kg)	Exceeded ER-M (mg/kg)		
Arsenic	8.37-108	N/A	VC1-ALT	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16 ft
			VC1B-ALT	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft
			VC2	0-3 ft, 6-9 ft
			VC3-ALT	0-3 ft, 3-6 ft, 6-9 ft, 9-10.5 ft
			VC4	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
Barium	--	82.5-135	VC1-ALT	***0-3 ft, 3-6 ft***
			VC1B-ALT	***0-3 ft, 3-6 ft***
Cadmium	1.62-2.36	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Chromium	97.6-145	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Cobalt	--	10.1-11.6	VC1-ALT	***3-6 ft, 12-15 ft, 15-16 ft***
			VC1B-ALT	***12-15 ft***
			VC3-ALT	***3-6 ft, 6-9 ft, 9-10.5 ft***
			VC4	***0-3 ft, 3-6 ft***
Copper	237-504	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Lead	47.4-235	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
			VC4	0-3 ft
Manganese	--	274-436	VC1-ALT	***3-6 ft, 6-9 ft, 9-12 ft***
			VC1B-ALT	***3-6 ft, 9-12 ft, 12-15 ft***
			VC3-ALT	***0-3 ft, 3-6 ft, 6-9 ft, 9-10.5 ft***
			VC4	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft***
Nickel	24.3-40.7	N/A	VC1-ALT	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16 ft
			VC1B-ALT	***0-3 ft, 3-6 ft, 9-12 ft, 12-15 ft***
			VC3-ALT	0-3 ft, 3-6 ft, 6-9 ft, 9-10.5 ft
			VC4	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
Selenium	--	1.22-18.6	VC1-ALT	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16 ft***
			VC1B-ALT	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft***
			VC2	***0-3 ft***
			VC3-ALT	***0-3 ft, 3-6 ft, 6-9 ft, 9-10.5 ft***
			VC4	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft***
Silver	3.06-4.58	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Vanadium	--	70.4-83.3	VC1-ALT	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16 ft***
			VC1B-ALT	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft***

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Table B-5 Sample Sites and Samples in Group A Exceeding ER-L and/or ER-M Thresholds for Metals

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded ER-L (mg/kg)	Exceeded ER-M (mg/kg)		
Zinc	227-336	N/A	VC1-ALT	***03- ft, 3-6 ft***
			VC1B-ALT	***03- ft, 3-6 ft***
Mercury	0.294-0.32	2.53-5.28	VC1-ALT	***0-3 ft, 3-6 ft***
			VC1B-ALT	***0-3 ft, 3-6 ft,*** 6-9 ft
			VC4	0-3 ft

Key:
-- = ER-L thresholds were not available.
N/A = Detected concentrations did not exceed the ER-M thresholds.

* Sample depths where ER-M thresholds were exceeded are in bold and italics.

Table B-6 Sample Sites and Samples in Group A Exceeding ER-L and/or ER-M Thresholds for PAHs

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)
	Exceeded ER-L (µg/kg)	Exceeded ER-M (µg/kg)		
2-Methylnaphthalene	80-144	N/A	VC1-ALT	3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Acenaphthene	26.1-61.2	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	3-6 ft
Acenaphthylene	73.4-83.1	N/A	VC1-ALT	3-6 ft
			VC1B-ALT	3-6 ft
Anthracene	89.5-156	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Benzo(A)Anthracene	304-643	N/A	VC1-ALT	3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Benzo(A)Pyrene	567	N/A	VC1-ALT	3-6 ft
Benzo(G,H,I)Perylene	200-477	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Chrysene	600	N/A	VC1-ALT	3-6 ft
Dibenz(A,H)Anthracene	93-116	N/A	VC1-ALT	3-6 ft
			VC1B-ALT	3-6 ft
Fluoranthene	691-903	N/A	VC1-ALT	3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Fluorene	21-138	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
			VC4	0-3 ft
Indeno(1,2,3-C,D)Pyrene	331-385	N/A	VC1-ALT	3-6 ft
			VC1B-ALT	3-6 ft
Naphthalene	174	N/A	VC1-ALT	3-6 ft
Phenanthrene	280-510	N/A	VC1-ALT	3-6 ft
			VC1B-ALT	3-6 ft
Pyrene	785-1110	N/A	VC1-ALT	3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Total PAHs	4230-7000	N/A	VC1-ALT	3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft

Key:
N/A = Detected concentrations did not exceed the ER-M thresholds.

Table B-7 Number of Detections at Sample Sites in Group B

Sample Site	Number of Detections							
	VOCs	PAHs	Other SVOCs	Dioxins/ Furans	Pesticides	PCB Congener	PCB Aroclor	Metals
VC5	0	7	2	2	0	24	0	26
VC6	0	17	7	14	5	30	2	27
VC7	0	17	9	14	5	29	2	27
VC8	0	0	2	4	0	5	0	25
VC9	0	2	1	2	0	13	0	23
VC10	0	7	1	1	0	2	0	21
VC11[1]	0	14	0	2	0	0	0	23
VC12[1]	0	16	0	2	0	0	0	25
VC13	0	17	0	2	0	0	0	24
VC14[1]	0	15	0	2	0	0	0	23
VC15	0	12	2	3	0	14	0	24
VC16	5	17	6	16	5	30	2	26
VC17	1	17	7	16	7	30	2	26
VC18	0	11	2	3	0	20	0	24
VC19[1]	0	16	0	2	0	0	0	24
VC20[1]	0	17	0	2	0	0	0	24
VC21[1]	0	15	0	2	0	0	0	23
VC22[1]	0	15	0	3	0	0	0	23
VC23[1]	0	14	0	3	0	0	0	24
VC24[1]	0	15	0	2	0	0	0	24
VC25[1]	0	18	0	1	0	0	0	22
VC26	0	1	0	2	0	**0**	0	23
VC27[1]	0	6	0	2	0	**0**	0	25
VC28[1]	0	10	0	2	0	**0**	0	24
VC29[1]	0	4	0	2	0	**0**	0	24
VC30[1]	0	5	0	1	0	**0**	0	23
VC31-ALT[1]	0	1	0	1	0	**0**	0	24
VC32[1]	0	0	0	2	0	**0**	0	23
VC33-ALT[1]	0	1	0	1	0	**0**	0	23
VC34[1]	0	0	0	2	0	**0**	0	23
VC35	0	0	0	2	0	**3**	0	24
VC36	0	11	0	2	0	**4**	0	26
VC37	0	17	5	5	0	**14**	0	27
VC38	0	17	4	5	2	**28**	0	27
VC39	NY	9	1	2	0	**24**	0	26
VC40	NY	1	1	3	0	**2**	0	25
VC41-ALT	NY	17	3	6	3	**27**	0	25
VC42[1]	NY	18	0	3	0	**0**	0	25
VC43[1]	NY	3	0	1	0	**0**	0	19
VC44[1]	NY	3	0	1	0	**0**	0	19

Key:

1 - Dioxins and furans were only analyzed in the samples taken from 0-3 feet at sites in New York where clamshell dredging is not anticipated to occur. Dioxin/furan samples for additional depths at these sites were archived. PCB Congeners were not tested at sites in New York where clamshell dredging is not anticipated to occur.

Table B-8 Sample Sites and Samples in Group B Exceeding Class A and/or Class C Thresholds for Metals

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded Class A (mg/kg)	Exceeded Class C (mg/kg)		
Arsenic	8.61-37.54	87.8	VC5	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC6	***0-3 ft***, 3-6ft, 6-9 ft, 9-12 ft
			VC7	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC8	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC9	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC12	0-3 ft
			VC13	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC14	3-6 ft, 6-9 ft
			VC15	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16.5 ft
			VC16	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft
			VC17	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-17.5 ft
			VC18	3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16.2 ft
			VC19	3-6 ft, 6-9 ft, 9-12 ft
			VC20	6-9 ft, 9-12 ft
			VC22	9-12 ft
			VC23	9-12 ft
			VC25	9-11.5 ft
			VC37	0-3 ft
			VC38	0-3 ft
			VC42	3-6 ft
Cadmium	1.3-3.5	N/A	VC6	0-3 ft
			VC7	0-3 ft
			VC16	3-6 ft, 6-9 ft, 9-12 ft
			VC17	6-9 ft, 9-12 ft, 12-15 ft
Chromium	86.5-202.0	N/A	VC6	0-3 ft
			VC7	0-3 ft
			VC16	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC17	3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft
Copper	37.3-265.0	N/A	VC6	0-3 ft
			VC7	0-3 ft, 3-6 ft
			VC16	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft
			VC17	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-17.5 ft
			VC37	0-3 ft
			VC38	0-3 ft
			VC42	3-6 ft
Lead	51.0-198.0	235.0-285.0	VC6	0-3 ft
			VC7	0-3 ft, 3-6 ft
			VC16	0-3 ft, 3-6ft, ***6-9 ft, 9-12 ft***, 12-15 ft
			VC17	0-3 ft, 3-6ft, ***6-9 ft***, 9-12 ft, 12-15 ft, 15-17.5 ft
			VC37	0-3 ft
			VC38	0-3 ft
			VC42	3-6 ft
Nickel	21.4-48.7	53.5	VC5	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC6	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC7	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC8	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC9	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC13	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC14	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC15	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16.5 ft
			VC16	0-3 ft, 3-6ft, ***6-9 ft,*** 9-12 ft, 12-15 ft
			VC17	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-17.5 ft

Table B-8 Sample Sites and Samples in Group B Exceeding Class A and/or Class C Thresholds for Metals

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded Class A (mg/kg)	Exceeded Class C (mg/kg)		
			VC18	3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16.2 ft
			VC19	3-6 ft, 6-9 ft, 9-12 ft
			VC20	3-6 ft, 6-9 ft, 9-12 ft
			VC23	6-9 ft
			VC27	0-3 ft, 3-6 ft
			VC29	9-12 ft
			VC38	0-3 ft
			VC42	3-6 ft
Silver	1.06-3.55	4.78-8.52	VC6	0-3 ft
			VC7	0-3 ft
			VC16	0-3 ft, ***3-6 ft, 6-9 ft, 9-12 ft,*** 12-15 ft
			VC17	0-3 ft, 3-6 ft, ***6-12 ft,*** 12-17.5 ft
			VC38	0-3 ft
Zinc	154-390	427-494	VC6	0-3 ft
			VC7	0-3 ft, 3-6 ft
			VC16	0-3 ft, 3-6 ft, ***6-9 ft, 9-12 ft***
			VC17	3-6 ft, 6-9 ft, ***9-12 ft,*** 12-17.5 ft
			VC38	0-3 ft
Mercury	0.265-0.515	0.904-4.22	VC6	***0-3 ft,*** 3-6 ft
			VC7	***0-3 ft, 3-6 ft***
			VC16	**0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft,** 12-15 ft
			VC17	***0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-17.5 ft***
			VC37	***0-3 ft***
			VC38	***0-3 ft***
			VC41-ALT	0-3 ft
			VC42	***3-6 ft***

Key:
* Sample depths where Class C thresholds were exceeded are in bold and italics.

Table B-9 Sample Sites and Samples in Group C Exceeding ER-L and/or ER-M Thresholds for PAHs

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded ER-L (µg/kg)	Exceeded ER-M (µg/kg)		
Acenaphthene	39.4-172	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	0-3 ft, 3-6 ft, 6-9 ft
Acenaphthylene	46.3-131	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Anthracene	122-989	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	0-3 ft, 3-6 ft, 6-9 ft
Benzo(A)Anthracene	282-1580	1670	VC54	0-3 ft, 3-6 ft, ***6-9 ft***
			VC54B	0-3 ft, 3-6 ft, 6-9 ft
Benzo(A)Pyrene	861-1330	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Benzo(G,H,I)Perylene	440-708	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Benzo(K)Fluoranthene	369-488	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Chrysene	733-1160	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Dibenz(A,H)Anthracene	134-211	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Fluoranthene	1680-2490	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Fluorene	46-263	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Indeno(1,2,3-C,D)Pyrene	457-753	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Phenanthrene	324-1440	1540-1570	VC54	0-3 ft, ***3-6 ft,*** 6-9 ft
			VC54B	3-6 ft, ***6-9 ft***
Pyrene	1530-2270	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Total PAHs	10400-15400	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft

Key:
* Sample depths where ER-M thresholds were exceeded are in bold and italics.
N/A = Detected concentrations did not exceed the ER-M thresholds.

Table B-10 Sample Sites and Samples where Metals Exceeded Class A and/or Class C Thresholds in the Alternative Group

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded Class A (mg/kg)	Exceeded Class C (mg/kg)		
Arsenic	8.27-49.6	N/A	VC70	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC71	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC72	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC73-ALT	0-3 ft, 9-12 ft
			VC74	6-9 ft
			VC75	0-3 ft
			VC76	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC77	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, VC12-15.6 ft
			VC78	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, VC12-15 ft, VC15-17 ft
			VC79	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, VC12-16 ft
			VC80	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, VC12-15 ft, VC15-16.5 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, VC12-14.75 ft
			VC82	0-3 ft, 6-9 ft, 9-12 ft
Cadmium	1.23-3.36	N/A	VC70	0-3 ft
			VC76	0-3 ft
			VC80	3-6 ft, 6-9 ft
			VC81	3-6 ft, 6-9 ft, 9-12 ft
Chromium	85.2-198	N/A	VC70	0-3 ft
			VC76	0-3 ft
			VC77	0-3 ft
			VC80	3-6 ft, 6-9 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
Copper	36.4-230	N/A	VC70	0-3 ft
			VC75	0-3 ft
			VC76	0-3 ft
			VC77	0-3 ft
			VC78	0-3 ft
			VC80	0-3 ft, 3-6 ft, 6-9 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC82	0-3 ft
Lead	67.9-194	223	VC70	0-3 ft
			VC75	0-3 ft
			VC76	0-3 ft
			VC77	0-3 ft
			VC78	0-3 ft, 6-9 ft
			VC80	0-3 ft, ***3-6 ft***, 6-9 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC82	0-3 ft
Nickel	22.3-48.4	N/A	VC70	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC71	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC72	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC75	0-3 ft
			VC76	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC77	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15.6 ft
			VC78	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-17 ft
			VC79-ALT	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-16 ft
			VC80	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16.5 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-14.75 ft
			VC82	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-16 ft

Table B-10 Sample Sites and Samples where Metals Exceeded Class A and/or Class C Thresholds in the Alternative Group

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded Class A (mg/kg)	Exceeded Class C (mg/kg)		
Silver	1.68-2.74	3.98-7.09	VC70	***0-3 ft***
			VC76	***0-3 ft***
			VC77	0-3 ft
			VC78	0-3 ft
			VC80	0-3 ft, ***3-6 ft, 6-9 ft***
			VC81	0-3 ft, ***3-6 ft, 6-9 ft, 9-12 ft***
Zinc	190-404	N/A	VC70	0-3 ft
			VC76	0-3 ft
			VC77	0-3 ft
			VC78	0-3 ft
			VC80	0-3 ft, 3-6 ft, 6-9 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
Mercury	0.191-0.337	1.03-3.49	VC70	***0-3 ft,*** 3-6 ft
			VC73-ALT	0-3 ft
			VC75	***0-3 ft***
			VC76	***0-3 ft***
			VC77	***0-3 ft,*** 3-6 ft
			VC78	***0-3 ft***
			VC79	0-3 ft
			VC80	***0-3 ft***
			VC81	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft,*** 12-14.75 ft
			VC82	***0-3 ft***

Key:
* Sample depths where Class C thresholds were exceeded are in bold and italics.
N/A = Detected concentrations did not exceed the Class C thresholds.

Table B-11 Sediment Grain Sizes from Vibracore Samples

Sample Site	Sample Depth	% Gravel (#4 Sieve)[1]	% Sand (#200 Sieve)[1]	% Fines (< #200 Sieve)	Water Content (%) [2]	USCS Symbol[1]
VC2	2'9.5"-3'9.5"	8.4	61.7	29.9	46.8	SC
VC2	8'3.5"-9'3.5"	0.0	2.4	97.6	28.0	CH
VC3-ALT	0-1'	0.0	17.4	82.6	124.1	CH
VC3-ALT	5'11.04"	ND	ND	89.9	87.7	ND
VC4	1-2'	0.3	4.5	95.2	95.0	CH
VC4	2-2'9.5"	0.0	5.2	94.8	90.9	CH
VC4	4'2.04"	ND	ND	98.0	88.4	ND
VC5	1-2'3.5"	0.0	4.5	95.5	105.2	CH
VC5	4'2.04"	ND	ND	97.0	114.5	ND
VC6	3'2.04"	ND	ND	98.9	134.0	ND
VC6	9'6"	ND	ND	95.4	95.4	ND
VC7	0-1'	1.1	6.1	92.8	132.8	CH
VC7	3'-4'	0.5	8.0	91.5	88.2	CH
VC7	6'2.04"	ND	ND	96.8	108.3	ND
VC8	7'10"-8'10"	0.0	5.0	95.0	104.5	CH
VC9	2'9.5"-4'	0.1	5.9	94.0	113.0	CH
VC9	9"0"	ND	ND	98.4	94.4	ND
VC10	2'8"-4'	32.6	65.4	2.0	45.9	SP
VC10	8'7"-10'	32.0	65.6	2.4	42.2	SP
VC11	0-1'	21.1	68.7	10.2	36.9	SP-SM
VC12	6'4"-7'4"	15.1	82.5	2.4	19.1	SP
VC14	0-1'	3.7	7.0	89.3	88.7	CH
VC14	2'10"-4'	0.3	7.6	92.1	105.8	CH
VC14	5'9"-7'	0.0	7.2	92.8	105.3	CH
VC15	1"-1'1"	0.2	6.8	93.0	86.6	CH
VC15	6'2.04"	ND	ND	96.3	87.2	ND
VC15	12'2.04"	ND	ND	95.3	84.5	ND
VC16	0-1'	0.0	4.2	95.8	152.7	CH
VC16	2'11.04"	ND	ND	94.8	132.1	ND
VC16	12"0"	ND	ND	98.7	129.0	ND
VC17	0-1'	0.0	12.5	87.5	169.9	MH
VC17	3'-4'	0.0	5.0	95.0	136.3	MH
VC17	6'2.04"	ND	ND	93.0	106.7	ND
VC17	9'2.04"	ND	ND	94.7	88.8	ND
VC17	12'2.04"	ND	ND	95.2	83.1	ND
VC18	2'8"-4'	0.0	6.7	93.3	92.7	CH
VC18	11'9"	ND	ND	98.0	78.9	ND
VC18	13'9"	ND	ND	98.3	89.8	ND
VC19	0-1'	0.3	15.2	84.5	61.3	CH
VC19	2'9"-4'	0.0	8.9	91.1	84.0	CH
VC19	5'9"	ND	ND	91.1	72.4	ND
VC20	1"-1'1"	0.1	23.3	76.6	60.9	CL
VC20	3'9.6"	ND	ND	81.9	65.7	ND
VC21	2.5"-1'2.5"	0.1	79.3	20.6	31.3	SM
VC21	2'7.5"-4'	6.9	51.0	42.1	47.3	SC
VC21	5'7.5"-7'	0.6	23.1	76.3	92.2	CL
VC21	8'8"-10'	0.2	36.9	62.9	85.6	CL
VC21	10'7"-11'7"	0.9	55.9	43.2	66.8	SC
VC22	0-1'	0.0	58.7	41.3	35.3	SC
VC22	3'-4'	40.2	46.4	13.4	29.2	SM
VC22	6'-7'	12.2	86.6	1.2	27.8	SP

Table B-11 Sediment Grain Sizes from Vibracore Samples

Sample Site	Sample Depth	% Gravel (#4 Sieve)[1]	% Sand (#200 Sieve)[1]	% Fines (< #200 Sieve)	Water Content (%) [2]	USCS Symbol[1]
VC22	9'-10'	29.7	68.2	2.1	37.8	SP
VC22	11'5"-12'5"	29.3	68.8	1.9	45.7	SP
VC23	0-1'	0.2	52.2	47.6	44.7	SC
VC23	2'7"-4'	0.7	29.2	70.1	73.7	CL
VC23	6'-7'	0.0	33.6	66.4	80.9	CL
VC23	9'-10'	0.3	28.9	70.8	80.7	CL
VC23	10'5.5-11'6	1.6	42.1	56.3	69.0	CL
VC24	0-1'	0.2	70.3	29.5	33.5	SC
VC24	2'8"-4'	2.6	49.1	48.3	51.6	SC
VC24	6'-7'	1.0	78.8	20.2	34.8	SC
VC24	9'-10'	0.3	96.8	2.9	20.6	SP
VC24	10'-11'5.5"	18.7	77.0	4.3	21.0	SP
VC25	0-1'	5.1	89.1	5.8	13.3	SP-SM
VC25	3'-4'	18.9	78.7	2.4	28.0	SP
VC25	6'-7'	4.2	89.8	6.0	20.8	SP-SM
VC25	10'-11'4.5"	29.3	67.9	2.8	34.8	SP
VC26	0-1'	0.2	97.8	2.0	12.8	SP
VC27	0-1'	10.2	82.5	7.3	22.0	SP-SM
VC27	5'7.5"-7'	3.9	93.8	2.3	32.9	SP
VC27	10'6"-11'6"	0.0	95.8	4.2	34.8	SP
VC28	0-1'	0.6	81.3	18.1	28.8	SM
VC28	6'-7'	14.8	80.4	4.8	24.4	SP
VC28	9'-10'	27.3	69.4	3.3	30.6	SP
VC29	0-1'	1.9	75.9	22.2	28.4	SM
VC29	6'-7'	5.2	92.1	2.7	31.9	SP
VC29	9'11"-11'3.5"	31.0	67.5	1.5	32.0	SP
VC30	3'-4'	3.2	93.9	2.9	28.4	SW
VC30	5'11.5"-6'11.5"	0.7	97.2	2.1	22.6	SP
VC30	9'-10'	1.1	96.4	2.5	31.9	SP
VC31-ALT	0-1'	0.0	93.3	6.7	22.6	SP-SM
VC31-ALT	5'7"-7'	2.1	35.0	62.9	63.2	CL
VC31-ALT	10'7"-11'7"	7.5	89.9	2.6	34.9	SP
VC32	3'-4'	5.3	91.7	3.0	34.0	SP
VC32	9'-10'	0.5	96.2	3.3	37.6	SP
VC32	10'-11'1"	0.0	96.1	3.9	48.0	SP
VC33-ALT	0-1'	0.8	94.9	4.3	15.8	SP
VC33-ALT	6'-7'	0.0	95.5	4.5	26.6	SP
VC33-ALT	10'-11'5.5"	2.4	94.7	2.9	32.9	SP
VC34	0-1'	0.8	96.9	2.3	11.2	SP
VC34	6'-7'	0.5	94.2	5.3	32.8	SP-SM
VC35	0-1'	3.5	87.4	9.1	15.3	SP-SM
VC35	3'-4'	0.0	83.8	16.2	31.9	SM
VC35	6'-7'	2.8	96.0	1.2	23.1	SP
VC36	0-1'	0.4	66.9	32.7	33.4	SC
VC36	6'-7'	10.7	87.6	1.7	30.5	SP
VC36	9'-10'	1.8	96.7	1.5	39.3	SP
VC36	12'-13'	1.3	97.5	1.2	47.3	SP
VC36	14'5"-15'5"	0.7	98.4	0.9	29.2	SP
VC37	3'-4'	0.0	91.0	9.0	29.9	SP-SM
VC37	6'-7'	2.8	91.8	5.4	28.3	SP-SM
VC37	9'-10'	0.8	97.3	1.9	47.7	SP

Table B-11 Sediment Grain Sizes from Vibracore Samples

Sample Site	Sample Depth	% Gravel (#4 Sieve)[1]	% Sand (#200 Sieve)[1]	% Fines (< #200 Sieve)	Water Content (%) [2]	USCS Symbol[1]
VC37	13'8.5"-15'5"	0.0	93.5	6.5	43.0	SP-SM
VC38	0-1'	0.0	31.6	68.4	70.3	OH
VC38	2'1"-4'	0.2	51.0	48.8	55.8	SM
VC38	6'-7'	2.0	87.9	10.1	39.1	SP-SM
VC38	9'-10'	1.2	97.1	1.7	46.9	SP
VC38	12'-13'	3.8	95.1	1.1	36.5	SP
VC38	14'3"-15'3"	2.5	94.2	3.3	42.5	SP
VC39	0-1'	0.5	80.7	18.8	27.3	SM
VC39	5'7.5"-7'	2.0	83.8	14.2	40.3	SM
VC39	9'-10'	4.2	91.0	4.8	25.4	SP
VC39	12'-13'	0.1	54.8	45.1	34.7	SC
VC39	14'2"-15'2"	0.0	80.6	19.4	30.0	SM
VC40	0-1'	0.0	94.7	5.3	26.8	SP-SM
VC40	6'-7'	0.5	88.7	10.8	28.1	SP-SM
VC40	12'-13'	0.0	80.9	19.1	38.8	SC
VC40	14'-15'5"	3.7	89.6	6.7	26.6	SP-SM
VC41-ALT	0-2.75"	12.1	77.9	10.0	19.3	SM
VC41-ALT	2'7.5"-3'11"	1.3	89.4	9.3	33.0	SP-SM
VC41-ALT	6'-7'	0.1	96.1	3.8	36.8	SP
VC41-ALT	9'-10'	0.0	97.0	3.0	35.4	SP
VC41-ALT	14'-15'6"	0.0	92.2	7.8	37.7	SP-SM
VC42	0-2"	10.2	86.5	3.3	6.8	SM
VC42	2"-1'2"	10.6	81.9	7.5	13.8	SP-SM
VC42	6'-7'	10.1	68.8	21.1	40.6	SM
VC42	10'-11'4"	0.4	97.8	1.8	43.9	SP
VC43	0-1'	0.2	99.4	0.4	7.4	SP
VC43	5'11"-7'	0.2	98.6	1.2	20.8	SP
VC43	10'11"-11'11"	0.1	97.7	2.2	31.8	SP
VC44	0-1'	0.6	99.0	0.4	9.7	SP
VC44	2'10"-4'	0.0	99.6	0.4	39.6	SP
VC44	5'10.5"-7'	1.3	98.2	0.5	24.9	SP
VC44	10'11.5"-12'	0.0	97.2	2.8	42.5	SP
VC45-ALT	0-1'	2.3	97.2	0.5	5.6	SP
VC45-ALT	2'9.5"-4'	0.1	99.6	0.3	28.2	SP
VC45-ALT	8'10"-10'	0.0	98.9	1.1	37.4	SP
VC45-ALT	10'10.5"-11'10"	0.0	98.3	1.7	39.4	SP
VC46	0-1'	12.9	73.8	13.3	21.6	SM
VC46	3'1"-4'1"	0.8	98.8	0.4	27.5	SP
VC46	6'1.5"-7'1.5"	0.2	98.7	1.1	42.9	SP
VC46	11'1.5"-12'2"	0.0	98.4	1.6	41.3	SP
VC47	0-1'	3.1	94.4	2.5	19.9	SP
VC47	3'6"	ND	ND	51.4	49.5	ND
VC47	5'9.5"-7'	0.9	98.7	0.4	30.4	SP
VC47	8'9"-10'	0.6	97.3	2.1	36.8	SP
VC47	10'8.5"-11'8.5"	0.1	97.8	2.1	39.8	SP
VC48-ALT	0-1'	1.0	98.1	0.9	12.9	SP
VC48-ALT	3'-4'	0.0	99.2	0.8	35.0	SP
VC48-ALT	6'-7'	0.3	99.0	0.7	39.3	SP
VC48-ALT	9'0.5"-10'0.5"	0.0	98.8	1.2	28.7	SP
VC48-ALT	10'6.5"-11'7"	0.0	98.1	1.9	52.0	SP

Table B-11 Sediment Grain Sizes from Vibracore Samples

Sample Site	Sample Depth	% Gravel (#4 Sieve)[1]	% Sand (#200 Sieve)[1]	% Fines (< #200 Sieve)	Water Content (%) [2]	USCS Symbol[1]
VC49	2'11"-4'	0.0	98.3	1.7	16.7	SP
VC49	6'-7'	0.2	99.5	0.3	18.7	SP
VC49	11'-12'	0.0	98.2	1.8	40.2	SP
VC50	0-1'	0.8	98.0	1.2	28.7	SP
VC50	1-2'	0.0	98.7	1.3	28.7	SP
VC50	2'10.5"-4'	0.9	98.6	0.5	20.6	SP
VC50	5'11.5"-7'	0.0	100.0	0.0	27.1	SP
VC51	3'-4'	0.0	98.7	1.3	37.3	SP
VC51	6'1"-7'2"	0.7	96.1	3.2	28.3	SP
VC51	9'1.5"-10'1.5"	0.0	98.0	2.0	29.1	SP
VC52	2'9.5"-4'	0.0	99.1	0.9	35.9	SP
VC52	8'10.5"-10'	0.2	98.0	1.8	25.0	SP
VC53	0-1'	0.1	99.6	0.3	9.4	SP
VC53	2'11.5"-4'	3.2	95.4	1.4	33.7	SP
VC53	6'-7'	1.3	96.0	2.7	27.3	SP
VC53	9'-10'	0.2	96.6	3.2	32.9	SP
VC53	11'1"-12'1"	0.5	95.9	3.6	34.3	SP
VC54	0-1'	0.2	97.0	2.8	12.6	SP
VC54	5'11.5"-7'	0.1	92.9	7.0	30.6	SP-SM
VC54	11'0.5"-12'0.5"	0.1	79.6	20.3	36.0	SC
VC55	0-1'	0.0	99.4	0.6	12.3	SP
VC55	3'1"-4'1"	4.3	94.5	1.2	24.3	SP
VC55	6'3"-7'3"	14.4	84.7	0.9	45.4	SP
VC55	9'7.5"-12'4"	0.4	98.1	1.5	5.8	SP
VC55	12'4"-13'4"	0.1	98.9	1.0	40.3	SP
VC55	15'5"-16'5"	4.1	95.3	0.6	30.8	SP
VC55	18'6"-21'6"	8.1	90.1	1.8	7.7	SP
VC55	21'6"-22'6"	3.8	94.0	2.2	24.2	SP
VC55	24'7"-25'7"	4.9	92.9	2.2	31.5	SP
VC55	27'7.5"-28'5"	0.7	96.6	2.7	22.6	SP
VC56	0-1'	6.6	93.1	0.3	11.4	SP
VC56	2'9"-4'	0.2	99.5	0.3	17.4	SP
VC56	8'10"-10'	3.0	96.3	0.7	15.7	SP
VC57	0-1'	8.0	91.4	0.6	13.5	SP
VC57	2'11.5"-4'	3.8	93.7	2.5	39.1	SP
VC57	6'-7'	18.4	78.9	2.7	45.9	SP
VC57	9'0.5"-10'	2.5	95.9	1.6	28.9	SP
VC57	11'1"-12'1.5"	0.1	98.2	1.7	35.2	SP
VC58	0-1'	4.1	95.9	0.0	12.2	SP
VC58	2'11"-4'	0.0	99.4	0.6	24.7	SP
VC58	9'0.5"-10'	0.0	98.5	1.5	27.7	SP
VC58	11'-12'	0.0	96.9	3.1	35.7	SP
VC59	0-1'	3.2	96.0	0.8	15.9	SP
VC59	2'10.5"-4'	0.1	94.8	5.1	25.8	SP-SM
VC59	5'10"-7'	3.3	96.2	0.5	33.9	SP
VC60	0-1'	22.8	76.4	0.8	32.9	SP
VC60	2'10.5"-4'	0.0	99.6	0.4	30.9	SP
VC60	6'0.5"-7'	0.0	99.5	0.5	25.8	SP
VC61-ALT	0-1'	1.2	97.4	1.4	11.3	SP
VC61-ALT	3'-4'	0.0	99.5	0.5	40.1	SP

Table B-11 Sediment Grain Sizes from Vibracore Samples

Sample Site	Sample Depth	% Gravel (#4 Sieve)[1]	% Sand (#200 Sieve)[1]	% Fines (< #200 Sieve)	Water Content (%) [2]	USCS Symbol[1]
VC62	0-1'	2.3	95.9	1.8	11.4	SP
VC62	3-4'2"	4.0	94.4	1.6	21.8	SP
VC63	0-1'	0.2	91.7	8.1	26.8	SP-SM
VC63	3'-4'	0.0	94.5	5.5	37.0	SP-SM
VC63	6'-7'0.5"	0.0	96.2	3.8	40.0	SP
VC64	0-1'	0.5	95.9	3.6	24.5	SP
VC64	3'-4'	0.3	96.3	3.4	36.9	SP
VC64	5'-6'0.5"	11.1	87.6	1.3	27.8	SP
VC65	0-1'	2.8	94.4	2.8	10.1	SP
VC65	3-4'2"	0.4	98.6	1.0	28.1	SP
VC66	0-1'	0.4	93.5	6.1	30.9	SP-SM
VC66	3'1"-4'8"	0.0	99.4	0.6	46.2	SP
VC67	0-1'	0.2	93.7	6.1	22.0	SP-SM
VC67	4'1"-4'11"	0.1	96.2	3.7	42.9	SP
VC68	0-1'	3.1	93.2	3.7	25.9	SP
VC68	3'-4'3"	0.0	95.4	4.6	32.2	SP
VC68	5'3"-6'3"	1.9	91.8	6.3	36.8	SP-SM
VC69-ALT	0-1'	4.1	93.5	2.4	20.8	SP
VC69-ALT	3'2"-4'2"	0.3	97.0	2.7	27.9	SP
VC69-ALT	6'3"-7'3"	0.7	92.9	6.4	38.9	SP-SM
VC70	3'-4'	2.2	8.0	89.8	115.1	CH
VC70	6'-7'	7.1	3.3	89.6	113.5	CH
VC71	0-1'	0.1	4.5	95.4	112.1	CH
VC72	6'6.5"-7'6.5"	0.0	2.5	97.5	115.8	CH
VC73-ALT	0-1'	4.0	91.9	4.1	20.4	SP
VC73-ALT	3'5.5"-4'5.5"	50.6	45.2	4.2	17.5	GP
VC73-ALT	9'-10'	19.6	78.0	2.4	14.3	SP
VC74-ALT	0-1'	5.3	88.5	6.2	17.2	SP-SM
VC74-ALT	6'-7'	8.7	89.6	1.7	39.4	SP
VC74-ALT	9'-10'	6.2	90.9	2.9	32.1	SP
VC75	0-1'	0.9	21.1	78.0	98.0	CH
VC75	2'6.5"-4'	19.0	68.5	12.5	48.2	SC
VC75	6'-7'	1.9	93.6	4.5	30.2	SP
VC75	9'-10'	0.7	96.8	2.5	27.7	SP
VC78	0-1'	0.9	10.7	88.4	114.9	CH
VC78	6'2.04"	ND	ND	94.8	90.8	ND
VC79-ALT	2'9.96"	ND	ND	93.5	94.5	ND
VC79-ALT	8'9"	ND	ND	96.9	105.8	ND
VC81	0-1'	0.0	12.2	87.8	187.0	CH
VC81	6'2.04"	ND	ND	96.7	131.3	ND
VC81	12'2.04"	ND	ND	94.9	87.7	ND
VC81	14'6.48"	ND	ND	98.1	75.9	ND
VC82	9'2.04"	ND	ND	82.1	61.5	ND
VC82	11'3"	ND	ND	96.7	70.0	ND
VC83-ALT	0-1'	1.2	94.6	4.2	24.6	SP
VC83-ALT	2'7"-4'	1.5	89.7	8.8	27.5	SP-SM
VC83-ALT	6'-7'	0.3	93.5	6.2	30.4	SP-SM
VC84-ALT	0-1'	4.6	87.4	8.0	21.6	SP-SM
VC84-ALT	5'8"-7'	26.3	70.9	2.8	26.8	SP
VC84-ALT	10'-11'1"	1.2	96.2	2.6	32.3	SP

Table B-11 Sediment Grain Sizes from Vibracore Samples

Sample Site	Sample Depth	% Gravel (#4 Sieve)[1]	% Sand (#200 Sieve)[1]	% Fines (< #200 Sieve)	Water Content (%) [2]	USCS Symbol[1]
VC85	0-1'	1.8	93.5	4.7	32.7	SP
VC85	8'5"-9'5"	0.0	96.1	3.9	31.9	SP
VC85	9'5"-10'5"	7.6	82.8	9.6	42.1	SP-SM
VC86-ALT	0-1'	2.1	92.3	5.6	26.7	SP-SM
VC86-ALT	3'-4'	0.1	96.5	3.4	49.6	SP
VC86-ALT	6'-7'	3.7	94.8	1.5	35.9	SP
VC87	0-1'	0.2	72.5	27.3	26.6	SM
VC87	8'9"-9'8.5"	0.0	95.2	4.8	19.1	SP

[1] Samples with reported values of "ND" did not have an accompanying sieve analysis and have no available data.

[2] Water Content (%) is a measure of the ratio of water to sediment in a given sample. When the reported value exceeds 100%, more water than sediment was present in the sample.

Key:

CH = Inorganic clays of high plasticity, fat clays
CL = Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
GP = Poorly-graded gravels, gravel-sand mixtures, little or no fines
MH = Inorganic silts, micaceous or diatomoaceous fine sandy or silty soils, elastic silts
ND = no data
OH = Organic clays of medium to high plasticity, organic silts
SC = Clayey sands, sand-clay mixtures
SM = Silty sands, sand-silt mixtures
SP = Poorly graded sands, gravelly sands, little or no fines
SW = Well-graded sands, gravelly sands, little or no fines
USCS = Unified Soil Classification System

Table B-12 Sediment Grain Sizes from Benthic Grab Samples

Sample ID	% Gravel (#4 Sieve)	% Sand (#200 Sieve)	% Fines (< #200 Sieve)	Water Content (%)	USCS Symbol[1]
VC1-ALT	0.2	90.4	9.4	45.8	SP-SM
VC2	1.3	88.8	9.9	30.2	SP-SM
VC3-ALT	18.3	22.4	59.3	60.1	OH
VC4	6.0	10.7	83.3	105.0	OH
VC5	4.7	13.9	81.4	109.9	OH
VC6	0.0	2.6	97.4	128.6	OH
VC7	0.0	4.3	95.7	137.4	OH
VC8	39.1	33.2	27.7	40.9	GC
VC9	16.3	49.9	33.8	50.5	SC
VC10	12.5	82.1	5.4	28.2	SP-SM
VC11	2.4	91.7	5.9	24.5	SP-SM
VC12	21.0	37.2	41.8	14.4	SM
VC13	26.8	36.6	36.6	46.7	SC
VC14	33.0	37.5	29.5	52.3	SM
VC15	16.1	44.0	39.9	50.6	SC
VC16	0.0	3.9	96.1	122.0	OH
VC17	0.0	6.0	94.0	131.8	OH
VC18	32.3	34.1	33.6	47.5	SC
VC19	0.0	45.2	54.8	55.8	CL
VC20	12.4	50.2	37.4	48.5	SC
VC21	7.4	74.6	18.0	27.2	SM
VC22	20.1	62.9	17.0	40.7	SM
VC23	20.7	64.6	14.7	33.7	SC
VC24	2.4	88.1	9.5	30.5	SP-SM
VC25	0.3	93.5	6.2	29.5	SP-SM
VC26	2.5	96.1	1.4	23.7	SP
VC27	1.4	93.3	5.3	23.0	SP-SM
VC28	1.5	93.1	5.4	24.0	SP-SM
VC29	4.3	90.6	5.1	25.0	SP-SM
VC30	0.3	94.5	5.2	23.7	SP-SM
VC31-ALT	0.7	93.9	5.4	25.5	SP-SM
VC32	4.6	92.5	2.9	26.2	SP
VC33-ALT	0.6	97.5	1.9	22.0	SP
VC34	0.4	96.8	2.8	28.5	SP
VC35	3.2	94.0	2.8	17.8	SP
VC36	0.4	96.7	2.9	24.6	SP
VC37	3.3	66.0	30.7	55.3	SM
VC38	4.3	62.7	33.0	61.8	SM
VC39	0.2	91.0	8.8	35.2	SP-SM
VC40	0.0	89.4	10.6	27.6	SP-SM
VC41-ALT	20.1	61.7	18.2	41.1	SC

Table B-12 Sediment Grain Sizes from Benthic Grab Samples

Sample ID	% Gravel (#4 Sieve)	% Sand (#200 Sieve)	% Fines (< #200 Sieve)	Water Content (%)	USCS Symbol[1]
VC42	9.8	88.2	2.0	21.1	SP
VC43	1.6	97.3	1.1	22.1	SP
VC44	6.0	93.2	0.8	22.2	SP
VC45-ALT	6.5	92.2	1.3	22.6	SP
VC46	0.6	98.5	0.9	24.1	SP
VC47	0.2	99.0	0.8	24.0	SP
VC48-ALT	2.1	96.3	1.6	27.5	SP
VC49	0.0	98.9	1.1	32.3	SP
VC50	0.2	98.8	1.0	25.9	SP
VC51	0.8	98.4	0.8	24.9	SP
VC52	0.0	99.0	1.0	27.2	SP
VC53	0.0	99.0	1.0	26.0	SP
VC54	0.0	99.1	0.9	21.8	SP
VC55	0.0	99.2	0.8	19.7	SP
VC56	0.4	98.8	0.8	22.5	SP
VC57	11.4	87.8	0.8	19.7	SP
VC58	1.0	97.5	1.5	27.3	SP
VC59	0.9	98.0	1.1	25.6	SP
VC60	2.6	96.6	0.8	19.7	SP
VC61-ALT	0.0	99.0	1.0	26.7	SP
VC62	1.4	96.4	2.2	26.7	SP
VC63	0.0	96.7	3.3	25.5	SP
VC64	0.5	95.7	3.8	26.5	SP
VC65	0.6	96.1	3.3	33.4	SP
VC66	0.5	96.2	3.3	29.3	SP
VC67	0.4	95.9	3.7	31.2	SP
VC68	0.0	97.8	2.2	30.4	SP
VC69-ALT	0.0	97.8	2.2	27.3	SP
VC70	0.4	2.2	97.4	155.4	OH
VC71	22.2	41.6	36.2	53.4	SC
VC72	15.9	60.5	23.6	57.6	SM
VC73-ALT	18.0	65.7	16.3	50.7	SM
VC74	4.1	89.7	6.2	34.9	SP-SM
VC75	43.2	13.9	42.9	55.0	GC
VC76	0.2	5.3	94.5	120.0	OH
VC77	0.0	5.5	94.5	117.3	OH
VC78	15.2	4.9	79.9	100.8	OH
VC79-ALT	0.3	12.7	87.0	113.3	OH
VC80	0.0	5.7	94.3	123.1	OH
VC81	0.0	4.5	95.5	132.9	OH
VC82	31.5	43.4	25.1	44.4	SC

Table B-12 Sediment Grain Sizes from Benthic Grab Samples

Sample ID	% Gravel (#4 Sieve)	% Sand (#200 Sieve)	% Fines (< #200 Sieve)	Water Content (%)	USCS Symbol[1]
VC83-ALT	1.5	96.6	1.9	25.6	SP
VC84-ALT	0.9	96.5	2.6	24.1	SP
VC85	6.5	91.5	2.0	21.6	SP
VC86-ALT	1.4	96.8	1.8	18.6	SP
VC87	0.3	91.5	8.2	27.1	SP-SM

[1] Water Content (%) is a measure of the ratio of water to sediment in a given sample. When the reported value exceeds 100%, more water than sediment was present in the sample.

Key:

CL = Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
GC = Clayey gravels, gravel-sand-clay mixtures
OH = Organic clays of medium to high plasticity, organic silts
SC = Clayey sands, sand-clay mixtures
SM = Silty sands, sand-silt mixtures
SP = Poorly graded sands, gravelly sands, little or no fines
USCS = Unified Soil Classification System

Table B-13 Physical Water Quality Sample Locations

Sample Site	State	Maximum Depth Sampled	Sample Site	State	Maximum Depth Sampled
VC1-ALT	NJ	16	VC45-ALT	NJ	18
VC2	NJ	12	VC46	NJ	20
VC3-ALT	NJ	14	VC47	NJ	18
VC4	NJ	16	VC48-ALT	NJ	18
VC5	NY	16	VC49	NJ	18
VC6	NY	18	VC50	NJ	18
VC7	NY	20	VC51	NJ	24
VC8	NY	18	VC52	NJ	22
VC9	NY	18	VC53	NJ	24
VC10	NY	14	VC54	NJ	40
VC11	NY	16	VC55	NJ	18
VC12	NY	16	VC56	NY	20
VC13	NY	16	VC57	NY	26
VC14	NY	18	VC58	NY	34
VC15	NY	22	VC59	NY	32
VC16	NY	34	VC60	NY	28
VC17	NY	36	VC61-ALT	NY	28
VC18	NY	20	VC62	NY	30
VC19	NY	18	VC63	NY	32
VC20	NY	22	VC64	NY	32
VC21	NY	20	VC65	NY	34
VC22	NY	20	VC66	NY	38
VC23	NY	20	VC67	NY	40
VC24	NY	18	VC68	NY	40
VC25	NY	20	VC69-ALT	NY	40
VC26	NY	18	VC70	NY	18
VC27	NY	20	VC71	NY	16
VC28	NY	22	VC72	NY	18
VC29	NY	22	VC73-ALT	NY	16
VC30	NY	20	VC74	NY	16
VC31-ALT	NY	18	VC75	NY	22
VC32	NY	18	VC76	NY	20
VC33-ALT	NY	20	VC77	NY	22
VC34	NY	22	VC78	NY	22
VC35	NY	24	VC79-ALT	NY	28
VC36	NY	24	VC80	NY	36
VC37	NY	24	VC81	NY	32
VC38	NY	28	VC82	NY	20
VC39	NY	36	VC83-ALT	NY	16
VC40	NY	36	VC84-ALT	NY	16
VC41-ALT	NY	28	VC85	NY	18
VC42	NY	16	VC86-ALT	NY	22
VC43	NY	16	VC87	NY	22
VC44	NY	18			

Table B-14 Benthic Macroinvertebrate Taxonomic List for Preferred Route

Taxonomic Name	Frequency of Occurrence	Count (N=207)	Relative Abundance (%)
Acanthohaustorius shoemakeri	11	25.0	0.0153
Acanthohaustorius similis	37	303.0	0.1851
Acteocina canaliculata	21	304.9	0.1862
Alitta succinea	15	226.9	0.1386
Amastigos caperatus	2	5.0	0.0031
Americhelidium americanum	18	53.7	0.0328
Ameritella agilis	116	2199.7	1.3437
Ampelisca abdita	32	1942.1	1.1864
Ampelisca vadorum	9	83.4	0.0509
Ampelisca verrilli	12	182.3	0.1113
Ampharete oculata	7	41.9	0.0256
Amphiporeia virginiana	1	1.0	0.0006
Amphiporus bioculatus	34	325.9	0.1991
Amphiporus ochraceus	4	42.0	0.0257
Anachis lafresnayi	1	1.0	0.0006
Anadara transversa	5	14.0	0.0086
Ancinus depressus	39	97.2	0.0594
Aphelochaeta sp.	1	2.3	0.0014
Apoprionospio pygmaea	28	276.7	0.1690
Aricidea (Acmira) *catherinae*	6	50.7	0.0309
Aricidea (Acmira) *cerruti*	1	1.0	0.0006
Aricidea (Acmira) sp.	20	198.7	0.1214
Aricidea wassi	7	17.0	0.0104
Asmunda elegantula	5	134.9	0.0824
Astyris lunata	1	1.0	0.0006
Batea catharinensis	5	24.1	0.0147
Bathyporeia quoddyensis	9	27.0	0.0165
Bivalvia	5	73.2	0.0447
Boonea impressa	1	8.0	0.0049
Boonea seminuda	2	24.0	0.0147
Brania wellfleetensis	18	232.3	0.1419
Caprella penantis	2	11.2	0.0068
Carinoma tremaphoros	19	38.1	0.0233
Caulleriella venefica	73	872.9	0.5332
Cerapus tubularis	1	1.8	0.0011
Cerebratulus lacteus	4	5.8	0.0035
Chaetozone diodonta	2	40.0	0.0244
Chaetozone sp.	1	1.0	0.0006
Chiridotea arenicola	5	14.0	0.0086
Chiridotea tuftsi	16	19.0	0.0116
Cirratulidae	64	3706.4	2.2641
Clymenella torquata	6	57.1	0.0349
Corambe obscura	3	16.2	0.0099
Crangon septemspinosa	3	10.0	0.0061

Table B-14 Benthic Macroinvertebrate Taxonomic List for Preferred Route

Taxonomic Name	Frequency of Occurrence	Count (N=207)	Relative Abundance (%)
Crepidula convexa	2	9.6	0.0059
Crepidula fornicata	30	527.7	0.3223
Crepidula plana	14	88.6	0.0541
Crepidula sp.	7	43.4	0.0265
Cyathura burbancki	37	524.2	0.3202
Deutella incerta	3	113.8	0.0695
Diastylis sculpta	6	9.3	0.0057
Diopatra cuprea	10	45.9	0.0280
Dipolydora socialis	6	30.1	0.0184
Dispio uncinata	12	17.0	0.0104
Donax variabilis	5	7.0	0.0043
Drilonereis longa	16	95.0	0.0580
Dyspanopeus sayi	13	149.5	0.0913
Edotia triloba	53	192.7	0.1177
Edwardsia elegans	1	3.2	0.0020
Elasmopus levis	7	87.1	0.0532
Ensis leei	43	128.7	0.0786
Eobrolgus spinosus	4	48.0	0.0293
Ericthonius brasiliensis	10	411.3	0.2512
Euclymene collaris	49	955.6	0.5837
Eumida sanguinea	56	1312.1	0.8015
Eupleura caudata	19	112.4	0.0687
Exogone dispar	61	2565.4	1.5671
Fargoa bartschi	5	50.1	0.0306
Gastropoda	3	7.8	0.0048
Gemma gemma	3	45.3	0.0277
Gilvossius setimanus	1	4.0	0.0024
Glycera americana	61	758.7	0.4635
Glycera dibranchiata	31	259.5	0.1585
Glycera sp.	15	65.5	0.0400
Glycinde solitaria	84	3268.7	1.9967
Goniadidae	2	4.2	0.0026
Grandidierella japonica	2	16.0	0.0098
Gyptis vittata	37	404.2	0.2469
Hemipodus sp.	6	19.0	0.0116
Hesionura elongata	8	22.0	0.0134
Heteromastus filiformis	36	551.0	0.3366
Heteromysis formosa	4	33.0	0.0202
Hypereteone heteropoda	12	182.3	0.1113
Hypereteone sp.	29	298.0	0.1820
Japonactaeon punctostriatus	9	245.3	0.1499
Jassa marmorata	2	7.0	0.0043
Kelliopsis elevata	1	10.7	0.0065
Leitoscoloplos fragilis	15	107.9	0.0659

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Table B-14 Benthic Macroinvertebrate Taxonomic List for Preferred Route

Taxonomic Name	Frequency of Occurrence	Count (N=207)	Relative Abundance (%)
Leitoscoloplos robustus	11	21.8	0.0133
Leitoscoloplos sp.	47	222.9	0.1362
Lepidonotus sublevis	5	87.5	0.0534
Levinsenia gracilis	1	2.0	0.0012
Libinia dubia	1	5.3	0.0033
Lyonsia arenosa	4	53.1	0.0325
Lyonsia sp.	14	165.7	0.1012
Lysianopsis alba	3	29.3	0.0179
Magelona ***sp.***	35	76.0	0.0464
Maldanidae	47	1128.3	0.6892
Mediomastus ambiseta	134	93041.8	56.8347
Mercenaria mercenaria	60	317.5	0.1939
Microphthalmus sczelkowii	4	23.4	0.0143
Microphthalmus similis	1	1.0	0.0006
Microphthalmus sp.	1	1.0	0.0006
Micrura sp.	1	1.0	0.0006
Monocorophium acherusicum	5	17.1	0.0105
Monocorophium sp.	1	2.0	0.0012
Monocorophium tuberculatum	13	96.6	0.0590
Mulinia lateralis	22	224.6	0.1372
Mya arenaria	7	26.0	0.0159
Myrianida prolifera	19	267.0	0.1631
Mytilus edulis	7	62.5	0.0382
Neanthes arenaceodentata	3	7.3	0.0045
Nemertea	4	20.7	0.0126
Nephtys bucera	31	50.6	0.0309
Nephtys picta	38	211.3	0.1290
Nereis zonata	1	4.0	0.0024
Neverita duplicata	5	5.0	0.0031
Notomastus sp.	7	13.0	0.0079
Nucula proxima	27	475.7	0.2906
Nudibranchia	3	11.7	0.0072
Odontosyllis fulgurans	5	55.5	0.0339
Odostomia eburnea	1	4.0	0.0024
Odostomia sp.	2	8.5	0.0052
Oligochaeta	177	8553.2	5.2247
Onuphis eremita	12	33.0	0.0202
Ophelia bicornis	6	36.0	0.0220
Opheliidae	4	5.0	0.0031
Opisthodonta longocirrata	17	228.3	0.1395
Orbinia ornata	9	11.0	0.0067
Orbinia sp.	1	2.0	0.0012
Ostrea edulis	1	1.0	0.0006
Ovalipes ocellatus	1	1.0	0.0006

Table B-14 Benthic Macroinvertebrate Taxonomic List for Preferred Route

Taxonomic Name	Frequency of Occurrence	Count (N=207)	Relative Abundance (%)
Oxydromus obscurus	25	386.1	0.2358
Oxyurostylis smithi	7	48.2	0.0295
Pagurus longicarpus	34	290.4	0.1774
Pagurus pollicaris	2	2.0	0.0012
Pandora glacialis	1	2.3	0.0014
Paracaprella tenuis	7	119.2	0.0728
Parahaustorius attenuatus	8	11.0	0.0067
Parahaustorius holmesi	6	10.0	0.0061
Parahaustorius longimerus	18	190.0	0.1161
Parametopella cypris	10	1055.8	0.6449
Paranaitis speciosa	32	365.4	0.2232
Paraonidae	5	12.1	0.0074
Paraprionospio pinnata	10	55.7	0.0340
Parougia caeca	1	1.0	0.0006
Pectinaria gouldii	13	115.0	0.0702
Petricolaria pholadiformis	6	91.3	0.0558
Photis pollex	1	1.0	0.0006
Phyllodoce arenae	24	114.5	0.0699
Phyllodoce sp.	1	3.2	0.0020
Phyllodocidae	1	5.3	0.0033
Pilargidae	1	2.7	0.0016
Pisione remota	3	3.0	0.0018
Pitar morrhuanus	1	1.0	0.0006
Politolana concharum	3	3.0	0.0018
Polycirrus eximius	1	6.4	0.0039
Polycirrus sp.	35	444.2	0.2713
Polydora cornuta	30	484.2	0.2958
Polydora sp.	2	2.0	0.0012
Polygordius jouinae	99	2814.4	1.7192
Protodorvillea gaspeensis	6	21.0	0.0129
*Protohaustorius cf. wigleyi [*In LeCroy, 2002]	26	1126.7	0.6882
Protohaustorius wigleyi	33	210.0	0.1283
Pseudoleptocuma minus	13	27.0	0.0165
Pseudunciola obliquua	1	2.0	0.0012
Retusa obtusa	2	9.3	0.0057
Rhepoxynius epistomus	40	273.2	0.1669
Rhepoxynius hudsoni	21	147.2	0.0899
Sabaco elongatus	6	33.9	0.0207
Sabellaria vulgaris	40	1798.7	1.0987
Sabellidae	2	19.2	0.0117
Salvatoria clavata	6	88.0	0.0538
Scalibregma inflatum	1	1.0	0.0006
Schistomeringos annulata	1	5.3	0.0033

Table B-14 Benthic Macroinvertebrate Taxonomic List for Preferred Route

Taxonomic Name	Frequency of Occurrence	Count (N=207)	Relative Abundance (%)
Scolelepis sp.	1	2.0	0.0012
Scolelepis squamata	1	1.0	0.0006
Scoletoma fragilis	5	6.6	0.0040
Scoloplos (Leodamus) rubra	7	24.2	0.0148
Sigalion arenicola	16	43.6	0.0266
Solenidae	3	40.5	0.0248
Sphaerodoridae	5	13.7	0.0083
Spiochaetopterus costarum oculatus	10	57.2	0.0349
Spionidae	5	16.3	0.0099
Spiophanes bombyx	50	279.0	0.1704
Spisula solidissima	73	491.6	0.3003
Stenothoe minuta	6	900.8	0.5502
Sthenelais boa	19	125.8	0.0769
Sthenelais limicola	9	12.7	0.0077
Streblospio benedicti	110	15020.7	9.1754
Streptosyllis verrilli	19	81.3	0.0497
Stylochus ellipticus	6	28.2	0.0173
Syllidae	5	15.0	0.0092
Syllis gracilis	1	16.0	0.0098
Tagelus plebius	2	10.7	0.0065
Tanaissus sp.	35	375.0	0.2291
Terebellidae	2	14.4	0.0088
Tharyx acutus	60	1330.9	0.8130
Travisia forbesii	4	4.0	0.0024
Tritia obsoleta	17	255.9	0.1563
Tritia trivittata	83	703.5	0.4298
Tubulanus pellucidus	49	654.2	0.3996
Turbellaria sp.	7	267.0	0.1631
Unciola dissimilis	5	12.8	0.0078
Unciola irrorata	17	85.5	0.0522
Unciola serrata	47	1147.6	0.7010
Upogebia affinis	1	2.7	0.0016
Urosalpinx cinerea	10	76.2	0.0465
Zygonemertes virescens	5	24.6	0.0150

Table B-15 Summary of Results for Benthic Species Analysis (50 Most Abundant Species)

	Rank	Total Number of Individuals[1]	VC1ALT	VC2	VC3ALT	VC4	VC5	VC6	VC7	VC8	VC9	VC10	VC11	VC12	VC13	VC14
Mediomastus ambiseta	1	93041.8	4606.0	4421.3	6200.0	2371.2	2150.4	319.3	438.7	5829.3	5628.8	1237.3	572.0	605.4	894.3	3748.3
Streblospio benedicti	2	15020.7	360.0	2490.7	896.0	307.2	106.7	0.0	4.5	762.7	1075.2	170.7	280.9	64.2	67.8	388.3
Oligochaeta	3	8553.2	440.0	288.0	64.0	121.6	24.5	18.7	24.5	272.0	297.6	693.3	77.3	9.5	68.5	109.9
Cirratulidae	4	3706.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	37.3	0.0	0.0	2.6	0.0
Glycinde solitaria	5	3268.7	16.0	32.0	176.0	200.0	265.6	130.7	106.4	165.3	188.8	0.0	15.1	165.3	75.7	86.4
Polygordius jouinae	6	2814.4	0.0	0.0	0.0	12.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Exogone dispar	7	2565.4	0.0	0.0	0.0	0.0	0.0	0.0	4.3	16.0	176.0	413.3	185.3	32.4	55.3	5.3
Ameritella agilis	8	2199.7	8.0	0.0	0.0	32.0	0.0	2.0	0.0	58.7	0.0	26.7	4.0	0.0	0.0	0.0
Ampelisca abdita	9	1942.1	48.0	0.0	64.0	156.8	220.8	382.0	70.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Sabellaria vulgaris	10	1798.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	42.7	51.2	0.0	12.5	2.3	30.5	16.0
Tharyx acutus	11	1330.9	0.0	16.0	0.0	0.0	0.0	0.0	0.0	32.0	0.0	29.3	0.0	0.0	0.0	12.8
Eumida sanguinea	12	1312.1	16.0	0.0	0.0	0.0	0.0	0.0	0.0	96.0	92.8	90.7	40.0	2.3	19.2	0.0
Unciola serrata	13	1147.5	0.0	21.3	0.0	0.0	0.0	0.0	0.0	0.0	41.6	21.3	0.0	0.0	13.2	5.3
Maldanidae	14	1128.3	8.0	80.0	0.0	0.0	25.6	0.0	9.3	32.0	172.8	0.0	90.2	2.3	0.0	0.0
Protohaustorius cf. wigleyi [In LeCroy, 2002]	15	1126.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Parametopella cypris	16	1055.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Euclymene collaris	17	955.5	0.0	0.0	8.0	0.0	5.3	0.0	7.2	0.0	32.0	0.0	1.8	80.9	46.3	53.3
Stenothoe minuta	18	900.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Caulleriella venefica	19	872.9	0.0	16.0	0.0	0.0	0.0	0.0	0.0	32.0	0.0	5.3	0.0	0.0	0.0	0.0
Glycera americana	20	758.7	0.0	0.0	0.0	16.0	0.0	8.7	6.7	0.0	115.2	18.7	13.3	13.7	31.2	10.7
Tritia trivittata	21	703.6	16.0	0.0	0.0	24.0	6.4	6.7	8.8	0.0	0.0	5.3	3.6	16.4	21.1	0.0
Tubulanus pellucidus	22	654.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	5.3
Heteromastus filiformis	23	551.0	0.0	32.0	16.0	16.0	64.0	15.3	7.2	16.0	16.0	0.0	0.0	23.2	13.4	21.3
Crepidula fornicata	24	527.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	16.0	8.0	1.8	0.0	5.3	0.0
Cyathura burbancki	25	524.2	0.0	0.0	8.0	12.8	0.0	2.7	5.9	80.0	115.2	66.7	0.0	9.5	57.4	34.1
Spisula solidissima	26	491.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Polydora cornuta	27	484.2	0.0	96.0	16.0	0.0	0.0	0.0	1.3	85.3	48.0	0.0	14.7	16.4	2.6	0.0
Nucula proxima	28	475.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	5.3
Polycirrus sp.	29	444.2	0.0	0.0	0.0	6.4	0.0	0.0	0.0	0.0	16.0	5.3	0.0	0.0	0.0	0.0
Ericthonius brasiliensis	30	411.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Gyptis vittata	31	404.2	8.0	0.0	56.0	54.4	30.9	12.0	9.9	26.7	32.0	21.3	0.0	4.9	22.6	18.1
Oxydromus obscurus	32	386.1	0.0	37.3	16.0	0.0	38.4	0.0	0.0	0.0	41.6	82.7	0.0	13.7	28.0	12.8
Tanaissus sp. A	33	375.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Paranaitis speciosa	34	365.4	0.0	0.0	16.0	0.0	5.3	9.3	0.0	0.0	16.0	18.7	0.0	6.9	11.5	65.1
Amphiporus bioculatus	35	325.9	0.0	21.3	8.0	14.4	0.0	0.0	2.7	0.0	0.0	5.3	0.0	0.0	0.0	0.0
Mercenaria mercenaria	36	317.5	2.0	6.0	4.0	8.0	0.0	1.0	0.0	44.0	3.0	24.3	8.1	8.0	19.9	2.0
Acteocina canaliculata	37	304.9	32.0	0.0	0.0	24.0	0.0	8.0	2.7	32.0	0.0	0.0	0.0	14.4	8.3	0.0
Acanthohaustorius similis	38	303.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Hypereteone sp.	39	298.0	0.0	69.3	8.0	8.0	5.3	4.7	4.0	32.0	57.6	16.0	8.0	11.8	5.3	5.3
Pagurus longicarpus	40	290.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	5.3	4.0	0.0	0.0	10.7
Spiophanes bombyx	41	279.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Apoprionospio pygmaea	42	276.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Rhepoxynius epistomus	43	273.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Myrianida prolifera	44	267.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	32.0	0.0	10.7	10.7	0.0	21.8	0.0
Turbellaria sp. 1 NAI	45	267.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Glycera dibranchiata	46	259.5	16.0	112.0	0.0	0.0	5.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Tritia obsoleta	47	255.9	0.0	0.0	0.0	0.0	10.7	2.0	4.5	0.0	67.2	0.0	0.0	13.7	7.9	5.3
Japonactaeon punctostriatus	48	245.3	0.0	0.0	0.0	0.0	0.0	0.0	2.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Brania wellfleetensis	49	232.4	0.0	0.0	0.0	6.4	0.0	0.0	0.0	0.0	0.0	8.0	0.0	0.0	0.0	0.0
Opisthodonta longocirrata	50	228.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	16.0	0.0	0.0	0.0	0.0	0.0	0.0

[1] Totals are fractional values because they reflect laboratory sub-sampling multiplication factors

Table B-15 Summary of Results for Benthic Species Analysis (50 Most Abundant Species)

	Rank	Total Number of Individuals[1]	VC15	VC16	VC17	VC18	VC19	VC20	VC21	VC22	VC23	VC24	VC25	VC26	VC27	VC28
Mediomastus ambiseta	1	93041.8	9840.0	3800.0	1507.7	4224.0	2474.7	9376.0	3470.2	5952.0	1782.4	1059.5	1385.3	2.1	1474.7	718.3
Streblospio benedicti	2	15020.7	896.0	21.3	8.0	490.7	160.0	298.7	588.4	1056.0	419.2	157.6	292.3	1.1	242.7	11.2
Oligochaeta	3	8553.2	128.0	181.3	86.9	133.3	69.3	186.7	241.8	560.0	36.8	85.6	302.9	5.3	269.3	143.2
Cirratulidae	4	3706.4	48.0	0.0	0.0	0.0	0.0	0.0	0.0	32.0	0.0	4.6	0.0	0.0	32.0	12.8
Glycinde solitaria	5	3268.7	128.0	88.0	33.6	176.0	290.7	197.3	124.4	80.0	41.6	139.8	49.3	0.0	50.7	99.5
Polygordius jouinae	6	2814.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	14.0	0.0	0.0
Exogone dispar	7	2565.4	64.0	0.0	8.0	90.7	130.7	240.0	56.9	368.0	147.2	36.9	128.0	96.3	178.7	86.1
Ameritella agilis	8	2199.7	16.0	8.0	0.0	0.0	21.3	10.7	30.2	64.0	35.2	26.4	30.7	29.1	32.0	125.5
Ampelisca abdita	9	1942.1	32.0	754.7	152.5	0.0	8.0	0.0	16.0	0.0	12.8	4.0	0.0	1.1	0.0	0.0
Sabellaria vulgaris	10	1798.7	16.0	0.0	0.0	48.0	0.0	16.0	14.2	192.0	67.2	6.4	0.0	0.0	0.0	0.0
Tharyx acutus	11	1330.9	16.0	0.0	0.0	0.0	0.0	0.0	40.9	32.0	14.4	24.1	25.1	0.0	16.0	5.9
Eumida sanguinea	12	1312.1	144.0	0.0	8.0	69.3	8.0	0.0	10.7	224.0	99.2	20.1	6.4	10.7	8.0	6.4
Unciola serrata	13	1147.5	0.0	0.0	0.0	32.0	0.0	64.0	24.9	0.0	8.0	27.5	18.1	0.0	26.7	0.0
Maldanidae	14	1128.3	80.0	0.0	3.2	48.0	0.0	64.0	0.0	96.0	20.8	12.8	4.0	1.0	0.0	0.0
Protohaustorius cf. wigleyi [In LeCroy, 2002]	15	1126.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Parametopella cypris	16	1055.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Euclymene collaris	17	955.5	96.0	0.0	0.0	26.7	317.3	21.3	28.4	0.0	0.0	51.0	18.1	0.0	13.3	41.5
Stenothoe minuta	18	900.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Caulleriella venefica	19	872.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	5.3	4.1	10.7	0.0
Glycera americana	20	758.7	96.0	10.7	0.0	16.0	61.3	10.7	10.7	64.0	12.8	31.5	0.0	0.0	10.7	7.9
Tritia trivittata	21	703.6	0.0	16.0	16.0	21.3	10.7	48.0	16.0	32.0	8.0	13.1	9.3	18.9	5.3	40.4
Tubulanus pellucidus	22	654.2	0.0	34.7	6.4	16.0	58.7	154.7	0.0	0.0	0.0	8.0	0.0	0.0	18.7	19.3
Heteromastus filiformis	23	551.0	32.0	0.0	0.0	10.7	21.3	154.7	28.4	0.0	12.8	24.6	0.0	0.0	0.0	3.2
Crepidula fornicata	24	527.7	16.0	0.0	0.0	0.0	0.0	0.0	46.2	48.0	22.4	20.8	0.0	0.0	5.3	0.0
Cyathura burbancki	25	524.2	32.0	0.0	0.0	0.0	0.0	0.0	0.0	16.0	8.0	8.0	0.0	3.0	5.3	2.7
Spisula solidissima	26	491.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Polydora cornuta	27	484.2	32.0	0.0	0.0	0.0	10.7	26.7	30.2	80.0	0.0	6.4	0.0	0.0	0.0	0.0
Nucula proxima	28	475.7	0.0	0.0	11.7	0.0	24.0	0.0	0.0	0.0	0.0	0.0	5.3	0.0	0.0	36.0
Polycirrus sp.	29	444.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	32.0	0.0	0.0	5.3	68.1	5.3	0.0
Ericthonius brasiliensis	30	411.3	0.0	0.0	0.0	0.0	10.7	0.0	0.0	32.0	0.0	0.0	5.3	0.0	5.3	10.0
Gyptis vittata	31	404.2	0.0	16.0	8.0	26.7	0.0	10.7	0.0	16.0	0.0	0.0	6.4	0.0	5.3	2.7
Oxydromus obscurus	32	386.1	16.0	0.0	0.0	26.7	0.0	0.0	0.0	48.0	20.8	0.0	0.0	2.1	0.0	0.0
Tanaissus sp. A	33	375.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Paranaitis speciosa	34	365.4	32.0	0.0	0.0	37.3	10.7	0.0	53.3	16.0	8.0	4.6	5.3	0.0	5.3	0.0
Amphiporus bioculatus	35	325.9	0.0	0.0	22.4	26.7	0.0	0.0	0.0	0.0	0.0	0.0	5.3	4.3	13.3	0.0
Mercenaria mercenaria	36	317.5	19.0	0.0	0.0	8.0	7.0	3.0	3.0	66.0	6.4	1.0	1.0	1.0	1.0	2.0
Acteocina canaliculata	37	304.9	0.0	8.0	5.3	10.7	8.0	10.7	48.0	0.0	0.0	12.8	0.0	0.0	0.0	64.0
Acanthohaustorius similis	38	303.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Hypereteone sp.	39	298.0	0.0	26.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.1	0.0	0.0
Pagurus longicarpus	40	290.4	16.0	0.0	0.0	0.0	0.0	16.0	0.0	32.0	8.0	0.0	0.0	0.0	16.0	9.6
Spiophanes bombyx	41	279.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	5.3	1.0	0.0	2.7
Apoprionospio pygmaea	42	276.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	6.4	0.0	0.0	0.0	0.0	0.0
Rhepoxynius epistomus	43	273.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Myrianida prolifera	44	267.0	32.0	0.0	0.0	0.0	8.0	0.0	0.0	32.0	35.2	11.0	0.0	0.0	0.0	2.0
Turbellaria sp. 1 NAI	45	267.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Glycera dibranchiata	46	259.5	0.0	0.0	0.0	0.0	0.0	0.0	16.0	16.0	0.0	8.6	10.7	3.1	10.7	6.4
Tritia obsoleta	47	255.9	32.0	8.0	3.2	16.0	85.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Japonactaeon punctostriatus	48	245.3	0.0	56.0	74.7	0.0	32.0	32.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	16.0
Brania wellfleetensis	49	232.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	8.6	26.7	0.0
Opisthodonta longocirrata	50	228.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	21.4	5.3	0.0

[1] Totals are fractional values because they reflect laboratory sub-sampling multiplication factors

Table B-15 Summary of Results for Benthic Species Analysis (50 Most Abundant Species)

	Rank	Total Number of Individuals[1]	VC29	VC30	VC31ALT	VC32	VC33ALT	VC34	VC35	VC36	VC37	VC38	VC39	VC40	VC41ALT	VC42
Mediomastus ambiseta	1	93041.8	311.8	637.3	1628.8	369.6	112.0	167.4	192.0	206.4	475.2	1453.3	682.7	458.7	240.0	0.0
Streblospio benedicti	2	15020.7	91.2	0.0	21.3	26.8	0.0	20.6	57.4	30.4	214.4	217.3	720.0	889.3	1096.0	8.0
Oligochaeta	3	8553.2	191.1	64.2	117.3	262.5	1204.6	103.8	219.7	33.2	200.5	468.0	90.7	180.0	92.8	12.2
Cirratulidae	4	3706.4	81.4	6.1	6.4	14.8	1391.2	414.1	194.9	49.2	81.6	238.7	5.3	65.3	833.6	117.2
Glycinde solitaria	5	3268.7	25.1	74.9	3.2	0.0	0.0	0.0	0.0	2.0	13.3	8.0	10.7	9.3	0.0	0.0
Polygordius jouinae	6	2814.4	0.0	10.7	29.9	50.4	373.3	130.4	275.7	62.8	19.7	8.0	5.3	13.3	163.2	277.0
Exogone dispar	7	2565.4	13.3	2.3	3.2	8.2	0.0	1.8	4.0	0.0	5.3	8.0	0.0	0.0	0.0	0.0
Ameritella agilis	8	2199.7	61.5	49.0	32.0	24.0	46.5	23.4	4.0	60.0	86.9	149.3	170.7	124.0	224.0	24.0
Ampelisca abdita	9	1942.1	0.0	4.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	10.7	4.0	0.0	0.0
Sabellaria vulgaris	10	1798.7	0.0	3.2	0.0	0.0	0.0	0.0	6.6	22.4	96.0	84.0	0.0	36.0	926.4	107.2
Tharyx acutus	11	1330.9	18.7	0.0	0.0	0.0	135.6	89.5	103.4	59.6	32.5	40.0	10.7	36.0	472.0	47.4
Eumida sanguinea	12	1312.1	14.4	32.0	0.0	18.3	5.3	11.8	21.1	25.6	59.7	66.7	5.3	16.0	64.0	0.0
Unciola serrata	13	1147.5	0.0	3.4	9.6	1.8	5.3	9.9	35.7	204.0	206.4	90.7	0.0	34.7	198.4	42.6
Maldanidae	14	1128.3	1.8	1.1	0.0	0.0	0.0	0.0	10.6	0.0	22.4	37.3	170.7	48.0	86.4	0.0
Protohaustorius cf. wigleyi [In LeCroy, 2002]	15	1126.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Parametopella cypris	16	1055.8	0.0	0.0	0.0	0.0	864.0	28.2	0.0	0.0	0.0	0.0	0.0	0.0	12.8	146.8
Euclymene collaris	17	955.5	30.4	9.9	10.7	7.2	0.0	19.7	10.6	0.0	18.7	0.0	0.0	0.0	0.0	0.0
Stenothoe minuta	18	900.8	0.0	0.0	0.0	0.0	672.0	4.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	222.2
Caulleriella venefica	19	872.9	9.1	63.6	145.1	44.9	24.4	80.9	107.1	24.8	11.7	0.0	0.0	0.0	40.0	129.6
Glycera americana	20	758.7	16.2	3.4	6.4	7.2	0.0	4.6	4.0	21.6	38.4	26.7	37.3	10.7	16.0	0.0
Tritia trivittata	21	703.6	6.2	18.8	19.2	4.0	22.1	6.4	18.0	32.0	34.1	32.0	16.0	36.0	68.8	1.0
Tubulanus pellucidus	22	654.2	40.2	49.6	24.5	0.0	0.0	4.6	10.0	0.0	41.6	97.3	26.7	4.0	8.0	0.0
Heteromastus filiformis	23	551.0	6.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	8.0	0.0	0.0	0.0	8.0	0.0
Crepidula fornicata	24	527.7	19.2	0.0	3.2	0.0	0.0	0.0	0.0	0.0	27.7	250.3	0.0	5.3	0.0	24.0
Cyathura burbancki	25	524.2	0.0	0.0	0.0	0.0	0.0	1.8	4.6	0.0	17.1	0.0	0.0	0.0	33.6	0.0
Spisula solidissima	26	491.6	0.0	0.0	0.0	0.0	4.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	4.0
Polydora cornuta	27	484.2	0.0	0.0	0.0	0.0	0.0	0.0	2.0	0.0	5.3	10.7	0.0	0.0	0.0	0.0
Nucula proxima	28	475.7	0.0	16.0	0.0	0.0	0.0	0.0	0.0	0.0	16.0	74.7	208.0	0.0	0.0	0.0
Polycirrus sp.	29	444.2	0.0	22.6	42.7	42.5	35.0	37.6	67.7	5.2	5.3	0.0	0.0	0.0	28.8	16.2
Ericthonius brasiliensis	30	411.3	0.0	0.0	0.0	0.0	320.0	10.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	16.0
Gyptis vittata	31	404.2	0.0	2.9	10.7	0.0	0.0	0.0	2.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Oxydromus obscurus	32	386.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	2.0	0.0	0.0	0.0	0.0	0.0	0.0
Tanaissus sp. A	33	375.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Paranaitis speciosa	34	365.4	3.6	0.0	3.2	0.0	0.0	0.0	4.0	0.0	0.0	14.7	10.7	0.0	8.0	0.0
Amphiporus bioculatus	35	325.9	0.0	8.4	6.4	8.0	120.4	18.9	8.6	6.0	0.0	0.0	10.7	0.0	12.8	0.0
Mercenaria mercenaria	36	317.5	1.0	0.0	3.0	14.5	0.0	4.6	1.0	0.0	0.0	42.7	1.0	0.0	0.0	0.0
Acteocina canaliculata	37	304.9	0.0	16.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Acanthohaustorius similis	38	303.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	16.0	0.0
Hypereteone sp.	39	298.0	0.0	0.0	10.7	0.0	0.0	7.1	0.0	0.0	0.0	0.0	0.0	0.0	16.0	0.0
Pagurus longicarpus	40	290.4	6.8	21.2	9.6	4.0	0.0	0.0	0.0	6.0	19.2	10.7	10.7	0.0	54.4	0.0
Spiophanes bombyx	41	279.0	15.3	8.4	13.9	0.0	2.3	2.7	4.0	22.4	10.7	8.0	16.0	24.0	54.4	32.0
Apoprionospio pygmaea	42	276.7	9.1	6.4	0.0	0.0	0.0	0.0	0.0	9.2	8.0	37.3	80.0	34.7	0.0	0.0
Rhepoxynius epistomus	43	273.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	3.2
Myrianida prolifera	44	267.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	4.0	11.7	0.0	0.0	0.0	48.0	8.0
Turbellaria sp. 1 NAI	45	267.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Glycera dibranchiata	46	259.5	5.3	7.8	3.2	1.8	2.3	0.0	2.0	2.0	5.3	0.0	5.3	14.7	0.0	0.0
Tritia obsoleta	47	255.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Japonactaeon punctostriatus	48	245.3	0.0	32.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Brania wellfleetensis	49	232.4	0.0	0.0	3.2	82.5	54.1	9.8	33.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Opisthodonta longocirrata	50	228.3	0.0	0.0	3.2	4.0	100.6	19.8	40.0	2.0	0.0	0.0	0.0	0.0	12.8	3.2

[1] Totals are fractional values because they reflect laboratory sub-sampling multiplication factors

Table B-15 Summary of Results for Benthic Species Analysis (50 Most Abundant Species)

	Rank	Total Number of Individuals[1]	VC43	VC44	VC45ALT	VC46	VC47	VC48ALT	VC49	VC50	VC51	VC52	VC53	VC54	VC55	VC56
Mediomastus ambiseta	1	93041.8	3.0	2.0	0.0	3.0	0.0	2.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Streblospio benedicti	2	15020.7	4.0	2.0	0.0	2.0	1.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Oligochaeta	3	8553.2	7.0	6.0	14.0	7.0	6.0	1.0	0.0	0.0	2.0	0.0	0.0	0.0	11.0	75.0
Cirratulidae	4	3706.4	14.0	3.0	5.0	0.0	1.0	0.0	0.0	0.0	2.0	0.0	0.0	0.0	1.0	0.0
Glycinde solitaria	5	3268.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Polygordius jouinae	6	2814.4	152.0	79.0	36.0	85.0	31.0	0.0	0.0	3.0	34.0	0.0	1.0	73.4	5.0	19.0
Exogone dispar	7	2565.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Ameritella agilis	8	2199.7	68.0	4.0	0.0	1.0	2.0	0.0	0.0	2.0	0.0	0.0	0.0	4.2	1.0	8.0
Ampelisca abdita	9	1942.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Sabellaria vulgaris	10	1798.7	2.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Tharyx acutus	11	1330.9	5.0	1.0	2.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Eumida sanguinea	12	1312.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Unciola serrata	13	1147.5	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Maldanidae	14	1128.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Protohaustorius cf. wigleyi [In LeCroy, 2002]	15	1126.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Parametopella cypris	16	1055.8	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Euclymene collaris	17	955.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Stenothoe minuta	18	900.8	2.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Caulleriella venefica	19	872.9	16.0	7.0	4.0	4.0	0.0	0.0	0.0	0.0	1.0	0.0	0.0	2.6	0.0	0.0
Glycera americana	20	758.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Tritia trivittata	21	703.6	1.0	1.0	1.0	0.0	1.0	0.0	0.0	0.0	3.0	0.0	0.0	1.0	0.0	0.0
Tubulanus pellucidus	22	654.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Heteromastus filiformis	23	551.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Crepidula fornicata	24	527.7	5.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0
Cyathura burbancki	25	524.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Spisula solidissima	26	491.6	49.0	21.0	38.0	26.0	23.0	23.0	11.0	13.0	25.0	33.0	3.0	1.0	5.0	11.0
Polydora cornuta	27	484.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Nucula proxima	28	475.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Polycirrus sp.	29	444.2	0.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	0.0
Ericthonius brasiliensis	30	411.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Gyptis vittata	31	404.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Oxydromus obscurus	32	386.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Tanaissus sp. A	33	375.0	20.0	98.0	11.0	0.0	0.0	0.0	0.0	0.0	1.0	3.0	18.0	150.0	9.0	0.0
Paranaitis speciosa	34	365.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Amphiporus bioculatus	35	325.9	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Mercenaria mercenaria	36	317.5	0.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Acteocina canaliculata	37	304.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Acanthohaustorius similis	38	303.0	40.0	13.0	52.0	5.0	5.0	0.0	0.0	26.0	11.0	1.0	7.0	9.0	0.0	0.0
Hypereteone sp.	39	298.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Pagurus longicarpus	40	290.4	1.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0
Spiophanes bombyx	41	279.0	0.0	1.0	0.0	1.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Apoprionospio pygmaea	42	276.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Rhepoxynius epistomus	43	273.2	6.0	3.0	6.0	8.0	2.0	4.0	44.0	2.0	0.0	0.0	2.0	20.0	0.0	0.0
Myrianida prolifera	44	267.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Turbellaria sp. 1 NAI	45	267.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	4.0	260.0
Glycera dibranchiata	46	259.5	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Tritia obsoleta	47	255.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Japonactaeon punctostriatus	48	245.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Brania wellfleetensis	49	232.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Opisthodonta longocirrata	50	228.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

[1] Totals are fractional values because they reflect laboratory sub-sampling multiplication factors

Table B-15 Summary of Results for Benthic Species Analysis (50 Most Abundant Species)

	Rank	Total Number of Individuals[1]	VC57	VC58	VC59	VC60	VC61ALT	VC62	VC63	VC64	VC65	VC66	VC67	VC68	VC69ALT
Mediomastus ambiseta	1	93041.8	0.0	0.0	1.0	0.0	0.0	0.0	2.0	1.0	0.0	1.3	2.0	0.0	0.0
Streblospio benedicti	2	15020.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Oligochaeta	3	8553.2	125.0	13.0	12.0	8.0	3.0	2.0	3.0	7.0	7.0	13.7	16.0	24.0	10.0
Cirratulidae	4	3706.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	0.0	1.3	1.0	5.0	3.0
Glycinde solitaria	5	3268.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Polygordius jouinae	6	2814.4	327.0	60.0	28.0	20.0	5.0	1.0	52.0	143.0	132.0	35.3	18.0	23.0	5.0
Exogone dispar	7	2565.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Ameritella agilis	8	2199.7	3.0	1.0	83.0	1.0	0.0	19.0	24.0	21.0	16.0	88.7	55.0	131.0	27.0
Ampelisca abdita	9	1942.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Sabellaria vulgaris	10	1798.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Tharyx acutus	11	1330.9	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	2.0	0.0	4.0	4.0	2.0
Eumida sanguinea	12	1312.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Unciola serrata	13	1147.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Maldanidae	14	1128.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Protohaustorius cf. wigleyi [In LeCroy, 2002]	15	1126.7	0.0	2.0	0.0	0.0	52.0	92.0	74.0	6.0	83.0	192.7	210.0	185.0	230.0
Parametopella cypris	16	1055.8	0.0	2.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	0.0	0.0	0.0	0.0
Euclymene collaris	17	955.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Stenothoe minuta	18	900.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Caulleriella venefica	19	872.9	0.0	4.0	9.0	11.0	5.0	2.0	1.0	1.0	16.0	10.7	6.0	9.0	9.0
Glycera americana	20	758.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Tritia trivittata	21	703.6	0.0	0.0	0.0	1.0	0.0	0.0	3.0	6.0	4.0	2.7	1.0	0.0	0.0
Tubulanus pellucidus	22	654.2	0.0	0.0	0.0	0.0	0.0	0.0	3.0	2.0	3.0	1.0	3.0	11.0	3.0
Heteromastus filiformis	23	551.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Crepidula fornicata	24	527.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	1.0	0.0	0.0	0.0	0.0
Cyathura burbancki	25	524.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Spisula solidissima	26	491.6	36.0	34.0	14.0	13.0	3.0	0.0	28.0	37.0	6.0	4.3	19.0	4.0	3.0
Polydora cornuta	27	484.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Nucula proxima	28	475.7	0.0	0.0	0.0	0.0	0.0	0.0	1.0	0.0	1.0	15.7	10.0	44.0	7.0
Polycirrus sp.	29	444.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Ericthonius brasiliensis	30	411.3	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Gyptis vittata	31	404.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Oxydromus obscurus	32	386.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Tanaissus sp. A	33	375.0	3.0	9.0	21.0	20.0	12.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Paranaitis speciosa	34	365.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Amphiporus bioculatus	35	325.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	0.0	0.0	0.0	0.0
Mercenaria mercenaria	36	317.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Acteocina canaliculata	37	304.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Acanthohaustorius similis	38	303.0	0.0	34.0	16.0	20.0	43.0	5.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Hypereteone sp.	39	298.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	0.0	0.0
Pagurus longicarpus	40	290.4	0.0	2.0	0.0	0.0	0.0	0.0	1.0	0.0	1.0	17.3	3.0	1.0	3.0
Spiophanes bombyx	41	279.0	0.0	1.0	0.0	0.0	0.0	1.0	8.0	4.0	6.0	14.0	8.0	7.0	4.0
Apoprionospio pygmaea	42	276.7	0.0	0.0	0.0	0.0	0.0	0.0	9.0	38.0	29.0	4.7	4.0	0.0	1.0
Rhepoxynius epistomus	43	273.2	0.0	27.0	23.0	20.0	56.0	45.0	0.0	0.0	0.0	1.0	0.0	0.0	1.0
Myrianida prolifera	44	267.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Turbellaria sp. 1 NAI	45	267.0	3.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Glycera dibranchiata	46	259.5	0.0	0.0	0.0	0.0	0.0	0.0	1.0	0.0	0.0	1.0	0.0	0.0	2.0
Tritia obsoleta	47	255.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Japonactaeon punctostriatus	48	245.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Brania wellfleetensis	49	232.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Opisthodonta longocirrata	50	228.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

[1] Totals are fractional values because they reflect laboratory sub-sampling multiplication factors

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Table B-16 Benthic Biota Richness, Abundance, Diversity and Evenness at Alternative Route Stations

Station	Rep	Richness	Abundance	Diversity (H')	Evenness (P')
VC70	C	11	176.0	1.46	0.28
VC70	N	12	352.0	1.37	0.23
VC70	S	20	403.2	1.73	0.29
VC71	C	8	1512.4	0.77	0.10
VC71	N	10	2385.0	0.83	0.11
VC71	S	12	2944.0	0.83	0.10
VC72	C	16	5473.0	1.34	0.16
VC72	N	13	3413.3	1.28	0.16
VC72	S	21	4940.8	1.55	0.18
VC73ALT	C	26	1107.0	2.33	0.33
VC73ALT	N	12	1039.7	0.88	0.13
VC73ALT	S	18	1664.0	1.72	0.23
VC74	C	15	1712.0	1.69	0.23
VC74	N	15	1521.0	1.39	0.19
VC74	S	11	1497.6	1.39	0.19
VC75	C	19	486.0	1.98	0.32
VC75	N	18	690.0	1.37	0.21
VC75	S	17	148.0	2.11	0.42
VC76	C	7	269.3	1.07	0.19
VC76	N	18	942.8	1.49	0.22
VC76	S	17	460.0	1.50	0.25
VC77	C	17	686.4	1.40	0.21
VC77	N	12	601.3	0.91	0.14
VC77	S	10	941.7	0.75	0.11
VC78	C	21	281.6	2.09	0.37
VC78	N	16	90.0	2.20	0.49
VC78	S	19	532.2	1.48	0.24
VC79ALT	C	13	434.0	1.55	0.25
VC79ALT	N	15	462.0	1.70	0.28
VC79ALT	S	11	304.0	1.67	0.29
VC80	C	19	250.0	1.28	0.23
VC80	N	8	888.0	0.56	0.08
VC80	S	18	628.0	0.89	0.14
VC81	C	15	561.0	1.94	0.31
VC81	N	10	672.0	0.87	0.13
VC81	S	10	416.0	0.56	0.09
VC82	C	26	458.2	2.29	0.37
VC82	N	24	1034.0	1.35	0.19
VC82	S	13	1940.0	0.90	0.12
VC83ALT	C	26	390.2	2.15	0.36
VC83ALT	N	15	272.0	1.80	0.32
VC83ALT	S	27	199.9	2.84	0.54
VC84ALT	C	25	674.0	2.40	0.37
VC84ALT	N	22	447.3	2.32	0.38
VC84ALT	S	12	544.0	1.82	0.29
VC85	C	22	883.2	2.14	0.32
VC85	N	24	1508.6	2.16	0.30
VC85	S	15	1143.3	1.98	0.28
VC86ALT	C	19	743.3	1.88	0.28

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Table B-16 Benthic Biota Richness, Abundance, Diversity and Evenness at Alternative Route Stations

Station	Rep	Richness	Abundance	Diversity (H')	Evenness (P')
VC86ALT	N	19	1174.3	2.14	0.30
VC86ALT	S	24	994.0	2.29	0.33
VC87	C	34	743.6	2.23	0.34
VC87	N	23	452.0	2.08	0.34
VC87	S	28	559.7	2.68	0.42

Table B-17 Benthic Macroinvertebrate Taxonomic List for Alternative Route

Taxonomic Name	Frequency of Occurrence	Count (N=54)	Relative Abundance (%)
Acteocina canaliculata	11	146.1	0.2655
Alitta succinea	4	61.2	0.1113
Ameritella agilis	28	685.0	1.2444
Ampelisca abdita	21	3467.5	6.2991
Ampelisca vadorum	6	68.5	0.1244
Ampharete oculata	2	5.0	0.0091
Amphiporus bioculatus	12	47.3	0.0860
Asmunda elegantula	10	100.6	0.1827
Batea catharinensis	2	8.9	0.0161
Bivalvia	3	28.7	0.0521
Boonea bisuturalis	2	7.2	0.0131
Boonea impressa	1	6.4	0.0116
Brania wellfleetensis	10	517.3	0.9398
Caprella sp.	1	8.0	0.0145
Carinoma tremaphoros	1	1.5	0.0026
Caulleriella venefica	13	596.1	1.0828
Cerapus tubularis	1	4.6	0.0083
Chaetozone sp.	1	6.4	0.0116
Cirratulidae	8	387.6	0.7040
Crepidula fornicata	4	41.1	0.0746
Crepidula plana	4	27.6	0.0501
Cyathura burbancki	11	205.7	0.3737
Diopatra cuprea	3	22.0	0.0400
Drilonereis longa	8	46.1	0.0838
Dyspanopeus sayi	11	125.9	0.2287
Edotia triloba	6	33.2	0.0602
Edwardsia elegans	2	6.0	0.0109
Elasmopus levis	1	3.2	0.0058
Ensis leei	14	27.0	0.0490
Ericthonius brasiliensis	8	90.3	0.1639
Euclymene collaris	24	679.7	1.2348
Eumida sanguinea	20	445.5	0.8092
Eupleura caudata	2	6.9	0.0126
Exogone dispar	46	1835.1	3.3338
Fargoa bartschi	2	59.2	0.1075
Gastropoda	1	16.0	0.0291
Glycera americana	25	192.2	0.3491
Glycera dibranchiata	3	11.4	0.0206
Glycera sp.	3	15.2	0.0277
Glycinde solitaria	38	1367.4	2.4841
Gyptis vittata	16	85.7	0.1556
Heteromastus filiformis	10	89.0	0.1616
Hypereteone heteropoda	5	64.6	0.1173
Hypereteone sp.	12	239.7	0.4355

Table B-17 Benthic Macroinvertebrate Taxonomic List for Alternative Route

Taxonomic Name	Frequency of Occurrence	Count (N=54)	Relative Abundance (%)
Japonactaeon punctostriatus	12	154.8	0.2813
Jassa sp.	1	1.0	0.0018
Leitoscoloplos fragilis	3	9.7	0.0176
Leitoscoloplos sp.	13	67.7	0.1230
Libinia dubia	1	4.0	0.0073
Lyonsia arenosa	4	23.5	0.0426
Lyonsia sp.	5	57.5	0.1045
Lysianopsis alba	2	13.3	0.0242
Magelona sp.	2	4.7	0.0085
Maldanidae	13	214.3	0.3893
Mediomastus ambiseta	54	28598.7	51.9533
Mercenaria mercenaria	34	487.9	0.8864
Microphthalmus sczelkowii	4	11.9	0.0216
Monocorophium acherusicum	2	6.9	0.0126
Monocorophium sp.	1	1.6	0.0029
Monocorophium tuberculatum	5	27.9	0.0506
Mulinia lateralis	14	431.6	0.7840
Myrianida prolifera	7	182.0	0.3307
Nephtys incisa	1	2.0	0.0036
Nephtys picta	3	12.1	0.0220
Nereis sp.	1	16.0	0.0291
Nereis zonata	1	10.7	0.0194
Neverita duplicata	1	1.0	0.0018
Nucula proxima	17	179.0	0.3252
Nudibranchia	1	13.7	0.0249
Odontosyllis fulgurans	3	81.6	0.1482
Oligochaeta	39	3373.9	6.1292
Opisthodonta longocirrata	13	759.7	1.3800
Orbiniidae	1	5.3	0.0097
Oxydromus obscurus	11	200.9	0.3650
Oxyurostylis smithi	2	18.9	0.0344
Pagurus longicarpus	6	25.1	0.0455
Pagurus sp.	1	2.7	0.0048
Pandora glacialis	5	41.5	0.0754
Paracaprella tenuis	1	3.6	0.0065
Paranaitis speciosa	8	80.1	0.1455
Paraprionospio pinnata	9	57.6	0.1045
Parasabella microphthalma	1	1.6	0.0029
Pectinaria gouldii	6	29.8	0.0541
Pectinariidae	4	28.5	0.0518
Petricolaria pholadiformis	1	5.3	0.0097
Phoronis sp.	1	2.7	0.0048
Phyllodoce arenae	6	33.5	0.0609
Polycirrus sp.	13	297.2	0.5398

Table B-17 Benthic Macroinvertebrate Taxonomic List for Alternative Route

Taxonomic Name	Frequency of Occurrence	Count (N=54)	Relative Abundance (%)
Polydora cornuta	12	310.9	0.5649
Polygordius jouinae	9	118.9	0.2160
Rhepoxynius hudsoni	3	24.3	0.0442
Sabaco elongatus	12	74.7	0.1358
Sabellaria vulgaris	9	245.5	0.4460
Salvatoria clavata	8	106.7	0.1938
Scolelepis sp.	1	3.2	0.0058
Spio setosa	1	8.0	0.0145
Spiochaetopterus costarum oculatus	1	4.0	0.0073
Spiophanes bombyx	1	1.5	0.0026
Sthenelais boa	3	20.9	0.0379
Streblospio benedicti	35	5884.9	10.6907
Streptosyllis verrilli	4	36.8	0.0669
Stylochus ellipticus	3	12.8	0.0233
Syllidae	1	21.3	0.0388
Tharyx acutus	7	101.3	0.1841
Tritia obsoleta	9	66.0	0.1200
Tritia trivittata	26	281.7	0.5117
Tubulanus pellucidus	19	149.1	0.2708
Turbonillinae	1	5.3	0.0097
Unciola dissimilis	1	6.0	0.0109
Unciola serrata	9	60.4	0.1096
Urosalpinx cinerea	4	30.6	0.0555

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C SAP/QAPP

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TRANSCONTINENTAL GAS PIPE LINE COMPANY, LLC

OFFSHORE SAMPLING AND ANALYSIS PLAN / QUALITY ASSURANCE PROJECT PLAN

VERSION 1.2

NORTHEAST SUPPLY ENHANCEMENT PROJECT

JUNE 2017

PUBLIC

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OFFSHORE SAMPLING AND ANALYSIS PLAN / QUALITY ASSURANCE PROJECT PLAN

REVISIONS TO VERSION 1.2

Page numbering was updated throughout.

Section 1.1, Table 1.1, page 2 and 3
Missing contractor information is provided for the Primary and Secondary Geotechnical Laboratory Contractors, and the Secondary Benthic Laboratory Contractor.

Section 1.2, page 4
Mileage of the Raritan Bay Loop has been updated.

Section 1.2.1, page 5
Information regard dredging of the Chapel Hill Channel has been added based a Freedom of Information Act request submitted to the USACE.

Section 1.3, page 7
The dates of pre-construction sampling and analysis activities have been updated.

Section 2.2.1, page 21
The depth discussed in reference to conducting a cone penetrometer test has been updated.

Section 2.2.2, page 21
During the survey, collection of vibracores for sediment chemistry analysis deviated from the previous version of the Sampling and Analysis Plan / Quality Assurance Project Plan (SAP/QAPP) in that certain samples were collected using a rigid plastic (polycarbonate) tube liner rather than a flexible polyethylene liner due to problems encountered with collapse of the flexible liner and retention of sample. The text has been updated to represent this modification of procedures.

Appendix B, Table 1A and Table 1B
In the previous version of the SAP/QAPP, sample site labels in Table 1B were incorrectly identified as "VCA". The table has been corrected.

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LIST OF ACRONYMS

ASTM	American Society for Testing and Materials
BTEX	benzene, toluene, ethylbenzene, xylene
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	chain-of-custody
CPT	cone penetrometer test
DQO	data quality objectives
E & E	Ecology and Environment, Inc.
ELAP	Environmental Laboratory Accreditation Program
EPA	U.S. Environmental Protection Agency
FTP	file transfer protocol
GPS	global positioning system
HDD	horizontal directional drill
IDW	investigation-derived waste
kg	kilogram
LCS	laboratory control sample
mg	milligram
mL	milliliter
MLLW	mean lower low water
MP	milepost
MS/MSD	matrix spike/matrix spike duplicate
M&R	meter and regulating
NELAP	National Environmental Laboratory Accreditation Program
NJDEP	New Jersey Department of Environmental Protection
NYSDEC	New York State Department of Environmental Conservation
PAH	polycyclic aromatic hydrocarbon
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
PCB	polychlorinated biphenyl
pdf	portable document file
Project	Northeast Supply Enhancement Project
QA/QC	quality assurance/quality control
RPD	relative percent difference
RDL	Rockaway Delivery Lateral

RTK	real time kinetic
RTS	[Neptune] Regional Transmission System
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan
SOP	Standard Operating Procedure
SPT	standard penetration test
SVOCs	semi-volatile organic compounds
TOC	total organic carbon
TOGS	Technical and Operational Guidance Series
Transco	Transcontinental Gas Pipe Line Company, LLC
USGS	U.S. Geological Survey
VOCs	volatile organic compounds

1 PROJECT MANAGEMENT

This combined Offshore Sampling and Analysis Plan and Quality Assurance Project Plan (SAP/QAPP) has been prepared by Ecology and Environment, Inc. (E & E) for Transcontinental Gas Pipe Line Company, LLC (Transco) in support of the Northeast Supply Enhancement Project (Project). This SAP/QAPP is applicable to any offshore sediment or water sampling activities and associated field or laboratory analyses that generate data that may be submitted to state or federal agencies to support the regulatory evaluation of the Project. This SAP/QAPP does not apply to offshore hydrographic (remote-sensing) survey activities.

This SAP/QAPP presents the policies, organization, objectives, functional activities, and specific quality assurance/quality control (QA/QC) procedures that will be used by Transco to help ensure that all offshore technical sampling data generated are accurate, representative, and ultimately capable of withstanding judicial scrutiny. The SAP/QAPP has been prepared in accordance with the *United States Environmental Protection Agency (EPA) Requirements for Quality Assurance Project Plans*, EPA QA/R-5 (March 2001) and is formatted to address the four major sections listed in this 2001 guidance document: Project Management, Data Generation and Acquisition, Assessment and Oversight, and Data Validation and Usability.

1.1 Project Organization

In general, the sampling and field analyses will be conducted by the sampling contractor, Alpine Ocean Seismic Survey, Inc. (Alpine) and overseen by Transco representatives, likely including staff from Transco's consultants – E & E and INTECSEA (a division of the Worley Parsons Group). Samples will be processed on the sampling vessel and conveyed to the laboratories by the sampling contractor or Transco's representatives. It is anticipated that geotechnical data and information from the sampling contractor and geotechnical laboratory will be delivered simultaneously to Transco and INTECSEA (and/or Lake Superior Consulting), while chemical/benthic data and information from the sampling contractor and chemical/benthic laboratories will be delivered simultaneously to Transco and E & E. It is expected that INTECSEA will review and process the geotechnical data and laboratory report(s), then provide a summary geotechnical report to Transco for review and approval prior to final application for Project design and construction purposes. It is also expected that E & E will review and process the chemical and benthic laboratory reports, then provide a summary environmental report to Transco for review and approval prior to submittal to regulatory agencies. Table 1.1-1 below identifies points-of-contact and their responsibilities with respect to the offshore sampling and analysis activities.

Table 1.1-1
Key Offshore Sampling and Analysis Personnel

Name	Role	Responsibilities
Transco		
Chris Martinez	Offshore Project Engineering Lead	Overall authority for application of geotechnical data and reports to offshore pipeline design and construction.
Webb Winston	Offshore Engineer	Final QA review and approval of geotechnical data and reports prior to application for offshore pipeline design for Project
Scott Horner	Offshore Environmental Manager	Final QA review and approval of environmental reports (including chemical and benthic analyses) prior to submittal to regulatory agencies.
Ecology & Environment, Inc.		
Sara Mochrie	Consulting Environmental Project Manager	Overall responsibility for chemical/benthic sampling and analysis activities.
Dave Albers, P.E. (NY, NJ)	Lead Environmental Engineer	Provides QA review of environmental reports.
Marcia Galloway, CQA, CQM	Quality Manager	Overall QA oversight of chemical and benthic sampling and analysis.
Steven MacLeod	Offshore Technical Lead	Responsible for developing environmental sampling and analysis report.
Nischint Sundar	System Controls Lead	Responsible for managing chemical/benthic sediment survey data and report files.
Gene Florentino, P.G., PMP	Lead Project Geologist	Responsible for field oversight of sediment sampling.
Lynne Parker	Lead Project Chemist	Responsible for QA review of chemical analysis data.
David Trimm	Lead Project Marine Biologist	Responsible for QA review of benthic analysis data.
INTECSEA / Worley Parsons Group		
Vance Nixon	Consulting Engineering Project Manager	Overall responsibility for geotechnical sediment sampling and design activities.
Mike Paulin	Principal Geotechnical Engineer	Jointly responsible for QA oversight on geotechnical investigations and providing technical information on geotechnical considerations to the Williams/Transco offshore pipeline design and construction teams.
Bernard Remmes	Principal Geotechnical Engineer	
Lake Superior Consulting		
Paul Beardon	Consulting Project Director	Responsible for QA review of geotechnical sediment sampling and design activities with respect to horizontal directional drill (HDD) activities.
Riley Shuoo	Engineering Supervisor	Responsible for review of geotechnical data and providing technical information with respect to HDD activities.
Sampling Contractor / Alpine		
Mark Kosakowski	Operations Manager	Responsible for development of the survey vessel plan and client/agency communications.
Chuck Dill	Party Chief and Senior Geologist	Overseeing mobilization and demobilization, testing, and equipment trials. Supervise survey operations, including QA and sample transfer to laboratories. Complete daily reports and other QA documents.
Laboratory Contractor (Geological - Primary) / Terra Sense, LLC		
Rosella Thomas	Geotechnical Laboratory Managing Member	Overall responsibility for geotechnical data QA review and development of primary geotechnical laboratory report.

Table 1.1-1
Key Offshore Sampling and Analysis Personnel

Name	Role	Responsibilities
Laboratory Contractor (Geological - Secondary) / Geotesting Express		
Steve Garmon	Geotechnical Laboratory Project Manager	Overall responsibility for geotechnical data QA review and development of primary geotechnical laboratory report.
Laboratory Contractor (Chemical - Primary) / Alpha Analytical		
Liz Porta	Chemical Laboratory Project Manager	Overall responsibility for development of chemical laboratory report.
James Todaro	Quality Assurance Officer	Responsible for overall laboratory QA protocols and QA review of sediment chemistry data.
Laboratory Contractor (Benthic - Primary) / Normandeau Associates		
Hannah Proctor	Biological Laboratory Project Manager	Overall responsibility for benthic data QA review and development of primary benthic laboratory report.
Laboratory Contractor (Benthic - Secondary) / ESS Group		
Matt Ladewig, CLM	Biological Laboratory Project Scientist	Responsible for overseeing analysis of QC samples.

1.2 Project Description/Background (Problem Definition)

Transco, a subsidiary of Williams Partners, L.P., is developing the Project to support National Grid's long-term growth, reliability, and flexibility beginning in the 2019/2020 heating season. The Project would provide up to 400,000 dekatherms per day of additional firm transportation capacity by adding to existing onshore Transco facilities in Pennsylvania and New Jersey, as well as expanding existing offshore facilities in New Jersey and New York waters. Transco anticipates that construction of the Project will begin mid-2018 to meet an in-service date in late 2019.

The offshore portion of the proposed Project includes a new 26-inch outer diameter pipeline that would extend from the Borough of Sayreville shoreline in Middlesex County, New Jersey, to the Rockaway Transfer Point, which is the interconnection point between Transco's existing Rockaway Delivery Lateral (RDL) and the Lower New York Bay Lateral (LNYBL) in New York waters. The new offshore pipeline, referred to as the "Raritan Bay Loop," would extend approximately 23.33 miles east-northeast across Raritan Bay and Lower New York Bay to the Rockaway Transfer Point, located in the Atlantic Ocean approximately 3 statute miles seaward of Rockaway, New York. From the Rockaway Transfer Point, the RDL supplies gas to an existing onshore meter and regulating (M&R) station located in Kings County, New York. Approximately 6.11 miles of the Raritan Bay Loop route crosses New Jersey waters, while the remaining 17.38 miles crosses New York waters. The route passes offshore of Monmouth County and Middlesex County, New Jersey, and Richmond County and Queens County, New York.

Figures showing the location of a proposed offshore study area are provided in Appendix A. This offshore study area is a 5,000-foot wide corridor centered along the preferred route that is expected to serve as the offshore workspace, i.e., the area in which construction vessels will operate and anchor. Thus, the total offshore study area along the 23.33-mile route is approximately 69.3 million square yards. In addition to the 502,682 square yards that may be disturbed during the excavation/dredging for the pipeline trench, three temporary pits will be dredged at the offshore pipeline horizontal directional drill (HDD) entry/exit points. The sizes of these pits may total roughly 12,000 square yards, and Transco plans to collect two vibracore samples for chemical analysis at the sample sites for these potential pit locations in anticipation of the greater disturbance volume (versus one vibracore sample for chemical analysis at all other sites). An additional amount of hand-jetting is expected near the Rockaway Transfer Point, but previous sediment sampling was conducted in this area in 2009/2010. The 2009/2010 Rockaway Delivery Lateral sample results were predominantly below the Class A thresholds identified in the New York State Department of Environmental Conservation (NYSDEC) Technical and Operational Guidance Series (TOGS) 5.1.9 document, In-Water and Riparian Management of Sediment and Dredged Material (2004), and none exceeded the Class B thresholds.

In response to agency comments, Transco extended the offshore study area to evaluate a route alternative in Raritan Bay near Staten Island. The figures in Appendix A include this study area extension.

To support the design and evaluation of the Project, Transco will collect and analyze geotechnical, chemical, and benthic characteristics of sediment located along the proposed offshore route. Certain site water properties will also be investigated to document conditions present during the sampling periods. To ensure that sediment sampling and analysis is sufficient for their review of an in-water dredge/fill project, NYSDEC and the New Jersey State Department of Environmental Protection (NJDEP) recommend that applicants submit a sampling and analysis plan for review before initiating sampling activities. The sampling design and analytical methods were developed based on a review of the environmental conditions and previous sampling data described below. This SAP/QAPP has been updated to incorporate NYSDEC and NJDEP input provided as of the latest revision date.

1.2.1 Environmental Setting

The proposed geotechnical study area will cross a continuous expanse of open marine and estuarine waters, which consists of three major waterbodies—Raritan Bay, Lower New York Bay, and the Atlantic Ocean. The Project area is located within the Monmouth watershed

management area and the Atlantic Ocean/Long Island Sound watershed, which drains most of the surrounding metropolitan areas and encompasses all marine waters in New York Harbor, Long Island Sound, Block Island Sound, Lower New York Bay/Raritan Bay, Sandy Hook Bay, and the waters that drain into them. The study area would cross three federally authorized navigation channels, including Ambrose Reach B (Ambrose Channel), Chapel Hill North (Chapel Hill Channel), and Raritan Bay West Reach (Raritan Bay Channel) (National Oceanic and Atmospheric Administration [NOAA] 2015). Ambrose Channel was dredged within the last 10 years as part of the U.S. Army Corps of Engineers' (USACE) overall New York/New Jersey Harbor deepening project. Based on a Freedom of Information Act request submitted to the USACE, Transco determined the Chapel Hill Channel was last dredged in March 2012. Transco has not yet determined when the Raritan Bay Channel was last dredged.

Measuring from mean lower low water (MLLW), water depths along the route range from 0 to 77 feet. The minimum water depth is 0 feet at the shore. A proposed HDD segment exits in approximately 8 feet of water approximately 1,800 feet from the shoreline. From the shoreline to the Raritan Bay Channel the water depth varies from approximately 0 feet to 20 feet. The authorized depth within the Raritan Bay Channel at the proposed crossing is 35 feet, although actual depths extend to 46 feet along the Project route. From the Raritan Bay Channel to the Chapel Hill Channel the water depth ranges between approximately 14 feet and 25 feet. The authorized depth within the Chapel Hill Channel at the proposed crossing is 30 feet, though actual depths extend to 35 feet. The water depth for the route from Chapel Hill Channel to Ambrose Channel varies from approximately 10 feet to 41 feet. The maximum water depth (77 feet) is located within the Ambrose Channel. From Ambrose Channel to the Rockaway Transfer Point the water depth ranges from approximately 15 feet to 40 feet.

1.2.2 Sediment Characteristics and Potential Contaminants of Concern

Several previous investigations have been completed within Raritan Bay and Lower New York Bay and extending into the Atlantic Ocean near the Rockaway Peninsula to identify the physical characteristics of the sediments as well as the degree to which contaminants may exist. Based on data collected by the U.S. Geological Survey (USGS) and available in USGS Open File Report OFR 03-241 and on recent investigations conducted by E & E as part of the Rockaway Delivery Lateral Project (E & E 2009, 2011), the predominant sediment type in this area has been identified as mostly fine-to-coarse sands. Greater silt and clay content was generally observed near the mouth of the Raritan River, the entrance to Upper New York Bay, and Sandy Hook Bay. However, a review of sediment sample data obtained in 2002 as part of the Neptune Regional

Transmission System (RTS) project (E & E 2003) indicates that material primarily classified as silt may also extend throughout Raritan Bay.

Additionally, the 2002 sediment sample data obtained during the Neptune RTS project indicated that a number of potential chemical contaminants had been identified in the vicinity of the proposed route for the Project (E & E 2003). Inorganic contaminants (i.e., metals) such as mercury, zinc, and arsenic were present, while organic contaminants such as polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) were detected to a much lesser degree. These sampling results are generally consistent with earlier sediment sampling in the vicinity of the Project (Adams and Benyi 2003; Adams et al. 1998), but there was a wide variation in results, depending upon sample location and depth. More recent investigations conducted by E & E as part of the Rockaway Delivery Lateral Project indicated that substrate immediately seaward of the Rockaway Peninsula was predominantly sandy with minimal contaminant levels (E & E 2009, 2011).

1.2.3 Benthic Community

The subsea conditions in the vicinity of the proposed Project are inherently dynamic and subject to regular disturbance, both anthropogenic and biogenic. Therefore, much of the benthic environment is inhabited by more adaptable fauna capable of rapid and prolific reproduction within silty and sandy environments. Data included in the Mid-Atlantic Ocean Data Portal indicate that the benthic environment within Raritan Bay is composed almost entirely of two benthic habitats—very shallow depression silt/mud and very shallow depression sand (depths ranging from 0 to 30 feet) (The Nature Conservancy 2010). Maps available from the NJDEP show that a segment of the proposed offshore route near the New Jersey shoreline will cross current or historical habitat for one or more species of shellfish, i.e., hard clam (*Mercenaria mercenaria*) and soft clam (*Mya Arenaria*) (Decanay 2016; NJDEP 2016). The proposed Project route will also cross a section of New York waters near Staten Island that is in a productive hard clam area (Barnes 2016). As the proposed route approaches Chapel Hill Channel and Ambrose Channel in Lower New York Bay, sediment size generally increases, and high gravel/sand flats and very shallow flat sand become the dominant types (The Nature Conservancy 2010). Near the Rockaway Transfer Point the substrate becomes more consistently sandy with relatively gradual slopes. Surveys conducted for Transco's Rockaway Delivery Lateral Project indicate that the Atlantic surfclam (*Spisula solidissima*) is the dominant shellfish species in this area (E & E 2009, 2011, 2015).

1.3 Sampling and Analysis Task Description

The goal of the sampling and analysis program is to support the design and evaluation of the Project. To achieve this goal at the pre-construction stage, the following major tasks will be performed:

1. Collect offshore sediment samples for geotechnical, chemical, and benthic analyses;
2. Evaluate physical/geotechnical properties of the sediment that could be disturbed during Project construction;
3. Identify the concentrations of chemical contaminants that are present in sediment that could be disturbed during Project construction; and
4. Characterize the macrobenthic invertebrate communities that may be affected by Project construction.

Figures showing the locations of the planned offshore sample sites are provided in Appendix A. Pre-construction sampling and analysis activities occurred between November and December, 2016.

1.4 Quality Goals and Objectives

Data quality objectives (DQOs) are qualitative and quantitative statements that clearly define the objectives of a project, define the most appropriate type of data, determine the appropriate procedures for data collection, and specify acceptable decision-error limits that establish the quantity and quality of data needed for decision-making. The Project's major sampling and analysis tasks are identified in Section 1.3 above. The general data quality objectives for each of these tasks are summarized in Table 1.4-1. Acceptance and performance criteria for field and analytical quality control samples are outlined in Section 2.

Acceptance and performance criteria are often specified in terms of precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) parameters. Numerical acceptance criteria cannot be assigned to all PARCCS parameters, which are briefly described below, but general performance goals are established for most data-collection activities. Data assessment procedures throughout this SAP/QAPP outline the steps to be taken, responsible individuals, and implications if QA objectives are not met. Measurement performance criteria, if applicable, were established following selection of the analytical laboratories (see Table 1.4-2).

Table 1.4-1
Data Quality Objects for Offshore Sampling and Analysis Tasks

Task	Data Quality Objective	Standards/Guidance[1]	Acceptance and Performance Criteria
Geotechnical Sampling and Analysis	Acquire geotechnical data sufficient to characterize offshore substrate for the purposes of engineering design, including the determination of suitable construction methods. Also determine sediment grain-size distribution as a basis for hydrodynamic modeling of potential Project-related sediment suspension and transport.	ASTM methods appropriate for sediment classification and each geotechnical test.	Criteria are based on the ASTM methods appropriate for each geotechnical test identified in Section 2 of this SAP/QAPP.
Sediment Chemistry Sample Collection and Analysis	Acquire sediment chemistry data (including basic physical properties) sufficient to characterize the potential level of contaminants within the sediment that may be disturbed during construction to assist in review of potential environmental effects of the Project.	NYSDEC TOGS 5.1.9 In-Water and Riparian Management of Sediment and Dredged Material (2004); NJDEP's The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters (1997) USACE/EPA Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal (2016)	Data must meet the acceptance and performance criteria documented in Section 2 of this SAP/QAPP.
Benthic Community Sample Collection and Analysis	Acquire macroinvertebrate population and surface sediment data sufficient to characterize the benthic community within the sediment that may be disturbed during construction to assist in review of potential environmental effects of the Project.	EPA National Coastal Condition Assessment 2015: Laboratory Operations Manual (EPA- 841-R-14-008)	For species identification, 90% concurrence between two independent laboratories.

Key:
ASTM = American Society for Testing and Materials
NJDEP = New Jersey Department of Environmental Protection
NYSDEC = New York State Department of Environmental Conservation
SAP/QAPP = Sampling and Analysis Plan / Quality Assurance Project Plan
TOGS = Technical and Operational Guidance Series

Notes:
(1) Actual procedures and criteria subject to Project-specific modification, as described in this SAP/QAPP.

Table 1.4-2
Summary of PARCCS Parameter Criteria

Sampling Parameter	Analytical Method	PARCCS Parameters	Measurement Performance Criteria
Geotechnical Analyses	See Section 2.4	Precision	Per ASTM methods
		Accuracy	Per ASTM methods
Inorganics/Metals	SW-846 6020A	Precision	MS/MSD or DUP RPD: ≤20%
		Accuracy	LCS: 75-125% MS/MSD: 75-125%
Mercury	SW-846 7474	Precision	MS/MSD or DUP RPD: ≤20%
		Accuracy	LCS: 80-120% MS/MSD: 80-120%
Volatiles	SW-846 8260C	Precision	MS/MSD RPD: 30%
		Accuracy	LCS: 70-130% MS/MSD: 70-130% For BTEX compounds and 1,4-dichlorobenzene.
Semi-volatiles	SW-846 8270D	Precision	MS/MSD RPD: ≤30%
		Accuracy	LCS and MS/MSD: Base/Neutral Compounds: 40-140% Acid Compounds: 30-130%
Pesticides	SW-846 8081B	Precision	MS/MSD RPD: ≤50%
		Accuracy	LCS: 40-140% MS/MSD: 40-140%
PCB Aroclors	SW-846 8082A	Precision	MS/MSD RPD: ≤50%
		Accuracy	LCS: 40-140% MS/MSD: 40-140%
PCB Congeners	EPA 1668C	Precision	MS/MSD RPD: ≤30%
		Accuracy	Recovery Native Analytes: 60-135% See laboratory SOP in Appendix D for Recoveries of Extraction Standards and Cleanup Standards.
Dioxins	SW-846 1613B	Precision	MS/MSD RPD: ≤20%
		Accuracy	LCS: In-house limits MS/MSD: In-house limits See laboratory SOP in Appendix D.
Cyanide	SW-846 9012	Precision	MS/MSD RPD: ≤35%
		Accuracy	LCS: 80-120% MS/MSD: ≤35%
TOC	EPA 9060A	Precision	MS/MSD RPD: ≤25%
		Accuracy	LCS: 75-125% MS/MSD: 75-125%

Key:
ASTM = American Society for Testing and Materials
BTEX = Benzene, toluene, ethylbenzene, xylene
LCS = Laboratory control samples
MS/MSD = Matrix spike/matrix spike duplicate
PARCCS = precision, accuracy, representativeness, completeness, comparability, and sensitivity (as described in Section 1.4)
RPD = Relative percent difference
SOP = Standard Operating Procedure

1.4.1 Precision

Precision measures the reproducibility of measurements under a given set of conditions. Specifically, it is a quantitative measure of the variability of a group of measurements compared with their average value, usually stated in terms of standard deviation or coefficient of variation. It may also be measured as the relative percent difference (RPD) between two values. Precision includes the interrelated concepts of instrument- or method-detection limits and multiple field sample variance. Sources of this variance are sample heterogeneity, sampling error, differences among observers, and analytical error. Communication between individuals in charge of sampling, via the field logbook or otherwise, should reduce any measurement- precision errors.

1.4.2 Accuracy

Accuracy measures the bias of the measurement system. Sources of this error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analysis. Data interpretation and reporting may also be significant sources of error. Typically, analytical accuracy is assessed through the analysis of spiked samples and may be stated in terms of percent recovery or the average (arithmetic mean) of the percent recovery. Blank samples may also be analyzed to assess sampling and analytical bias (i.e., sample contamination). Background measurements similarly assess measurement bias. The number of samples collected will affect the confidence of the statistical data evaluation.

1.4.3 Representativeness

Representativeness expresses the degree to which data represent a characteristic of a population, a parameter variation at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that is most concerned with proper design of the measurement program. Sample/measurement locations may be biased (judgmental) or unbiased (random or systematic). Representativeness of the sampling scheme will be determined with evaluation of the historical data and statistical evaluation of the results compared with the reference site or existing data.

1.4.4 Completeness

Completeness is defined as the percentage of measurements that are judged to be valid. Although a quantitative goal must be specified, the completeness goal is the same for all data uses — that a sufficient amount of valid data is generated. A completeness goal of 95% will be established for this Project.

1.4.5 Comparability

Comparability is a qualitative parameter that expresses the confidence with which one dataset may be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved through the use of standard techniques to collect and analyze samples. Historical data will be evaluated to ensure the methods and reporting limits are comparable to the proposed sampling. Data will only be evaluated if it is determined to be comparable.

1.4.6 Sensitivity

Sensitivity refers to the capability of a method or instrument to detect a given analyte concentration and reliably quantitate the analyte at that concentration. Analytical results for samples that are non-detect for a particular analyte that have reporting limits greater than the applicable standards and/or screening levels cannot be used to demonstrate compliance with the applicable standards and/or screening levels.

1.5 Special Training/Certification

The geotechnical field and laboratory investigations are expected to be managed by engineers licensed in New York and New Jersey. The geotechnical core borings are expected to be conducted by a well driller or soil borer licensed in New Jersey pursuant to Chapter 7:9D New Jersey Administrative Code. The laboratory conducting geotechnical analyses is expected to be certified by the American Society for Testing and Materials (ASTM) for testing soil and rock. The laboratory conducting sediment chemistry analyses will be certified by the New York State Department of Health (NYSDOH) Environmental Laboratory Accreditation Program (ELAP) for Solid and Hazardous Waste and Contract Laboratory Program and/or current certification by the National Environmental Laboratory Accreditation Program (NELAP) in New Jersey.

1.6 Documentation and Records

Data generated during the sampling and analysis activities will be documented through the formats listed in Table 1.6-1. Field records will be generated in hard copy field logs or on Project-specific datasheets (i.e., paper media), then scanned or transferred into electronic format. Electronic Project records will be maintained on internal, secure Project drives that are accessible only to the Project team.

The Project documents identified in this SAP/QAPP that will be submitted to regulatory agencies include the sediment chemistry and benthic community laboratory reports, which will be provided as appendices to an Offshore Environmental Sampling and Analysis Summary Report

(see Sections 1.6.2 and 1.6.3 below for further discussion). The core logs and/or the overall Geotechnical Site Investigation Report may also be provided to regulatory agencies, as warranted. As applicable, Project documents will be submitted to regulatory agencies as portable document format (pdf) files via e-mail, electronic media (i.e., CD or DVD), and/or posted on a password-protected file transfer protocol (FTP) site. Abridged hard copies (i.e., certain appendices may not be printed) will also be provided to agencies.

Table 1.6-1
Offshore Sampling and Analysis Data Records

Document Type	Format	Originator	Repository	Submittal Frequency
Field Logbooks	Hard copy and Electronic (pdf and/or Excel spreadsheet)	Sampling Contractor	Sampling Contractor	As needed to consultant (E & E or INTECSEA) or Transco
Progress Reports and Sediment Core Log Sheets	Hard copy and Electronic (pdf and/or Excel spreadsheet)	Sampling Contractor	Sampling Contractor and Consultant (E & E or INTECSEA)	Electronic form only: Daily to Consultant (E & E and/or INTECSEA) and Transco Hard copy: Included as appendix with field report.
Laboratory Reports	Hard copy (with exceptions)[1] and electronic (pdf and Excel spreadsheet or CSV)	Laboratories	Laboratories and Consultant (E & E or INTECSEA)	Preliminary and/or final sediment chemistry reports to consultant and Transco within 30 days of sample delivery. Preliminary and/or final benthic report to consultant and Transco within 60 days of sample delivery. Final sediment chemistry and benthic lab reports to regulatory agencies as part of survey summary report.[2]
Summary Reports	Hard copy and Electronic (pdf)	Sampling Contractor, Consultant (E & E)	Sampling Contractor and/or Consultant (E & E or INTECSEA)	Draft and final survey summary reports to Transco. Final environmental (benthic/chemistry) survey summary report(s) to regulatory agencies.[2]

Key:
CSV = comma separated value
E & E = Ecology and Environment, Inc.
Notes:
(1) Very large appendices may be submitted only in electronic format, at Transco's discretion.
(2) Reports submitted to regulatory agencies as part of license/permit applications will be retained by those agencies according to their policies and state/federal laws.

1.6.1 Field Documentation and Reporting

The sampling contractor will maintain a daily operations log that records the time and description of all significant events during the investigation, which will be made available to Transco on request. Based on the daily operations logs, the sampling contractor will prepare a daily progress report that includes the following:

1) Brief summary of work performed and narrative of events
2) Changes to scope
3) Disputed time
4) Delays, malfunctions, deviations, and / or special problems encountered
5) Weather
6) Safety incidents
7) Tally of events including working time, depth of corings, downhole tests performed, standby time, downtime and cause (i.e., weather, equipment or vessel), consumables
8) Percentage of Work scope completed
9) Vessel location
10) Brief outline of findings
11) Work planned for next 24-hour period and three-day look-ahead
12) Company and contractor comments.

The sampling contractor will provide the daily progress report to Transco by 08:00 a.m. (Central Time) the following day.

The sampling contractor and/or Transco's designated field geologist or geotechnical engineer will visually inspect all deep and shallow core samples, documenting measurements for individual horizons or strata in a field logbook.

Individual field logs will be made for each geotechnical core sample. Logs will include the following information:

1) Full information on position, coordinate system, and water depth
2) Length in feet of final penetration and sample recovery
3) Visual description of and length of each individual sample
4) Results of index and shear strength testing

For sediment chemistry core samples, basic information collected will be recorded on a sediment core log and will include the following:

1) Project and core sample identification numbers
2) Latitude/longitude coordinates of the sample location
3) Digital photographs of each sample
4) Type of equipment used to obtain the sample
5) Visual classification of each major sediment horizon or strata encountered in accordance with the United Soil Classification System
6) Water depth and sea state core penetration length
7) Core recovery length
8) Presence of debris, contaminants, or living organisms.

Sediment testing results completed in the field will be reported in accordance with the procedures as defined in the various standards for the applicable tests (see Section 2.4 for a list of testing methods). Sediment test results will be produced in a hard copy format with data represented in appropriate tables of results and graphs showing test results. All test results will be tabulated, the tabulated summary of test results will be formatted as a Microsoft (MS) Excel data file. All geotechnical core logs (hard copies and electronic files) will be presented at the same vertical scale. The sampling contractor will provide these data to Transco within 24 hours after completion of activities for each sample site.

Site information for surface sediment grab samples will also be recorded in a field logbook, including the actual sampling coordinates, sample number, water depth, stage of the tide, time, and the sea/weather conditions.

The sampling contractor will compile a Field Investigation Report of the field sampling and analysis activities. The sampling contractor will submit the draft field report to INTECSEA and Transco following completion of all their field activities, at the time of vessel demobilization. The field report will include, at a minimum, the following:

- Brief assessment of sampling activities performed;
- Set of preliminary core logs as produced in the field, including full-size photocopies for any interpretation logs that are hand-drawn;
- Summary log of core information (samples, descriptions, and results of onboard tests);
- Sampling activity summary that concisely highlights all significant features;
- Copy of the daily operations log (hard and digital); and
- Results of geotechnical field tests.

The sampling contractor will issue a final version of the Field Investigation Report following review and approval by INTECSEA, Transco, and/or Lake Superior Consulting.

1.6.2 Laboratory Data Reporting

The sampling contractor and/or INTECSEA will be responsible for obtaining results of geotechnical analyses from the onshore laboratory in a format that can be incorporated into the Geotechnical Site Investigation Report (see Section 1.6.3 below).

After completing the sediment chemistry laboratory analysis program, it is anticipated that the laboratory will submit a Chemistry Analytical Data Report to the sampling contractor and the sampling contractor will provide the reports to Transco and E & E for review. The laboratory will provide a full laboratory report (Level IV or Contract Laboratory Program [CLP]-like deliverable) including QA/QC documentation in pdf format, electronic data deliverables, and excel summary tables of all results. The laboratory will submit revised reports as necessary to address QA/QC deficiencies or errors.

After completing the benthic analyses, it is anticipated that the laboratory will submit a draft Benthic Community Sampling Report to the sampling contractor, and the sampling contractor will provide the reports to Transco and E & E for review. The document will include summary tables of all results (e.g., number and type of species) and any relevant laboratory QA/QC documentation. Some report appendices may be in electronic format only, depending on size and content. The laboratory will submit revised reports as necessary to address data deficiencies or errors.

1.6.3 Summary Reports

It is anticipated that the sampling contractor or INTECSEA will develop a Geotechnical Site Investigation Report(s). This report will include a summary of all information collected during the geotechnical investigation, the Field Investigation Report, and the results of the standard and any advanced geotechnical testing that takes place at an onshore laboratory. A draft version of the Geotechnical Site Investigation Report will be submitted to Transco and Lake Superior Consulting following completion of all geotechnical laboratory analyses. The report will include the following information:

- An overview of the deep (core boring) and shallow (vibracoring) core results, with highlights of all hazards, obstructions, constraints and significant anomalies encountered;

- Purpose and defining limits of geotechnical investigation task, dates of acquisition, brief description of utilized equipment and vessel;
- Discussion of local sediment stratigraphy and an interpretation of the local sediment properties;
- Laboratory test results, including 'grouping' summaries for similar facies;
- Description of positioning systems, calibrations, data reduction, results, and performance;
- Description of geotechnical acquisition systems, calibrations, data reduction, results, and performance; and
- Supporting data, including final core profiles, vessel specifications, coring systems, positioning and water-depth measurements, field-sampling procedures, field-sediment testing, drill logs, and daily progress reports, if not previously submitted in the field report.

The sampling contractor or INTECSEA will issue a final version of the Geotechnical Site Investigation Report following Transco's review and approval of final edits.

In addition to reporting on hard copy media, the sampling contractor will submit the final Geotechnical Site Investigation Report in electronic format, including charts, drawings, figures, and plots. The media and precise format will be confirmed during reporting, but it is anticipated to be a browseable CD or DVD with the report presented in pdf format and all data in Microsoft Excel tables. All charts and maps will be made available in hard copy and AutoCAD 2000-compatible digital format.

An Offshore Environmental Sampling and Analysis Summary Report that synthesizes the results of the sediment chemistry and benthic community laboratory reports will also be prepared by E & E for inclusion as part of Project license and permit application packages. E & E will provide a draft version of this summary report in electronic format to Transco for review and approval. Following Transco's approval, a final version of the Offshore Environmental Sampling and Analysis Summary Report will be submitted to agencies to provide a more concise description of chemical and benthic characteristics of the sediment for ease of agency review. This summary report will include the original laboratory reports as appendices.

1.6.4 Record Retention

Transco's sampling contractor will keep accurate logs and records of all sampling activities and will provide Transco with complete, legible copies of logs and records upon completion of activities at each sample site. The sampling contractor will preserve all records in good condition until they are delivered and deemed acceptable by Transco. Transco will have the right to examine such records at any time prior to delivery.

The laboratories will submit all data and laboratory reports to the sampling contractor, who will forward them to Transco and the appropriate consultant (E & E, INTECSEA, and/or Lake Superior Consulting). The laboratories will retain data and reports for a period of time dictated by their individual protocols and certifications. Laboratory data and reports will also be retained by the consultants for the minimum period identified in Table 1.6-1.

2 DATA GENERATION AND ACQUISITION

This section of the SAP/QAPP describes the design and implementation of field, laboratory, and data-handling procedures for the Project's offshore sampling activities. The SAP/QAPP provides the basis for ensuring that appropriate methods are used and thoroughly documented.

2.1 Sampling Process Design

Deep core samples extending up to 125 feet below the seafloor will be collected from up to 22 sample sites[1], all of which are within New Jersey waters. The deep core sample sites are identified as "BHA" in Table 1B in Appendix B, which presents the planned coordinates and core depths for each site, which have been approved by the New Jersey Historic Preservation Office. Sites identified with "-ALT" indicate those that have been shifted to avoid potential cultural resources. These deep core sites are located near the New Jersey shoreline and across Ambrose Channel, the two offshore areas where Transco proposes to install the Project pipeline using HDD. The deep core depths represent the maximum depths for HDD during the Project. Deep core samples will be analyzed for geotechnical properties only (see Table 1.4-1). The geotechnical data will be used for planning the routing of the HDD portion of the pipeline.

Shallow core samples will be collected at 87 sites, 15 of which are in New Jersey waters and 72 of which are in New York waters. Of these, 69 sample sites are centered along the

[1] Three additional core boring sites associated with the Raritan Bay Loop shore crossing are located onshore and are therefore not discussed in detail in this document.

preferred route, while the remainder are along a route alternative. The shallow core sample sites are identified as "VC" in Tables 1A and 1B in Appendix B, all of which have been reviewed and approved by the New Jersey Historic Preservation Office and New York State Historic Preservation Office, as appropriate. Sites identified with "-ALT" indicate those that have been shifted to avoid potential cultural resources. The shallow cores will extend at least 12 feet below the seafloor outside navigation channels and up to 30 feet below the seafloor near navigation channels and designated anchorage areas. The shallow core samples will be collected in areas where pipeline would be installed by trenching methods and where HDD pits would be excavated, potentially using a clamshell dredge, a mechanical plough, and/or a jet sled. Dredged material may be side-cast adjacent to the trench and, following pipeline installation, would be used to backfill the trench to the extent practicable.

The NYSDEC TOGS 5.1.9 document recommends applying Balduck's method for determining an appropriate number of sediment sample sites. Transco currently estimates that approximately 530,000 square yards of seafloor may be disturbed by trenching methods to install the pipeline along the entire offshore route (not including anchoring). An additional 12,000 square yards may be excavated for offshore HDD pits. Conservatively assuming a potential for contamination along the entire offshore route, a "dredge factor" of three would be applied to the Balduck equation, suggesting that 69 sample sites should be used to characterize the dredge area. This is consistent with the number of sample sites currently proposed in this SAP/QAPP.

Transco will collect samples to depths that reflect the maximum anticipated depth of disturbance during Project construction (see Appendix B, and Tables 1A and 1B), accounting for appropriate pipeline burial depths. The USACE New York District provided the following anticipated pipeline burial requirements for the Project based on information presented as of June 2016 (Handell 2016):

- Navigable waters located outside of designated channels and anchorage areas:
  - Minimum 4 feet of cover below present bottom in soft sediment
  - Minimum 2 feet of cover below present bottom in consolidated rock
- Designated navigation channels
  - Minimum burial 8 feet below authorized depth (including side-slopes), and
  - Minimum 4 feet of cover in soft sediment
- Designated (charted) anchorage grounds
  - Minimum 7 feet of cover below present bottom in soft sediment for un-maintained anchorage

- Minimum burial 11 feet below authorized (or actively maintained) depth for maintained (dredged) anchorage and minimum 7 feet of cover in soft sediment.

Shallow core samples will be collected for testing and analysis of both geotechnical and bulk sediment chemistry properties (see Table 1.4-1). Due to sample volume and analysis requirements, two separate shallow core samples will be collected at each shallow core sample site—one for geotechnical analyses and one for sediment chemistry analyses. In addition to these two shallow core samples, a third shallow core sample will also be collected for sediment chemistry analyses at the three shallow core sample sites that are closest to the anticipated HDD entry/exit locations (i.e., VC-1 ALT, VC-54, and VC-55). Thus, a total of 177 shallow core samples will be collected. Shallow core samples will be divided into sections at the time of collection. The size of the planned core sections will provide sufficient volume to allow for QA/QC analyses.

Surface sediment grab samples will be collected for analysis of benthic invertebrate populations as well as supplemental physical (grain size and total organic carbon [TOC]) analyses. Three surface sediment grab samples will be collected at each shallow core sample location identified as "VC" in Tables 1A and 1B in Appendix B, for a total of 270 sediment grab samples. Sufficient volume will be collected to allow for QA/QC analyses.

2.2 Sampling Methods

Summaries of offshore sampling methods are provided below. Unless otherwise noted in this SAP/QAPP, sampling procedures will substantially adhere to the applicable testing methods identified in Section 2.4. Sample sites will be located in the field using a real-time kinetic (RTK) differential global positioning system (GPS) equipment with an accuracy of approximately ± 3 feet. Water depths will be determined using a survey-grade echo sounder with an accuracy and precision of 3 feet or better.

2.2.1 Core Boring

A rotary drill system will be used to collect the deep core samples. Offshore core boring in deeper water (more than 20 feet) will use a jack-up barge with a drill rig mounted on the barge platform, working during daylight hours. The jack-up platform is lifted out of the water by jacking against the legs mounted on the platform. In shallow areas near the New Jersey shoreline, the drill rig used to collect deep cores may be mounted on a pontoon barge held in position with spuds or anchors.

A temporary 6-inch-diameter steel casing extending through the water column from the deck of the barge will be used to stabilize the sediments immediately below the seafloor; drilling

fluid additives will be used to stabilize the remaining depth of the borehole. The drilling fluid additives will consist primarily of attapulgite clay powder (e.g., ZeoGel) and possibly barite or guar gum, as necessary, to provide additional weight/density. Sediment samples will generally be taken at 5-foot intervals. The sediment samples will be obtained with a standard 2-inch outer-diameter split-spoon sampler, which is driven through four 6-inch intervals with a 140-pound hammer that freefalls for 30 inches. A standard penetration test (SPT) will be used to determine resistance at each sample interval, and the number of blows required to drive the sampler through each of the four 6-inch increments will be recorded, up to 100 blows (i.e., point of refusal). The procedures will generally adhere to ASTM D1586.

In uniformly cohesive soils, high quality (minimal sample disturbance) samples will be obtained utilizing 3-inch-diameter thin-walled Shelby tubes subsequent to obtaining a disturbed sample. Transco's designated field geologist or geotechnical engineer will make a field determination as to when it is appropriate to attempt a Shelby tube sample. The associated procedures generally follow ASTM D1587.

Core boring through all other substrate, which would require the use of diamond or tungsten bits, will be carried out using a split-inner, double-tube, swivel type core barrel (such as NWD4) to allow full core recovery. A conventional double-tube, swivel type core barrel may be employed. Single tube core barrels will not be used. Continuous core recovery will be obtained unless otherwise specified by the on-site Transco representative. Where a split inner tube is used, rock core will be transferred directly from the split inner tube to the core box (sheathed as required). Additional core boring procedures generally follow ASTM D2113. Each core box will be photographed in the field in accordance with ASTM D5079. In addition, the rock quality designation will be determined in accordance with ASTM D6032.

It is possible that a cone penetrometer test (CPT) will be conducted in addition to or in lieu of core boring at certain deep core sample sites. No sediment sample would be collected during the CPT. Rather, the CPT unit would be lowered to the seafloor and push rods would force the cone assembly through the sediment to the desired depth. Conducting a CPT to a depth of up to 125 feet may take a few hours, depending on the substrate encountered. The CPT procedures generally follow ASTM D5778 and/or ASTM D3441.

When the casing is raised to an elevation of approximately 1 to 3 feet below the mudline (i.e., seafloor), seawater will be pumped in through the bottom of the drill casing until fluid collected on deck runs clear. Approximately one deep core will be collected per day depending on site conditions, including weather and type of substrate encountered.

2.2.2 Vibracoring

Vibracoring will be used to collect shallow cores with a Rossfelder P-5 model vibracorer, or equivalent.[2] All vibracores samples will be taken from a vessel held on position under power; no seabed disturbance will occur at these sites other than taking the samples themselves. At each sample site, vibracore samples that are collected concurrently will be spaced approximately 20 feet from each other to avoid sediment disturbance. A steel core barrel with a 4-inch diameter will be employed. Prior to use, this barrel will be cleaned with a washdown pump or pressure washer using site water. Following the cleaning of the core barrel, a new core liner will be inserted into the core barrel. The liner for samples intended for geotechnical analyses will be a rigid plastic (polycarbonate); a flexible food-grade polyethylene liner will be used for sediment chemistry samples. Alternatively, a new, dedicated (single-use) rigid plastic liner may also be used for collection of sediment chemistry samples if the flexible liner results in poor recovery. A stainless steel core catcher will then be inserted into the end of the barrel and riveted or threaded in place. The vibracorer will then be deployed into the water column. When the core nose reaches the bottom, the vibracoring unit will be turned on. The vibracorer will continue vibrating and coring until reaching the desired depth or a point of refusal (i.e., penetration of less than 6 inches over a one-minute period). The unit will be laid horizontally on the deck, the core catcher will be removed, and the liner will be pulled from the core. The shallow core (vibracore) samples intended for geotechnical analyses will likely be cut into 3-foot sections, then capped and taped following any field tests. The shallow core samples intended for bulk sediment chemistry analyses will be cut into 3-foot (maximum) sections, which will be treated as sub-samples according to the depth intervals identified in Section 2.3. The physical features of the sediment sub-sample, such as color, texture, and odor will be noted in a field logbook. Distinct layers more than 1-foot thick will be separated as unique sub-samples subject to sediment chemistry analyses if they appear contaminated, based on the field geologist's evaluation of appearance and/or odor. The minimum 1-foot thickness will provide sufficient sample volume for all sediment chemistry analyses. Depending on site conditions such as weather and type of substrate encountered, approximately five to ten vibracore samples will be collected per day.

2.2.3 Sediment Grabs

Surface sediment grab samples will be collected separately from the vibracores samples and/or at a sufficient distance from the specific vibracore sample locations to ensure that the

[2] As a contingency if vibracoring activity is problematic at a particular sample site, shallow core samples may be obtained using a gravity-type piston core capable of penetrating the seabed to a minimum depth of 12 feet.

surface samples are undisturbed prior to collection. The grab samples will be used to evaluate the macrobenthic community (i.e., organisms larger than approximately 0.02 inch [500 microns]). Grab samples will be collected using a Day Grab sampler or equivalent. The grab sampler will be positioned above each sampling location, hoisted with a boom or winch, and allowed to freefall through the water column to the bottom. Upon contact with the bottom, the trigger on the spring-loaded jaws will be activated and the sampler will collect the sediment in a 1 square-foot (0.1 square-meter) section of the seabed. The sampler will be slowly hoisted through the water column to the surface and then brought onto the vessel where the contents of the sampler will be emptied into a clean catch basin. The approximate volume will be recorded and the physical features of the sediment sample, such as color, texture, and odor will be noted in a field logbook. The sample will be mixed and, from one grab sample per sample site, a sub-sample (minimum 250 milliliters [mL]) will be removed and stored in a clean container for subsequent grain size and TOC analysis at an onshore laboratory. The remaining catch basin contents will be poured carefully into a bucket sieve consisting of a 1,000-micron sieve, nested over a 500-micron sieve, and the sample will be gently washed through the sieves using seawater from the site. Any material that flows through the 500-micron sieve will be discarded. The material retained on both the 500- and 1,000-micron sieves will be collected and transferred to a laboratory container capable of holding all of the remaining sample volume. The liquid volume of the sample will be reduced to 50% of the container volume. A 20% formalin solution will be added until the container is filled, resulting in a 10% formalin concentration for sample preservation. The sample will then be transferred to a laboratory for analysis, where macrofauna will be identified to the lowest practicable taxa. Depending on site conditions such as weather and type of substrate encountered, approximately 15 to 30 surface sediment grab samples will be collected per day.

2.2.4 Contingency Resampling Procedures

When three contiguous samples or tests are missed during core boring, the sampling contractor will either restart the boring and testing program from the seafloor or obtain approval from Transco for a strategy capable of addressing the missed-sample issue. In the event that unsatisfactory deep-core recovery is persistent, the length of successive drill runs may be reduced until satisfactory core recovery is obtained, or the hole may be re-drilled, at the discretion of the sampling contractor with approval from Transco's on-site representative.

In the event of poor recovery, collapsing of the liner, or buckling of the core barrel during vibracoring, a second (and potentially third) attempt will be made in the same target area unless the presence of a hard layer is expected to be the cause of poor recovery or core barrel buckling.

If poor recovery or buckling of the core barrel is judged to be the result of encountering a hard layer, the Transco representative onboard the vessel will provide instructions on whether a second attempt shall be made at the same location, an offset location, or at all.

In the event that core boring or vibracoring is aborted for whatever reason, and the Transco representative onboard the vessel confirms that an additional attempt will be made at an offset location, the sampling contractor will relocate to not more than 20 feet from the primary location, and actual coordinates will be logged. For core boring, boreholes will be drilled without sampling or testing to one test cycle above the adjacent missed-sample depth, at which point testing and sampling will be resumed.

2.2.5 Equipment Decontamination

Because of the direct contact with sample material, the vibracorer nose catcher assembly will be washed with soap (Alconox or equivalent) and water to remove any debris before collecting each sample. Seawater from the site may be used to rinse the nose catcher assembly, given that it will be submerged in seawater immediately prior to sample collection. Implements used to process samples for sediment chemistry analyses (e.g., spoons, bowls, mixing blades) will be decontaminated by washing with soap (Alconox or equivalent) and thoroughly rinsing with distilled water, if available, or drinking water. A dilute acid rinse may also be used if allowed by the sampling contractor's vessel-safety policies. Contaminated protective clothing (i.e., disposal gloves) will be kept in plastic garbage bags to be isolated for separate disposal.

2.2.6 Investigation-Derived Waste (IDW)

Cuttings, drilling fluids, and hazardous decontamination fluids (e.g., acid rinse) will be disposed by the sampling contractor in compliance with state and federal requirements. The sampling contractor will record the quantity of cuttings retained at each core boring location.

2.3 Sample Handling and Custody

This section describes the general procedures that will be used from the time samples are initially logged onboard the sampling vessel to the time they are transferred to the onshore laboratories.

2.3.1 Sample Containers, Preservation, and Holding Times

Deep core (boring) samples extruded for geotechnical analyses will be wrapped or bagged in plastic and/or aluminum foil. Deep-core samples collected in Shelby tubes will be capped and retained in their tubes. Shallow-core (vibracoring) samples intended for geotechnical analyses will be retained in capped sections of the tube liners in which they are collected. All untested

samples will be retained by the sampling contractor (or designated laboratory) up to two years from the date of acquisition.

The volume of material, containers, and holding times required for each shallow core sub-sample intended for sediment chemistry analyses are indicated in Table 2.3-1. Table 2.3-2 presents the corresponding volume of water, containers, and holding times for equipment blanks. The laboratory will provide the necessary containers, labels, and chain-of-custody forms sufficient to accommodate any quality control replicates and an additional 10% contingency to cover breakage, etc. The laboratory will maintain a record of the sample bottle-lot numbers that were shipped in the event of a contamination problem.

Before processing, material from surface sediment grab samples will be collected in two 4-ounce glass jars with a Teflon-lined cap and stored at 4 degrees Celsius (°C) +/- 2°C. The material intended for TOC analysis will be tested within 28 days. Following bucket sieving, sediment grab samples will be transferred to plastic containers at least 2 liters in size and preserved with a 20% formalin solution (with a resulting liquid formalin concentration of 10%), then stored between 2°C and 26°C.

Table 2.3-1
Sediment Container and Holding Times for Chemical Analyses

Test Description	Type and Size of Container for each Group of Analytes	Number of Containers and Volume (per sample)	Preservative	Holding Time
Inorganics/Metals	16-oz glass jar with Teflon-lined cap	1 - fill completely	Cool to 4±2°C	6 months
Mercury				28 days
Cyanide				14 days
Semi-volatiles (including PAHs)				Extract within 14 days, analyze within 40 days
Pesticides				Extract within 14 days, analyze within 40 days
PCB Aroclors				Extract within 14 days, analyze within 40 days
Total Organic Carbon				28 days
Total Solids				7 days

Table 2.3-1
Sediment Container and Holding Times for Chemical Analyses

Test Description	Type and Size of Container for each Group of Analytes	Number of Containers and Volume (per sample)	Preservative	Holding Time
Dioxin/Furans	8-oz glass jar with Teflon-lined cap	1 - fill completely	Cool to 4±2°C	Extract within 365 days, analyze within 365 days.
PCB Congeners				
Volatiles	Terra Core Kit or equivalent (Method 5035)	One 40-mL vial with 15mL Methanol; fill with 15g sediment. Two 40-mL vials with 5mL Water; fill with 5g sediment.	Cool to 4±2°C (Freezing optional)	48 hours (up to 14 days if frozen)

Key:
mL = Milliliters
oz = Ounces
PAH = Polycyclic aromatic hydrocarbon
PCB = Polychlorinated biphenyl
TOC = Total organic carbon

Table 2.3-2
Equipment Blank Container and Holding Times for Chemical Analyses

Test Description	Type and Size of Container for each Group of Analytes	Number of Containers and Volume (per sample)	Preservative	Holding Time
Inorganics/Metals	250-mL HDPE preserved to pH ≤2 with HNO_3	1	Cool to 4±2°C	6 months
Mercury				28 days
Cyanide	250-mL HDPE preserved to pH ≥10 with NaOH	1	Cool to 4±2°C	14 days
Semi-volatiles (including PAHs)	1-L Amber glass bottle with Teflon-lined cap	6 (3 minimum, plus 3 contingency)	Cool to 4±2°C	Extract within 7 days, analyze within 40 days
Pesticides				Extract within 7 days, analyze within 40 days
PCB Aroclors				Extract within 7 days, analyze within 40 days

Table 2.3-2
Equipment Blank Container and Holding Times for Chemical Analyses

Test Description	Type and Size of Container for each Group of Analytes	Number of Containers and Volume (per sample)	Preservative	Holding Time
Dioxin/Furans	1-L Amber glass bottle with Teflon-lined cap	4 (2 minimum, plus 2 contingency)	Cool to 4±2°C	Extract within 365 days, analyze within 365 days.
PCB Congeners				
Volatiles	40-mL VOA vial preserved to pH ≤2 with HCl	3	Cool to 4±2°C	14 days

Key:
HCl = Hydrochloric Acid
HDPE = High Density Polyethylene
HNO_3 = Nitric Acid
L = Liter
mL = Milliliters
NaOH = Sodium Hydroxide
oz = Ounces
PAH = Polycyclic aromatic hydrocarbon
PCB = Polychlorinated biphenyl

2.3.2 Sample Handling

All sediment samples and core sections will be handled in a manner that minimizes disturbance, maintains integrity of the samples, and prevents detrimental effects due to the nature of the samples (e.g., contaminants). Any material potentially considered hazardous will be packaged, marked, labeled, and shipped according to the U.S. Department of Transportation regulations in 49 Code of Federal Regulations (CFR) 171 through 177.

Core samples extruded for geotechnical analyses will be sealed in bags (if disturbed) or wrapped (if intact) and placed in protective containers. Following sample preparation, all extruded sections will be photographed with a length scale and the core's identifying numbers or letters. Unextruded samples will be sealed and capped in a manner that preserves moisture content, with a clear indication of top and bottom. Both extruded and unextruded deep core samples will be labeled with company/project name, date, boring (location reference) number, sample number, sub-sample designation, and/or sample depth interval. Unextruded cores will be stored vertically according to their original orientation in the seabed. Rock core samples will be handled in general accordance with ASTM D5079, Standard Practices for Preserving and Transporting Rock Core Samples. The sampling contractor will transport deep-core samples to shore every 24 to 48

hours, weather permitting. The sampling contractor will transport shallow- core samples intended for geotechnical analyses to the shore approximately every 7 to 10 days.

Following inspection and logging of the shallow cores intended for sediment chemistry analyses, sediment sub-samples will be prepared for storage and transportation to the onshore laboratory. If these samples cannot be processed immediately, they will be stored in capped/sealed liners at 4°C ± 2°C until processing. Samples for volatiles analysis will be collected first from each segment upon opening the liner in order to prevent loss of the analytes. After the samples for volatiles analysis have been collected, the core will be sampled for dioxin analysis and, in New Jersey and clamshell dredge sample sites only, PCB congener analysis. Samples for dioxin and/or PCB congener analyses will be collected from the area of the core sub-sample that most likely contain dioxins (e.g., high clay/silt content), as estimated by Transco's designated field geologist or geotechnical engineer. Each core sub-sample will then be composited. All sediment intended for chemical analyses (other than dioxin, PCB congeners, and volatiles) will be collected from this composite mixture and stored in suitable containers as noted in Table 2.3-1. Clean, dedicated implements (e.g., bowls, spoons, mixing blades) will be used to process each sub-sample for sediment chemistry analyses; stainless steel may be re-used following decontamination (see Section 2.2.6) while other materials will be considered disposable. The sediment chemistry sub-samples will be properly labelled (including sample name, date and time of collection, project name, and preservation), recorded on the chain-of-custody, and placed in a dedicated storage area immediately following processing and maintained at a temperature of 4°C ± 2°C. Samples intended for volatile analyses may be frozen if they cannot be analyzed within 48 hours. The sampling contractor will transport sediment chemistry samples to the shore every 24 to 48 hours, weather permitting.

At each benthic sample site, two 4-ounce jars (or one 8-ounce jar) of representative sediment will be collected from one of the three surface sediment grab samples and analyzed for grain size and TOC (at an onshore laboratory). The contents of each grab sample will then be carefully poured into a bucket sieve that consists of a 1,000-micron sieve nested over a 500-micron sieve. The sample will be gently washed through the sieves with site water. All material that flows through the 500-micron sieve will be discarded. The material retained on both the 500 and 1,000 micron sieves will be collected and transferred to a 2-liter (or larger) plastic container and the liquid volume will be reduced to 50% of the container volume. A 20% formalin solution will then be added until the container is filled, resulting in a 10% formalin concentration. The benthic sample container will be labeled and stored between 2°C and 26°C until delivered to the

laboratory. The sampling contractor will transport benthic samples to the shore approximately every 7 to 10 days.

2.3.3 Sample Identification

Samples (or sub-samples) will be assigned a unique, alphanumeric sample identification number by the sampling contractor. All pertinent information associated with this sub-sample will be entered into the sample log system, e.g., client name (Transco), date/time, sample description, and initials of the person entering the data. This number will be recorded on the core tube or sample container using a permanent (indelible) marker.

The sample identification format for samples that will be analyzed for geotechnical properties will include the sampling site number and the corresponding depth interval, e.g., “BHA-5, 20'-22'”” for the interval of the deep core (boring) between 20 feet and 22 feet below the seafloor at sample site BHA5, and “VC1-ALT, 0 '-3’” for the interval of a shallow core (vibracore) between 0 feet and 3 feet below the seafloor.

For sediment chemistry analyses, the sample numbering system will consist of the site number followed by a dash (-) and the subsequent depth (D) intervals, in feet. It is anticipated that the samples will be sub-sampled in the following intervals (in feet): 0-3, 3-6, 6-9, 9-12, and deeper intervals as applicable. The sub-sample intervals will not exceed 3 feet. Transco will notify and consult with NYSDEC and the NJDEP regarding any changes to the sample intervals. The letter “E” (for environmental) will follow this number. Table 2.3-3 contains the proposed list of sample identifications for the environmental cores. The final identification numbers will reflect the actual depths represented in the sub-sample, e.g., if a sub-sample is taken at a depth of 9 and 11 feet at location VC1-ALT, the final sample identification will be VC1-ALT-D9-11E. Sub-samples that are field-duplicated for quality control purposes will be identified in a manner that allows for “blind” submittal to the laboratory.

Table 2.3-3
Examples of Identification Numbers for Sediment Chemistry Sub-Samples

Location	Sample No. 0- to 3-Foot	Sample No. 3- to 6-Foot	Sample No. 6- to 9-Foot	Sample No. 9- to 12-Foot
VC1-ALT	VC1-ALT-D0-3E	VC1-ALT-D3-6E	VC1-ALT-D6-9E	VC1-ALT-D9-12E
VC2	VC2-D0-3E	VC2-D3-6E	VC2-D6-9E	VC2-D9-12E
VC3	VC3-D0-3E	VC3-D3-6E	VC3-D6-9E	VC3-D9-12E
Etc.	Etc.	Etc.	Etc.	Etc.

For benthic analyses, the sample numbering system will consist of the letter "B" (for benthic), followed by the site number, then the letter "C" (center line), the letter "N" (north of the centerline), or the letter "S" (south of the centerline). The sample identification number will end with the year of acquisition (i.e., 2016). Table 2.3-4 contains the proposed list of sample identifications for the benthic samples.

Table 2.3-4
Examples of Identification Numbers for Sediment Chemistry Sub-Samples

Location	Sample No. at Centerline	Sample No. North of Centerline	Sample No. South of Centerline
VC1	B1-C-2016	B1-N-2016	B1-S-2016
VC2	B2-C-2016	B2-N-2016	B2-S-2016
VC3	B3-C-2016	B3-N-2016	B3-S-2016
Etc.	Etc.	Etc.	Etc.

2.3.4 Sample Custody

A chain-of-custody (COC) will be prepared for all samples. The COC will contain the following information: sample identification number or borehole designation, date/time of sample collection, type of sample, sample matrix, preservative (if applicable), sample collection location (state), sample volume or core diameter and length, the analyses requested, and the number and type of sample containers.

The primary objective of COC procedures is to provide an accurate written or computerized record that can be used to trace the possession and handling of a sample from time of sampling through completion of all required analyses. A sample is in custody if it is:

- In the physical possession or view of an individual signing the COC (or representative designated by such an individual);
- Kept in a limited-access storage area on the sampling vessel that is reserved for samples only, in a container that is locked or taped shut with a custody seal at the end of each work day;
- Placed in a locked room or taped carrying container with a custody seal; or
- Stored at the laboratory in a manner consistent with the laboratory's standard operating procedure (SOP).

The COC will initially be signed by an individual who assisted in the collection of the samples. A copy will be retained by this person while the remaining carbon copies of the form accompany the samples to the onshore laboratory.

2.4 Analytical Method Requirements

A standard testing program for deep core samples (i.e., core boring samples) will be used to identify some or all of the geotechnical properties listed in Table 2.4-1. The assigned geotechnical analyses will be determined in the field by Transco's designated geotechnical engineer based on the field characterization of the soil.

Table 2.4-1
Potential Standard Geotechnical Analyses for Deep Core Sediment Samples

Test Property	ASTM Standard
Soil Classification	ASTM D2487
Description and Identification of Soils	ASTM D2488
Moisture Content	ASTM D2216
Density (unit weight)	ASTM D7263
Relative density	ASTM 4254
Atterberg Limits[1]	ASTM D4318
Particle Size Distribution - sieve	ASTM D6913
Particle Size Distribution - hydrometer	ASTM D7928
Hand Vane (Torvane and/or torque watch) and/or Pocket Penetrometer Tests	ASTM WK27337 for pocket penetrometer
Miniature Vane Test	ASTM D4648
Remolded Miniature Vane Test	ASTM D4648
Unconsolidated Undrained Triaxial (UU)	ASTM D2850
Specific Gravity	ASTM D854
Carbonate Content (total and inorganic)	ASTM D4373
Soil pH	ASTM G51
Electrical Resistivity	ASTM G187
Sulfate Content	ASTM C1580
Rock Only	
Point Load Test	ASTM D5731
Compressive strength	ASTM D7012
Mohs Hardness	ASTM MNL46
Cerchar Abrasivity	ASTM D7625
Splitting Tensile Strength (Brazilian tensile strength)	ASTM D3967

Notes:
(1) Test for Atterberg limits to be conducted at same elevation level as moisture contents.
(2) The ASTM standard for pocket penetrometer is under development. There is no ASTM standard for Torvane or pocket vane. However, processes and procedures to use these tools exist.

An advanced testing program may be conducted on selected deep-core sub-samples to provide additional geotechnical data in support of the engineering design. The advanced geotechnical program may include one or more tests for the properties listed in Table 2.4-2.

Table 2.4-2
Potential Advanced Geotechnical Analyses for Deep-Core Sediment Samples

Test Property	ASTM Standard
Consolidation (one-dimensional using incremental loading)	ASTM D2435
Consolidation (one-dimensional using controlled strain loading)	ASTM D4186
Constant Head Permeability (for granular soils)	ASTM D2434[1]
Falling Head Permeability (for fine-grained soils)	ASTM D5856
Static Shear Tests (Consolidated-drained)	ASTM D3080
Static Shear Tests (Consolidated-undrained) (for cohesive soils)	ASTM D6528[2]
Static Triaxial Compression Tests (Ko- and anisotropically consolidated-undrained) with pore measurements	ASTM D4767
Thermal Conductivity	ASTM D5334

Notes:
(1) This still exists but was withdrawn by ASTM in 2015 as too much time had elapsed since last update.
(2) This still exists but was withdrawn by ASTM in 2016, as too much time had elapsed since last update.

Geotechnical analyses that may be performed on shallow core (i.e., vibracore) samples are identified in Table 2.4-3.

Table 2.4-3
Potential Standard Geotechnical Analyses for Shallow-Core Sediment Samples

Test Property	Field (F) or Laboratory (L) Analysis	ASTM Standard	Frequency
Soil Classification	F, L	ASTM D2487	As required
Description and Identification of Soils	F, L	ASTM D2488	As required
Moisture Content	F, L	ASTM D2216	In field, every sample interval, as appropriate. In lab, once every 3 feet
Density (Unit Weight)	L	ASTM D7263	Once every 3 feet
Soil pH	L	ASTM G51	Once every 3 feet
Electrical Resistivity	L	ASTM G187	Once every 3 feet
Sulfate Content	L	ASTM C1580	Once every 3 feet
Cohesive Soils Only			
Torvane or Pocket Penetrometer	F	ASTM WK27337 for Pocket Penetrometer[1]	As appropriate every sample interval
Atterberg Limits	L	ASTM D4318	Once every 3 feet
Miniature Vane Test	L	ASTM D4648	Once every 3 feet
Remolded Miniature Vane Test	L	ASTM D4648	Once every 6 feet
Particle Size - Hydrometer	L	ASTM D7928	Once every 6 feet
Specific Gravity	L	ASTM D854	One per core
Non-cohesive Soils Only			
Particle Size - Sieve	L	ASTM D6913	Once every 3 feet
Particle Size - Hydrometer	L	ASTM D7928	Once every 6 feet
Specific Gravity	L	ASTM D854	Two per core
Relative Density	L	ASTM D4254	One every 3 feet
Carbonate Content (Total and Inorganic)	L	ASTM D4373	Where applicable

Notes:
(1) The ASTM standard for pocket penetrometer is under development. There is no ASTM standard for Torvane or pocket vane. However, processes and procedures to use these tools do exist.

Chemical analyses to be conducted on shallow core samples are summarized in Tables A and B in Appendix C (assuming sufficient sub-sample volume is obtained) and may include metals, cyanide, volatiles, semi-volatiles (including PAHs), pesticides, PCBs, and dioxins. The samples will be analyzed in accordance with the laboratory's ELAP or NELAC certification and as outlined in the laboratory's SOPs included in Appendix D. Table A in Appendix C identifies the chemical analyses that will be performed for sample sites in New York waters where clamshell dredging would not occur during the Project. Table B in Appendix C identifies a more extensive list of chemical analyses that will be performed for sample sites in all New Jersey waters and in New York waters where clamshell dredging may occur during the Project. Tables 2.4-4 and 2.4-5 specify which sample sites are associated with Tables A and B in Appendix C. All analyses in Appendix C will be conducted on all sediment sub-samples, with the exception of dioxin analysis.

For each shallow core sample site in New York waters where clamshell dredging would not occur during the Project (Table 2.4-4), dioxin analysis will only be immediately performed on the sediment sub-sample most likely to contain dioxin (e.g., a high clay or silt content), as determined in the field by the field geologist or geotechnical engineer. Samples selected for immediate dioxin analysis will be indicated as such on the label and COC. Material from the other shallow core sub-samples will be archived for future dioxin analysis. The analytes noted in Appendix C were selected based on chemicals of concern listed in the NYSDEC TOGS 5.1.9 document, the NJDEP 1997 document titled *The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters*, the USACE/EPA 2016 document titled *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal*, as well as feedback from NYSDEC and NJDEP agency staff. Laboratory analyses are not proposed for water samples at the pre-construction stage since it is generally not worthwhile to characterize the chemical components in an open water environment that is constantly changing when such results would only indicate a snapshot of water quality during the limited sampling period.

Table 2.4-4
Sample Sites in New York where Clamshell Dredging Will Not Occur

VC11	VC42	VC65	For sediment chemistry analyses to be performed at these sites, see Table A in Appendix C. New York Waters (no clamshell dredging)
VC12	VC43	VC66	
VC13	VC44	VC67	
VC14	VC45-ALT	VC68	
VC19	VC46	VC69-ALT	
VC20	VC47	VC70	
VC21	VC48-ALT	VC71	
VC22	VC49	VC72	
VC23	VC50	VC73-ALT	
VC24	VC51	VC74	
VC25	VC52	VC75	
VC26	VC56	VC76	
VC27	VC57	VC82	
VC28	VC58	VC83-ALT	
VC29	VC59	VC84-ALT	
VC30	VC60	VC85	
VC31-ALT	VC61-ALT	VC86-ALT	
VC32	VC62	VC87	
VC33-ALT	VC63		
VC34	VC64		

Table 2.4-5
Sample Sites in New Jersey and in New York where Clamshell Dredging May Occur

VC1 ALT	VC15	VC41-ALT	For sediment chemistry analyses to be performed at these sites, see Table B in Appendix C. New Jersey Waters or New York Waters (clamshell dredging)
VC2	VC16	VC53	
VC3-ALT	VC17	VC54	
VC4	VC18	VC55	
VC5	VC35	VC77	
VC6	VC36	VC78	
VC7	VC37	VC79-ALT	
VC8	VC38	VC80	
VC9	VC39	VC81	
VC10	VC40		

Benthic taxonomic identification will be performed on organisms isolated in each surface sediment grab sample. Organisms will be keyed to the lowest practical taxa. If more than 100 organisms are found in a particular sample, the sample will be sub-sampled and all of the organisms in the sub-sample keyed. Data from the triplicate samples will enable statistical analyses to be performed. The following community metrics will be calculated: abundance, richness, Shannon-Weiner diversity index, and evenness. In addition to the taxonomy work, grain size and TOC analyses will be performed on each sample to aid in interpretation of the data collected. The TOC analysis for surface sediment grab samples will be conducted by the sediment chemistry laboratory using the TOC analysis methodology that will be applied for the shallow core samples. The grain size analysis for sediment grab samples will be conducted by the geotechnical laboratory using the sieve-based particle size analysis methodology for unconsolidated material in the shallow core samples.

2.5 Quality Control

QC data are necessary to determine precision and accuracy and to demonstrate the absence of interferences such as contamination of sample containers/glassware and reagents.

Field and laboratory QC sample frequencies for chemical analyses are summarized on Tables 2.5-1 and 2.5-2, respectively. For geotechnical analyses, QC limits are described in the appropriate ASTM testing methods.

Table 2.5-1
Field Quality Control Samples for Chemical Analyses

QC Sample	Description	Acceptance Criteria	Corrective Action
Field Duplicate	One per matrix per 20 samples for each analysis.[1]	RPDs of 70% for sediments	Qualify related samples and assess data usability.
Field Equipment Blank	One per equipment set per day. Only equipment sets that are subject to decontamination require equipment blanks. Dedicated or disposable equipment do not require equipment blanks.	No positive detections >one-half the reporting limit in the equipment blank samples. Equipment blanks are not required for single-use (disposal) sample processing implements (e.g., spoons and bowls).	Qualify related samples and assess data usability. If time permits, modify equipment decontamination procedures.
Trip Blank	One per cooler containing samples for volatiles analysis.	No positive detections in the trip blank.	Qualify related samples assess data usability.

Note:
(1) Field duplicates are not required for sediment archived for dioxin analysis.
Key:
RPD = Relative percent difference

Table 2.5-2
Laboratory Quality Control Checks for Chemical Analyses

Methods	Quality Check	Frequency	Acceptance Criteria	Corrective Action
All methods	MB	One per matrix per preparation batch.	The goal is for method blanks to be free of contamination. Low-level contamination may be present but must be less than the reporting limit.	If contamination is more than the reporting limit, samples will be reanalyzed. If contaminants are present in the method blank but not in Project samples, no further action is required.
All methods	LCS	One per matrix per preparation batch for each analysis. The LCS must contain all target analytes of concern at the site.	The LCS recovery must be within method control limits to demonstrate acceptable method performance. Sporadic marginal failures of a few target analytes are allowed when more than five target analytes are reported.	If LCS recoveries are outside QC criteria for more than a few target analytes, recoveries are significantly low, or the compounds were detected in the samples, then corrective action should be taken according to the laboratory's standard operating procedures. Corrective action is normally to extract and reanalyze the batch if within holding times.
Organic Methods	Surrogate Spikes	All samples and QC checks.	Surrogate recoveries must be within QC criteria for method blanks and LCSs to demonstrate acceptable method performance. Surrogate recoveries that are outside QC criteria for a sample indicate a potential matrix effect.	If sample surrogates recoveries are outside of QC criteria, the laboratory will re-analyze the sample to confirm matrix effects. If confirmed, the results will reviewed along with corresponding laboratory QC results.

Table 2.5-2
Laboratory Quality Control Checks for Chemical Analyses

Methods	Quality Check	Frequency	Acceptance Criteria	Corrective Action
All methods	MS/MSD	One per matrix for each analysis batch. The spike solution must contain a broad range of the analytes of concern at the site. The overall frequency of MS/MSD on project samples must be at least one set per 20 samples. [1]	Method QC criteria or LCS criteria if not specified. MS recoveries outside the control limits applied to the LCS indicate matrix effects. QC criteria for MSD RPDs are noted for each method in Table 1.4-2.	Sample clean-up procedures may be warranted for samples with severe matrix effects.

Note:
(1) MS/MSD not required for sediment archived for dioxin analysis.

Key:
LCS = Laboratory control sample
MB = Method blank
MS/MSD = Matrix spike/matrix spike duplicate
SDG = Sample delivery group

2.6 Instrument/Equipment Testing, Inspection, and Maintenance

Offshore sampling activities will not be authorized until Transco has accepted the sampling vessel(s) as operational and ready for work.

2.6.1 Field Equipment Testing and Maintenance

All of the onboard equipment will be supplied with adequate spare parts and back-up units for ordinary maintenance and repair at sea. The sampling contractor will provide Transco with a complete inventory of equipment, back-up systems, and spare parts available on the vessel(s). The sampling contractor will identify all equipment that is critical to maintaining full sampling vessel operability and define the contingency action in event of failure.

Deck trials will be conducted at time of vessel mobilization to demonstrate that all sampling and processing systems are operational. The trials will include testing all core boring, vibracoring, and grab sampling equipment and validating vessel positioning systems unless the sampling contractor can suitably document functionality or equipment redundancy to Transco through other means.

During offshore activities, all lifting equipment such as cranes, booms, hoists, spreader bars, slings, etc. will be inspected at least once per shift during the sampling activities.

All above activities will be documented as appropriate and reported to Transco.

2.6.2 Analysis Instrument Maintenance

Laboratory and field instruments and equipment used for sample analyses will be serviced and maintained only by qualified personnel. Laboratory instrument maintenance procedures will be evaluated according to the laboratory's SOP to verify that there will be no impacts on analysis of Project samples due to instrument malfunction. For example, the laboratory must have duplicate instrumentation and/or maintained major equipment under service agreements with the manufacturer that require rapid response by manufacturer-approved service agents.

Field instruments owned by the sampling contractor or rented through approved suppliers will have manufacturer-approved maintenance programs. Field equipment will be checked upon receipt to verify that instruments are in good working condition and are accompanied by appropriate calibration records or certifications. On-site operation will be performed in accordance with manufacturer manuals. If any problems occur, the instrument will be recalibrated and verified. If calibration cannot be verified, the instrument will be replaced.

2.7 Instrument/Equipment Calibration and Frequency

Before initiating sampling, the sampling contractor will calibrate any vessel instrumentation according to manufacturer's instructions and record the findings in the field logbook or records. The sampling contractor will provide the following information for Transco's review and approval:

- Evidence of recent (validated) calibration of field sampling equipment and onboard testing instruments;
- Calibration methods for vessel navigation and positioning systems;
- Detailed field calibration and operating procedures for sampling equipment and onboard testing instruments (including probes); and
- Calibration verification procedures for vessel positioning systems, field sampling equipment, and onboard testing instruments.

2.8 Inspection/Acceptance of Supplies and Consumables

The sampling contractor will be responsible for inventorying and ensuring that adequate supplies are available for the offshore sampling activities before beginning sampling, including spare equipment parts and personal protection equipment. At the time of delivery, the sampling contractor or Transco's designated representative will verify that coolers/crates, sample containers, custody seals, and COCs are in good condition and sufficient in number to maintain

the offshore sampling activities until the scheduled date/time for the next supply pick-up or delivery.

2.9 Non-Direct Measurements

Sampling activities will rely on some data collected by outside sources such as tidal stage and elevation information from NOAA tidal stations. If available, the precision and accuracy of such sources will be identified and reported as appropriate.

2.10 Data Management

The sampling contractor will create electronic logs or spreadsheets for all data collected in the field and transmit that information to Transco and the appropriate consultant(s). The laboratory performing geotechnical analyses will submit the results to the sampling contractor in a format that was determined following selection of the laboratory, and such data would be incorporated into the Geotechnical Site Investigation Report by the sampling contractor or INTECSEA. The laboratory performing sediment chemistry analyses will provide an electronic data deliverable for all Project data to E & E. Electronic spreadsheets will also be submitted to E & E for results of benthic laboratory analyses. Data would be retained on a secure server by the consultant(s) that use this data to produce summary reports for or on behalf of Transco. Electronic data for sediment chemistry analyses will be submitted to regulatory agencies in a format to be determined in consultation with NYSDEC and the NJDEP (e.g., NYSDEC EQuIS™ format and NJDEP Hazsite format).

3 ASSESSMENT AND OVERSIGHT

Individual consultants and contractors will operate under their own internal quality programs that apply to their routine technical oversight programs. Project-specific assessment and oversight procedures are discussed below.

3.1 Assessment and Response Actions

The sampling contractor will provide checklists in the Geotechnical, Chemical, and Benthic Investigation Procedures for the offshore trials and calibrations. During the offshore sampling, the sampling contractor will complete procedural checklists for sampling activities and equipment calibrations, to be witnessed by a Transco representative.

3.1.1 Peer Review

All contractors and consultants are required to perform an independent technical review on the sampling and analysis reports before submitting the reports to Transco and regulatory agencies. The sample contractor's lead field personnel will review summary geotechnical and/or environmental reports to ensure accurate descriptions of field sampling activities, as warranted (e.g., statements not taken directly from the sampling contractor's field report).

3.1.2 Technical Systems Assessments

The entire Project offshore sampling and analysis team is responsible for ongoing assessment of the technical work performed by the team, identification of nonconformance with the Project objectives, and initiation, implementation, and documentation of corrective action.

During offshore activities, a Transco representative will be in the field observing sampling activities to help ensure compliance with the technical specifications of the SAP/QAPP and associated sampling and field testing methods. The Transco representative will have authority to stop work if the field operations do not comply with the specifications. The sampling vessel(s) will be subject to ongoing audits of safety and management systems by Transco. The sampling contractor will provide Transco with reasonable access to any part of the vessel, operational, or maintenance records as appropriate for the audit. Field audits will be coordinated through Transco.

The laboratories will implement a comprehensive program of internal audits to verify compliance of their systems with applicable laboratory-specific standard operating procedures and QA/QC manuals, consistent with their respective certifications (e.g., ASTM, ELAP or NELAP). External laboratory audits will be performed by the appropriate state agency in accordance with relevant certification programs.

For benthic population analysis, a second experienced, independent (third-party) onshore laboratory will perform QA/QC analysis of benthic sediment samples. A total of 10% of all samples will be randomly selected in the field by the sampling contractor or Transco's designated representative and, after analysis at the primary benthic laboratory, sent to the third-party laboratory for independent taxonomic verification.

3.1.3 Corrective Action

If field or laboratory QA/QC audits or checks identify any deficiencies, deviations, or nonconformance events, action will be taken to correct any deficiency and minimize the possibility

of recurrence. The sampling contractor or laboratory will notify Transco immediately of any potential impacts on the results for the Project.

If there is more than a 10% difference in individual sample results of benthic population analyses received from the third-party laboratory taxonomist, the company QA officer will arrange for a review of the identification procedures and achieve a consensus between the two taxonomists. This may result in the review of certain organisms in all of the samples.

3.2 Reports to Management

There will be regularly scheduled progress meetings between the Sampling Contractor, consultants and Transco on sampling and testing progress, results, and schedule, as appropriate. Other reports will be provided as detailed in Section 1.6.

4 DATA VALIDATION AND USABILITY

4.1 Data Review, Validation, and Verification Requirements

For sediment chemistry analyses, analytical reporting limits and target compounds and QC summary data for surrogates, method blanks, laboratory control samples (LCS), and matrix spike/ matrix spike duplicates (MS/MSD) samples will be compared with the limits listed in Table 1.4-2.

Data review and validation requirements for geotechnical analyses are identified in the respective ASTM testing methods.

4.2 Validation and Verification Methods

The analytical laboratories are responsible for internal data review. The QA officer and/or manager for each laboratory is expected to review at least 10% of the data packages. (Note: this percentage was verified following selection of the laboratories.) All levels of laboratory review will be fully documented and available for review by Transco if requested or if a laboratory is audited.

The results of the geotechnical field and laboratory analyses will be reviewed for quality by INTECSEA's geotechnical engineers.

For sediment chemistry analyses, calibration summary data will be checked by the laboratory to verify that all positive results for target compounds were generated under an acceptable calibration as defined by the analytical method. All deviations will be noted in the laboratory's case narrative.

After receipt of sediment chemistry results from the laboratory, E & E will evaluate the data for completeness by verifying that the laboratory information matches the field information and that the following items are included in the hard copy data package:

- COC forms and sample summary forms
- Case narrative describing any out-of-control events and summarizing analytical procedures
- Data report forms
- QA/QC summary forms.

Field data such as sample identifications and sample dates will be checked against the laboratory report. Raw data files from the field and laboratory will not be reviewed unless there is a significant problem noted with the summary information. If the data package is incomplete, the laboratory will be required to provide all missing information within one working day.

The complete sediment chemistry data packages will be evaluated for compliance by E & E's lead Project chemist (and/or designee), who will follow EPA Region 2 Data Validation Standard Operating Procedures. Compliance checks will include:

- Review of raw data for any apparent QC anomalies;
- Confirming that all analytical problems and corrections are reported in the case narrative and that appropriate laboratory qualifiers are added;
- If any problems are identified, review of these concerns with the laboratory, obtaining additional information if necessary, and checking all related data to determine the extent of the error; and,
- Review of non-analytical data in field logs to help identify potential causes for discrepancies.

The lead Project chemist will alert the E & E Project manager to any QC problems, obvious anomalous values, or other data discrepancies that may impact data usability. Tables summarizing samples with detectable concentrations of any analytes will be prepared for inclusion with the Offshore Environmental Sampling and Analysis Summary Report, along with a summary of data discrepancies and any associated corrective actions taken in accordance with Sections 3.1 and 4.3.

The results of benthic laboratory analyses will be reviewed by E & E's lead Project biologist (and/or designee), who will verify that laboratory sample information matches field information (e.g., consistent sample identification) and review calculations for accuracy. Results may also be compared with historical benthic data for the Project area, if available.

4.3 Reconciliation with User Requirements

The laboratory QA officer and/or manager, as well as Transco's appropriate consultant (E & E or INTECSEA) will work with Transco and relevant agencies in assessing data quality.

If there are discrepancies in the geotechnical data, the laboratory tests will typically be repeated. If there is inadequate sample to repeat the test, then a determination will be made by an INTECSEA or Transco geotechnical engineer on the validity and use of the original test results, accounting for potential inaccuracies.

The data quality assessment for sediment chemistry analyses may include some or all of the following actions:

- Data that are determined to be incomplete or not usable for the Project will be identified. E & E will discuss resolution of the issue with Transco and agency technical staff and implement appropriate corrective actions (e.g., re-testing if samples are within hold times or considering other sample/analyte results as surrogate data).
- Data that are non-detect but have elevated reporting limits due to blank contamination or matrix interference will be compared with screening criteria values (if available). If reporting limits exceed the screening criteria values, then results will be handled as incomplete data as described above.
- Data that are qualified as estimated will be used for all Project decision-making. Data assessors comparing results with screening criteria will have to account for the higher level of uncertainty in their statistical analysis.

If significant disagreements or uncertainties in benthic sample species identifications persist, additional samples may be sent to the second and/or a third independent laboratory for additional verification. However, it is more likely that the uncertain species will simply be classified at a higher taxonomic level that is agreed upon by all laboratories.

If discrepancies are identified in data results that may impact data usability following submittal of summary reports to agencies, Transco will notify such agencies and identify any proposed measures to correct or resolve resulting data gaps.

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Transcontinental Gas Pipe Line Company, LLC

Appendices to Offshore Sampling and Analysis Plan / Quality Assurance Project Plan

Northeast Supply Enhancement Project

July 2016

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Appendix A Sample Site Figures

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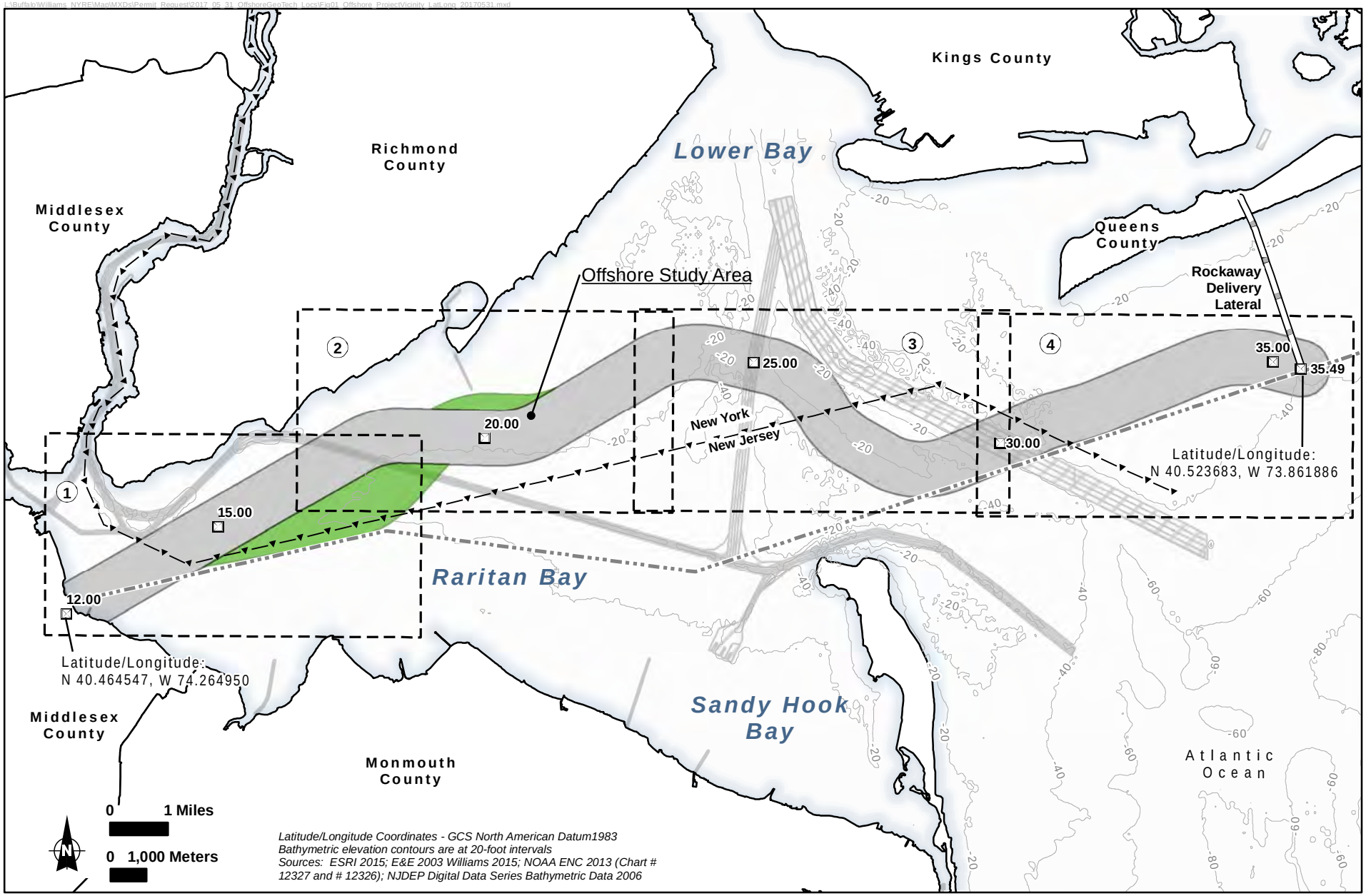
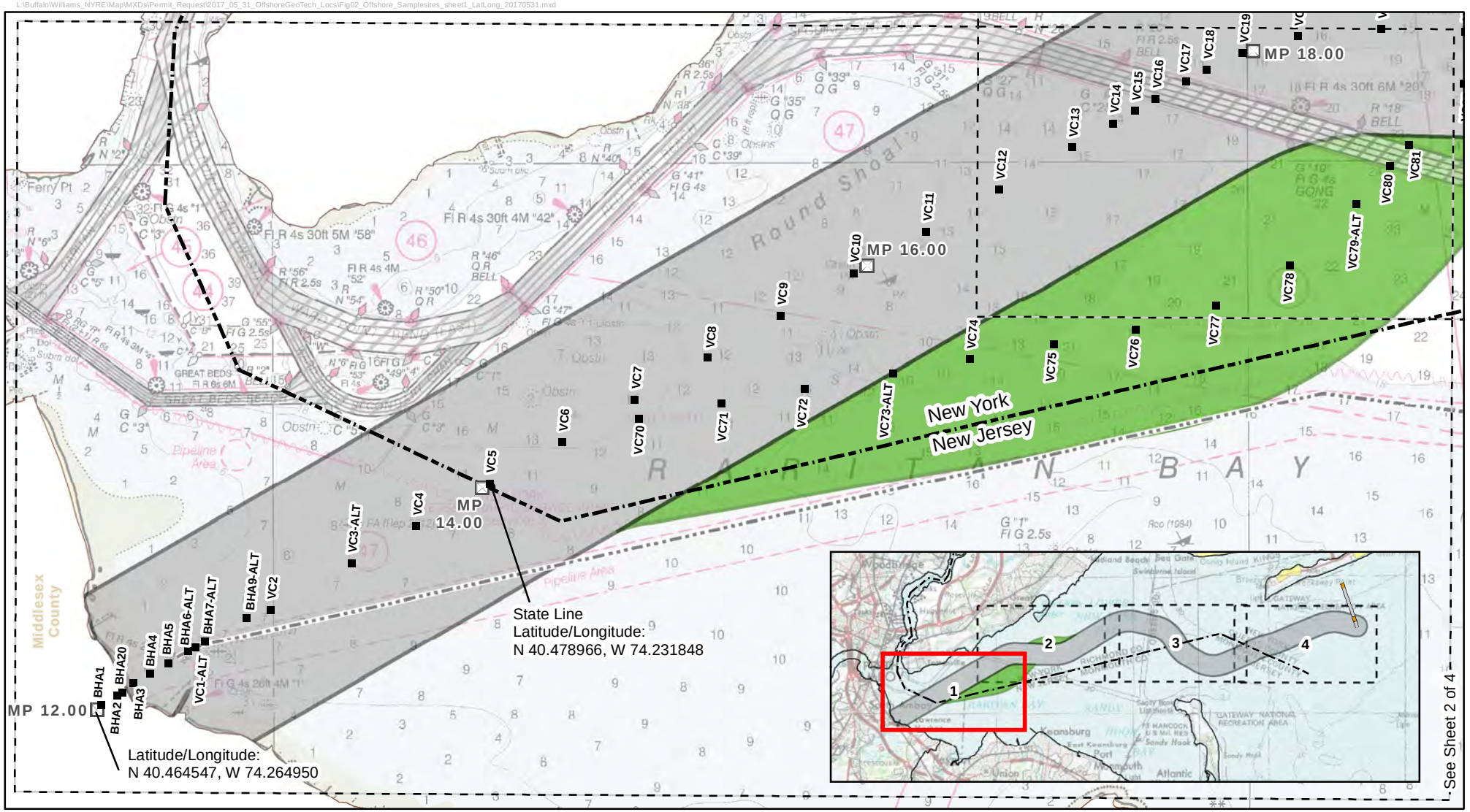


Figure 1: Raritan Bay and Lower New York Bay Vicinity Map
New Jersey, New York

Revision Date: 5/31/2017



0 0.25 Miles

0 500 Meters

- Sample Site
- Milepost for Offshore Study Area
- Offshore Study Area (5,000-ft)
- Study Area Extension
- Maintained Navigation Channel
- Map Sheet Match Line
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral

Latitude/Longitude Coordinates - GCS North American Datum1983

NOTE: Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigational charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

Figure 2: Sample Site Map

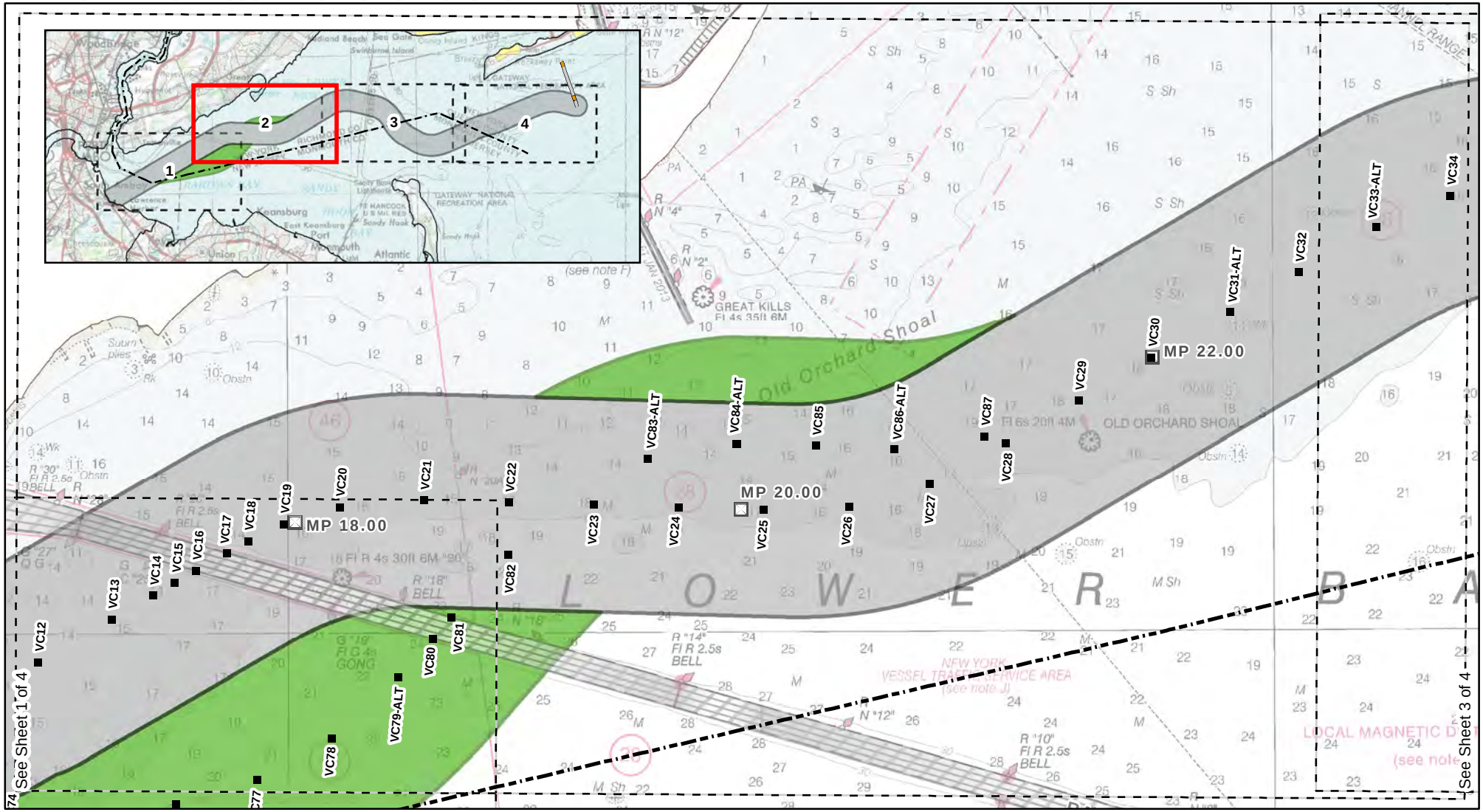
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New Jersey, New York

Service Layer Credits:
NOAA RNC Chart # 12327;
ESRI 2015; E&E 2003
Williams 2015; USFWS 1982-1997; NOAA ENC 2013

Revision Date: 5/31/2017

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0 0.25 Miles

0 500 Meters

- Sample Site
- Milepost for Offshore Study Area
- Offshore Study Area (5,000-ft)
- Study Area Extension
- Maintained Navigation Channel
- Map Sheet Match Line
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral

Latitude/Longitude Coordinates - GCS North American Datum1983

NOTE: Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigational charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

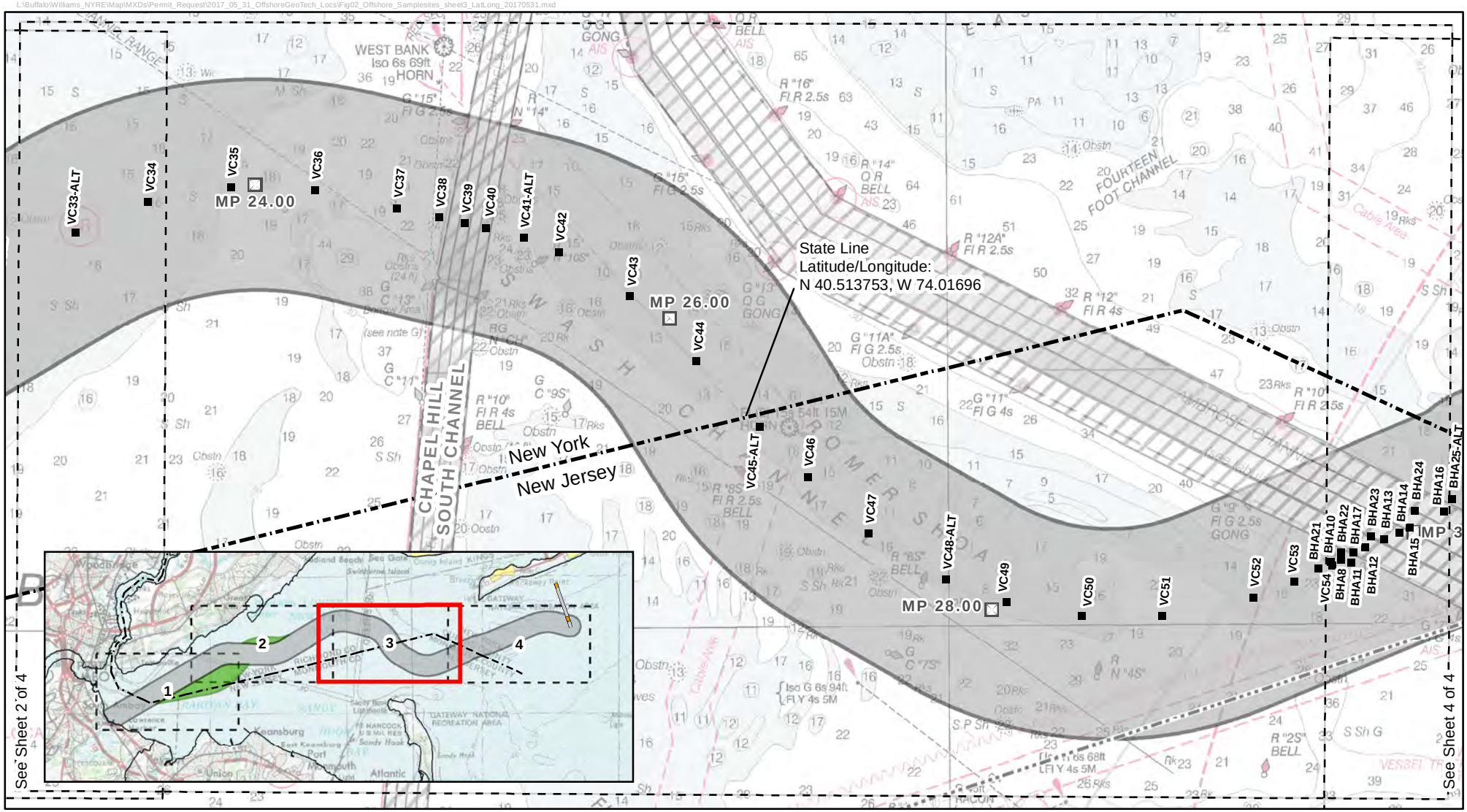
Figure 2: Sample Site Map

(Sheet 2 of 4)

New Jersey, New York

Service Layer Credits:
NOAA RNC Chart # 12327;
ESRI 2015; E&E 2003
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Revision Date: 5/31/2017



0 0.25 Miles

0 500 Meters

- Sample Site
- Milepost for Offshore Study Area
- Offshore Study Area (5,000-ft)
- Study Area Extension
- Maintained Navigation Channel
- Map Sheet Match Line
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral

Latitude/Longitude Coordinates - GCS North American Datum1983

NOTE: Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigational charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

Figure 2: Sample Site Map

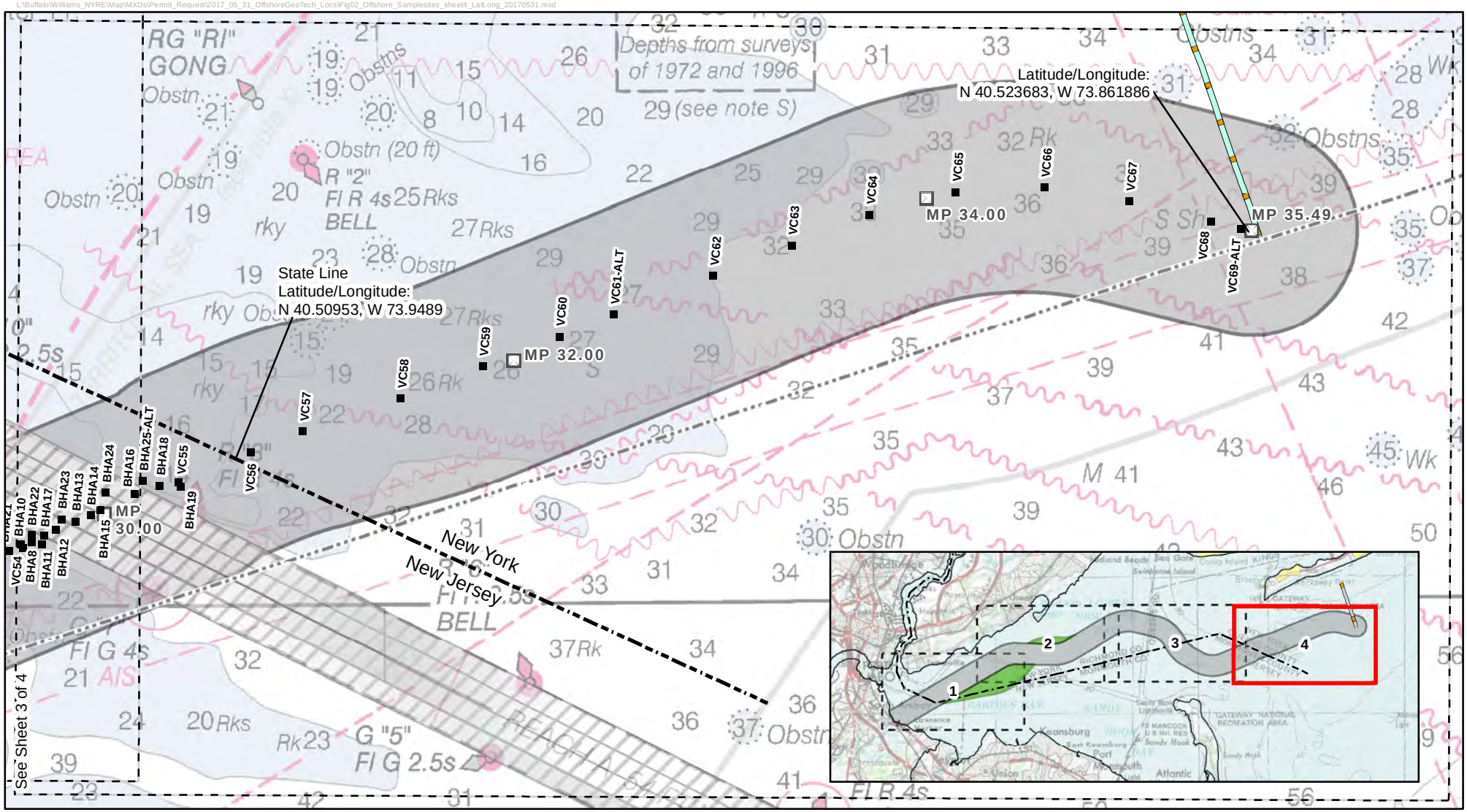
(Sheet 3 of 4)

New Jersey, New York

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Williams 2015; USFWS 1982-1997; NOAA ENC 2013

Revision Date: 5/31/2017

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0 0.25 Miles

0 500 Meters

- Sample Site
- Milepost for Offshore Study Area
- Offshore Study Area (5,000-ft)
- Study Area Extension
- Maintained Navigation Channel
- Map Sheet Match Line
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral

Latitude/Longitude Coordinates - GCS North American Datum1983

NOTE: Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigational charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

Figure 2: Sample Site Map

(Sheet 4 of 4)

New Jersey, New York

Service Layer Credits:
NOAA RNC Chart # 12326;
ESRI 2015; E&E 2003
Williams 2015; USFWS 1982-1997; NOAA ENC 2013

Revision Date: 5/31/2017

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Appendix B Sample Site Coordinate Tables

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Table 1A: Sediment Sample Sites in New York State Waters[a]

Sample Site [b,c,d]	Latitude	Longitude	Coring Depth	Sample Site [b,c,d]	Latitude	Longitude	Coring Depth
VC5	***40.479100***	***-74.231542***	***12***	***VC41-ALT***	***40.525453***	***-74.036149***	***20***
VC6	***40.481830***	***-74.225304***	***12***	VC42	40.524540	-74.033142	12
VC7	***40.484562***	***-74.219061***	***12***	VC43	40.521631	-74.027063	12
VC8	***40.487286***	***-74.212836***	***12***	VC44	40.517348	-74.021425	12
VC9	***40.490016***	***-74.206593***	***12***	VC56	40.509925	-73.947645	12
VC10	***40.492746***	***-74.200352***	***12***	VC57	40.511318	-73.943213	12
VC11	40.495473	-74.194115	12	VC58	40.513384	-73.934771	12
VC12	40.498201	-74.187873	12	VC59	40.515447	-73.927729	12
VC13	40.500929	-74.181632	12	VC60	40.517366	-73.921185	12
VC14	40.502472	-74.178102	12	VC61-ALT	40.518756	-73.916515	12
VC15	***40.503286***	***-74.176237***	***20***	VC62	40.521246	-73.907949	12
VC16	***40.504057***	***-74.174471***	***20***	VC63	40.523207	-73.901256	12
VC17	***40.505196***	***-74.171867***	***20***	VC64	40.525164	-73.894571	12
VC18	***40.505968***	***-74.170079***	***20***	VC65	40.526621	-73.887209	12
VC19	40.507049	-74.167031	12	VC66	40.526863	-73.879610	12
VC20	40.508123	-74.162337	12	VC67	40.525935	-73.872341	12
VC21	40.508567	-74.155187	12	VC68	40.524584	-73.865376	12
VC22	40.508392	-74.147998	12	VC69-ALT	40.524078	-73.862787	12
VC23	40.508217	-74.140810	12	VC70	40.483336	-74.218728	12
VC24	40.508041	-74.133621	12	VC71	40.484286	-74.211647	12
VC25	40.507865	-74.126433	12	VC72	40.485233	-74.204565	12
VC26	40.508033	-74.119256	12	VC73-ALT	40.486194	-74.196990	12
VC27	40.509493	-74.112343	12	VC74	40.487125	-74.190401	12
VC28	40.512078	-74.106004	12	VC75	40.488070	-74.183318	12
VC29	40.514823	-74.099775	12	VC76	40.489015	-74.176235	12
VC30	40.517568	-74.093546	12	***VC77***	***40.490549***	***-74.169347***	***20***
VC31-ALT	40.520506	-74.086879	12	***VC78***	***40.493204***	***-74.163070***	***20***
VC32	40.523057	-74.081086	12	***VC79-ALT***	***40.497147***	***-74.157372***	***20***
VC33-ALT	40.525932	-74.074540	12	***VC80***	***40.499630***	***-74.154438***	***20***
VC34	40.527932	-74.068241	12	***VC81***	***40.500985***	***-74.152842***	***20***
VC35	***40.528898***	***-74.061174***	***12***	VC82	40.505013	-74.148096	12
VC36	***40.528653***	***-74.054002***	***20***	VC83-ALT	40.511150	-74.136218	12
VC37	***40.527473***	***-74.046977***	***20***	VC84-ALT	40.512075	-74.128742	12
VC38	***40.526834***	***-74.043371***	***20***	VC85	40.511970	-74.122006	12
VC39	***40.526449***	***-74.041158***	***20***	VC86-ALT	40.511734	-74.115363	12
VC40	***40.526125***	***-74.039373***	***20***	VC87	40.512506	-74.107716	12

Reference datum: NAD 1983.

Notes:

[a] Sample Sites as of November 11, 2016.

[b] Sample Sites labeled "VC" indicate where vibracore and grab-sampling will occur.

[c] Sample Sites labeled "-ALT" indicate shifts that occurred based on a Phase I archaeological survey.

[d] Sample Sites in ***bold and italicized*** indicate where clamshell dredging may occur.

Table 1B
Sediment Sample Sites in New Jersey State Waters[a]

Sample Site [b,c,d]	Latitude	Longitude	Coring Depth	Sample Site [b,c,d]	Latitude	Longitude	Coring Depth
BHA 1 [e]	40.464648	-74.265026	127	BHA21	40.503601	-73.968359	71
BHA 2 [e]	40.465324	-74.263463	147	BHA22	40.504657	-73.966412	88
BHA 3	40.466122	-74.261990	117	BHA23	40.505645	-73.963866	71
BHA 4	40.466756	-74.260539	117	BHA24	40.507361	-73.960056	65
BHA 5	40.467438	-74.258979	112	BHA25-ALT	40.508106	-73.956877	122
BHA 6-ALT	40.468215	-74.257285	102	***VC1-ALT***	***40.468475***	***-74.256647***	***20***
BHA 7-ALT	40.468837	-74.255837	102	***VC2***	***40.470904***	***-74.250259***	***12***
BHA 8	40.504154	-73.966412	95	***VC3-ALT***	***40.473933***	***-74.243344***	***12***
BHA 9-ALT	40.470373	-74.252342	72	***VC4***	***40.476370***	***-74.237777***	***12***
BHA 10	40.504030	-73.967324	77	VC45-ALT	40.513056	-74.016094	12
BHA 11	40.503991	-73.965562	72	VC46	40.509752	-74.011992	12
BHA 12	40.504967	-73.964348	92	VC47	40.506057	-74.006768	12
BHA 13	40.505491	-73.962680	77	VC48-ALT	40.503008	-74.000169	12
BHA 14	40.505903	-73.961371	92	VC49	40.501529	-73.995029	12
BHA 15	40.506229	-73.960524	72	VC50	40.500576	-73.988667	12
BHA 16	40.507266	-73.957570	92	VC51	40.500540	-73.981704	12
BHA 17	40.504620	-73.965346	79	VC52	40.501745	-73.973943	12
BHA 18	40.507776	-73.955418	127	***VC53***	***40.502756***	***-73.970427***	***12***
BHA 19	40.507737	-73.953656	82	***VC54***	***40.503776***	***-73.967187***	***20***
BHA20 [e]	40.465515	-74.262945	130	***VC55***	***40.507991***	***-73.953793***	***30***

Reference datum: NAD 1983.
Notes:
[(a)] Sample Sites as of November 11, 2016.
[(b)] Sample sites labeled “BHA” indicate where core boring will occur. Sites labeled “VC” indicate where vibracore and grab-sampling will occur.
[(c)] Sample Sites labeled “-ALT” indicate shifts that occurred based on a Phase I archaeological survey.
[(d)] Sample Sites in ***bold and italicized*** indicate where clamshell dredging may occur.
[(e)] Onshore sites.

Appendix C Sediment Chemistry Test Tables

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Table A: Sediment Chemistry Tests for New York Sites with NO Clamshell Dredging

Vibracore Site Numbers [Total of 50]: VC11 through VC14; VC19 through VC34; VC42 through VC 44; VC 56 through VC 69; VC70 through VC76; and VC82 through VC87								
Test Analyte	**EPA Method**	**NYSDEC Required Method Detection Limits (mg/kg, ppm)**	**NJDEP or USACE Required Reporting Limits[1] (mg/kg, ppm)**	**NYSDEC No Appreciable Contamination Threshold Values[2] (mg/kg, ppm)**	**NJDEP Low Effects Range[3] (mg/kg, ppm)**	**Listed in NYSDEC TOGs 5.1.9**	**Listed in USACE Reg2 Guidance**	**Listed in NJDEP 1997 Manual**
METALS								
Aluminum	EPA 6020A	N/A	40	N/A	18000*			X
Antimony	EPA 6020A	N/A	12	N/A	9.3*			X
Arsenic	EPA 6020A	3^	1	<14 (<8.2)	8.2	X	X	X
Barium	EPA 6020A	N/A	40	N/A	48*			X
Beryllium	EPA 6020A	N/A	1	N/A	N/A			X
Cadmium	EPA 6020A	1	1	<1.2 (<1.2)	1.2	X	X	X
Calcium	EPA 6020A	N/A	1,000	N/A	N/A			X
Chromium	EPA 6020A	N/A	1	(<81)	81		X	X
Cobalt	EPA 6020A	N/A	10	N/A	10*			X
Copper	EPA 6020A	5^	1	<33 (<34)	34	X	X	X
Iron	EPA 6020A	N/A	20	N/A	N/A			X
Lead	EPA 6020A	2^	0.6	<33 (<47)	47	X	X	X
Magnesium	EPA 6020A	N/A	1,000	N/A	N/A			X
Manganese	EPA 6020A	N/A	3	N/A	260*			X
Mercury	EPA 7474	0.2^	0.1	<0.17 (<0.15)	0.15	X	X	X
Molybdenum	EPA 6020A	N/A	N/A	N/A	N/A			
Nickel	EPA 6020A	N/A	1	(<21)	21		X	X
Potassium	EPA 6020A	N/A	1,000	N/A	N/A			X
Selenium	EPA 6020A	N/A	1	N/A	1*			X
Silver	EPA 6020A	N/A	1	(<1.0)	1		X	X
Sodium	EPA 6020A	N/A	1,000	N/A	N/A			X
Strontium	EPA 6020A	N/A	N/A	N/A	N/A			
Thallium	EPA 6020A	N/A	2	N/A	N/A			X
Tin	EPA 6020A	N/A	N/A	N/A	N/A			
Titanium	EPA 6020A	N/A	N/A	N/A	N/A			
Vanadium	EPA 6020A	N/A	10	N/A	57*			X
Zinc	EPA 6020A	N/A	4	(<150)	150			X
PCB AROCLORS (Aroclor list from 1997 NJDEP manual applied for NY sum.)								
Aroclor-1016	EPA 8082A	N/A	0.033	N/A	0.007			(X)
Aroclor-1221	EPA 8082A	N/A	0.067	N/A	N/A			(X)
Aroclor-1232	EPA 8082A	N/A	0.033	N/A	N/A			(X)
Aroclor-1242	EPA 8082A	N/A	0.033	N/A	N/A			(X)
Aroclor-1248	EPA 8082A	N/A	0.033	N/A	0.03			(X)
Aroclor-1254	EPA 8082A	N/A	0.033	N/A	0.06			(X)
Aroclor-1260	EPA 8082A	N/A	0.033	N/A	0.005			(X)
PCBs (sum of aroclors)	EPA 8082A	0.033	<0.1	0.023 (<0.10)	0.023	X		
DIOXINS AND FURANS (ONLY ONE SUB-SAMPLE - ARCHIVE OTHERS)								
2,3,7,8 -TCDD	EPA 1613B	N/A	0.000001	(<0.0000005)	0.0000036*	X	X	X
1,2,3,7,8-PeCDD	EPA 1613B	N/A	0.0000025	N/A	N/A	X	X	X
1,2,3,4,7,8-HxCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,6,7,8-HxCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,7,8,9-HxCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,4,6,7,8-HpCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
OCDD	EPA 1613B	N/A	0.00001	N/A	N/A	X	X	X
2,3,7,8-TCDF	EPA 1613B	N/A	0.000001	N/A	N/A	X	X	X
1,2,3,7,8-PeCDF	EPA 1613B	N/A	0.0000025	N/A	N/A	X	X	X
2,3,4,7,8-PeCDF	EPA 1613B	N/A	0.0000025	N/A	N/A	X	X	X
1,2,3,4,7,8-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,6,7,8-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
2,3,4,6,7,8-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,7,8,9-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,4,6,7,8-HpCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,4,7,8,9-HpCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
OCDF	EPA 1613B	N/A	0.00001	N/A	N/A	X	X	X

Table A: Sediment Chemistry Tests for New York Sites with NO Clamshell Dredging

Test Analyte	EPA Method	NYSDEC Required Method Detection Limits (mg/kg, ppm)	NJDEP or USACE Required Reporting Limits[1] (mg/kg, ppm)	NYSDEC No Appreciable Contamination Threshold Values[2] (mg/kg, ppm)	NJDEP Low Effects Range[3] (mg/kg, ppm)	Listed in NYSDEC TOGs 5.1.9	Listed in USACE Reg2 Guidance	Listed in NJDEP 1997 Manual
Dioxins/Furans - Total Toxicity Equivalency Factor	EPA 1613B	0.000002	N/A	<0.0000045	0.00000365	X	X	X
PESTICIDES								
4,4'-DDE	EPA 8081B	N/A	0.001	N/A	0.0022		X	X
2,4'-DDE	EPA 8081B	N/A	0.001	N/A	N/A		X	
4,4'-DDD	EPA 8081B	N/A	0.001	N/A	0.002		X	X
2,4'-DDD	EPA 8081B	N/A	0.001	N/A	N/A		X	
4,4'-DDT	EPA 8081B	N/A	0.001	N/A	0.001		X	X
2,4'-DDT	EPA 8081B	N/A	0.001	N/A	N/A		X	
Sum of DDT+DDE+DDD	EPA 8081B	0.0033	N/A	<0.003 (<0.044)	0.0016	X	X	X
Mirex	EPA 8081B	0.189	N/A	<0.0014 (<0.12)	0.007	X	X	X
Chlordane (Total/Technical)	EPA 8081B	0.0017	N/A	<0.003 (<0.063)	0.007	X	X	X
Dieldrin	EPA 8081B	0.0033	0.001	<0.11 (<0.006)	0.002	X	X	X
PAHs								
Acenaphthene	EPA 8270D	N/A	0.1	N/A	0.016	X	X	X
Acenaphthylene	EPA 8270D	N/A	0.1	N/A	0.044	X	X	X
Anthracene	EPA 8270D	N/A	0.1	N/A	0.085	X	X	X
Benz(a)anthracene	EPA 8270D	N/A	0.1	N/A	0.261	X	X	X
Benzo(b)fluoranthene	EPA 8270D	N/A	0.1	N/A	1.8*	X	X	X
Benzo(k)fluoranthene	EPA 8270D	N/A	0.1	N/A	0.24	X	X	X
Benzo(g,h,i)perylene	EPA 8270D	N/A	0.1	N/A	0.17	X	X	X
Benzo(a)pyrene	EPA 8270D	N/A	0.1	N/A	0.43	X	X	X
2-Chloronaphthalene	EPA 8270D	0.1	0.66	N/A	N/A	X		X
Chrysene	EPA 8270D	N/A	0.1	N/A	0.384	X	X	X
Dibenz(a,h)anthracene	EPA 8270D	N/A	0.1	N/A	0.063	X	X	X
Fluoranthene	EPA 8270D	N/A	0.1	N/A	0.6	X	X	X
Fluorene	EPA 8270D	N/A	0.1	N/A	0.019	X	X	X
Indeno(1,2,3-c,d)-pyrene	EPA 8270D	N/A	0.1	N/A	0.2	X	X	X
2-Methylnaphthalene	EPA 8270D	0.1	0.66	N/A	0.07	X		X
Naphthalene	EPA 8270D	N/A	0.1	N/A	0.16	X	X	X
Phenanthrene	EPA 8270D	N/A	0.1	N/A	0.24	X	X	X
Pyrene	EPA 8270D	N/A	0.1	N/A	0.665	X	X	X
Total PAH (Sum of Target Compound List above)	EPA 8270D	0.33	N/A	<4 (<4)	4	X	X	X
VOLATILES								
Benzene	EPA 8260C	0.0003	0.01	<0.59 (<0.46)	0.34	X		X
Ethylbenzene	EPA 8260C	N/A	0.01	(<0.11)	1.4	X		X
Toluene	EPA 8260C	N/A	0.01	(<0.80)	2.5	X		X
Xylenes	EPA 8260C	N/A	0.01	(<0.091)	>0.12	X		X
Total BTEX	EPA 8260C	0.0008	0.01	<0.96	N/A	X		
MISCELLANEOUS								
Total Organic Carbon (TOC)	EPA 9060A	Not established	Not established	N/A	N/A	X	X	X

KEY:

N/A = Not available

1. Unshaded cells present reporting limits from USACE/EPA *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal* (2014). Shaded Cells present reporting limits from *The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters* (1997). If both documents provide reporting limits, only the lowest is shown here.

2. Primary threshold value from NYS TOGS 5.1.9 (2004). Value in parathesis from NYS *Screening and Assessment of Contaminated Sediment* (2014).

3. NJDEP Ecological Screening Criteria (2009). Values denoted with * are Effects Range Medium.

^Use the laboratory reporting limit if the value is less than the method detection limit shown in the table.

Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Vibracore Site Numbers [Total of 37]: VC1 through VC10; VC15 through VC18; VC35 through VC41; VC45 through VC55; and VC77 through VC 81								
Test Analyte	**EPA Method**	**NYSDEC Required Method Detection Limits (mg/kg, ppm)**	**NJDEP or USACE Required Reporting Limits[1] (mg/kg, ppm)**	**NYSDEC No Appreciable Contamination Threshold Values[2] (mg/kg, ppm)**	**NJDEP Low Effects Range[3] (mg/kg, ppm)**	**Listed in NYSDEC TOGs 5.1.9**	**Listed in USACE Reg2 Guidance**	**Listed in NJDEP 1997 Manual**
METALS								
Arsenic	EPA 6020A	3^	1	<14 (<8.2)	8.2	X	X	X
Aluminum	EPA 6020A	N/A	40	N/A	18000*			X
Antimony	EPA 6020A	N/A	12	N/A	9.3*			X
Barium	EPA 6020A	N/A	40	N/A	48*			X
Beryllium	EPA 6020A	N/A	1	N/A	N/A			X
Cadmium	EPA 6020A	1	1	<1.2 (<1.2)	1.2	X	X	X
Calcium	EPA 6020A	N/A	1,000	N/A	N/A			X
Chromium	EPA 6020A	N/A	1	(<81)	81		X	X
Cobalt	EPA 6020A	N/A	10	N/A	10*			X
Copper	EPA 6020A	5^	1	<33 (<34)	34	X	X	X
Iron	EPA 6020A	N/A	20	N/A	N/A			X
Lead	EPA 6020A	2^	0.6	<33 (<47)	47	X	X	X
Magnesium	EPA 6020A	N/A	1,000	N/A	N/A			X
Manganese	EPA 6020A	N/A	3	N/A	260*			X
Mercury	EPA 7474	0.2^	0.1	<0.17 (<0.15)	0.15	X	X	X
Molybdenum	EPA 6020A	N/A	N/A	N/A	N/A			
Nickel	EPA 6020A	N/A	1	(<21)	21		X	X
Potassium	EPA 6020A	N/A	1,000	N/A	N/A			X
Selenium	EPA 6020A	N/A	1	N/A	1*			X
Silver	EPA 6020A	N/A	1	(<1.0)	1		X	X
Sodium	EPA 6020A	N/A	1,000	N/A	N/A			X
Strontium	EPA 6020A	N/A	N/A	N/A	N/A			
Thallium	EPA 6020A	N/A	2	N/A	N/A			X
Tin	EPA 6020A	N/A	N/A	N/A	N/A			
Titanium	EPA 6020A	N/A	N/A	N/A	N/A			
Vanadium	EPA 6020A	N/A	10	N/A	57*			X
Zinc	EPA 6020A	N/A	4	(<150)	150			X
PCB Congeners (NJDEP congener list confirmed by Mr. Jeff Thein on 10/19/2016)								
PCB 8	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 18	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 28	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 44	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 49	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 52	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 66	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 87	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 101	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 105	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 118	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 128	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 138	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 153	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 170	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 180	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 183	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 184	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 187	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 195	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 206	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCB 209	EPA 1668C	N/A	0.001	N/A	N/A		X	(X)
PCBs - Total Toxicity Equivalency Factor	EPA 1668C	N/A	N/A	0.023 (<0.10)	0.023			
PCB AROCLORS (Aroclor list from 1997 NJDEP manual applied for NY sum.)								
Aroclor-1016	EPA 8082A	N/A	0.033	N/A	0.007			(X)
Aroclor-1221	EPA 8082A	N/A	0.067	N/A	N/A			(X)
Aroclor-1232	EPA 8082A	N/A	0.033	N/A	N/A			(X)
Aroclor-1242	EPA 8082A	N/A	0.033	N/A	N/A			(X)
Aroclor-1248	EPA 8082A	N/A	0.033	N/A	0.03			(X)
Aroclor-1254	EPA 8082A	N/A	0.033	N/A	0.06			(X)
Aroclor-1260	EPA 8082A	N/A	0.033	N/A	0.005			(X)

Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Test Analyte	EPA Method	NYSDEC Required Method Detection Limits (mg/kg, ppm)	NJDEP or USACE Required Reporting Limits[1] (mg/kg, ppm)	NYSDEC No Appreciable Contamination Threshold Values[2] (mg/kg, ppm)	NJDEP Low Effects Range[3] (mg/kg, ppm)	Listed in NYSDEC TOGs 5.1.9	Listed in USACE Reg2 Guidance	Listed in NJDEP 1997 Manual
PCBs (sum of aroclors)	EPA 8082A	0.033	<0.1	0.023 (<0.10)	0.023	X		
DIOXINS AND FURANS								
2,3,7,8 -TCDD	EPA 1613B	N/A	0.000001	(<0.0000005)	N/A	X	X	X
1,2,3,7,8-PeCDD	EPA 1613B	N/A	0.0000025	N/A	N/A	X	X	X
1,2,3,4,7,8-HxCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,6,7,8-HxCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,7,8,9-HxCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,4,6,7,8-HpCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
OCDD	EPA 1613B	N/A	0.00001	N/A	N/A	X	X	X
2,3,7,8-TCDF	EPA 1613B	N/A	0.000001	N/A	N/A	X	X	X
1,2,3,7,8-PeCDF	EPA 1613B	N/A	0.0000025	N/A	N/A	X	X	X
2,3,4,7,8-PeCDF	EPA 1613B	N/A	0.0000025	N/A	N/A	X	X	X
1,2,3,4,7,8-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,6,7,8-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
2,3,4,6,7,8-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,7,8,9-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,4,6,7,8-HpCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,4,7,8,9-HpCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
OCDF	EPA 1613B	N/A	0.00001	N/A	N/A	X	X	X
Dioxins/Furans - Total Toxicity Equivalency Factor	EPA 1613B	0.000002	N/A	<0.0000045	0.00000365	X	X	X
PESTICIDES								
alpha-BHC	EPA 8081B	N/A	0.0019	N/A	N/A			X
beta-BHC	EPA 8081B	N/A	0.0033	N/A	N/A			X
delta-BHC	EPA 8081B	N/A	0.0017	N/A	N/A			X
gamma-BHC (Lindane)	EPA 8081B	N/A	0.002	(<0.001)	N/A			X
Heptachlor	EPA 8081B	N/A	0.001	(<0.071)	0.0003*		X	X
Aldrin	EPA 8081B	N/A	0.001	N/A	0.002		X	X
Heptachlor epoxide	EPA 8081B	N/A	0.001	(<0.015)	0.005		X	X
Endosulfan I	EPA 8081B	N/A	0.001	(<0.0001)	N/A		X	X
Endrin	EPA 8081B	N/A	0.0036	(<0.006)	0.003			X
Endosulfan II	EPA 8081B	N/A	0.001	N/A	N/A		X	X
Endosulfan sulfate	EPA 8081B	N/A	0.001	N/A	N/A		X	X
Methoxychlor	EPA 8081B	N/A	0.017	(<0.059)	N/A			X
Endrin ketone	EPA 8081B	N/A	0.0033	N/A	N/A			X
Endrin aldehyde	EPA 8081B	N/A	0.0033	N/A	N/A			X
alpha-Chlordane	EPA 8081B	N/A	0.001	N/A	N/A		X	X
gamma-Chlordane	EPA 8081B	N/A	0.0017	N/A	N/A			X
Toxaphene	EPA 8081B	N/A	0.17	(<0.054)	N/A			X
Trans nonachlor	EPA 8081B	N/A	0.001	N/A	N/A		X	X
4,4'-DDE	EPA 8081B	N/A	0.001	N/A	0.0022		X	X
2,4'-DDE	EPA 8081B	N/A	0.001	N/A	N/A		X	
4,4'-DDD	EPA 8081B	N/A	0.001	N/A	0.002		X	X
2,4'-DDD	EPA 8081B	N/A	0.001	N/A	N/A		X	
4,4'-DDT	EPA 8081B	N/A	0.001	N/A	0.001		X	X
2,4'-DDT	EPA 8081B	N/A	0.001	N/A	N/A		X	
Sum of DDT+DDE+DDD	EPA 8081B	0.0033	N/A	<0.003 (<0.044)	0.0016	X	X	X
Mirex	EPA 8081B	0.189	N/A	<0.0014 (<0.12)	0.007	X	X	X
Chlordane (Total/Technical)	EPA 8081B	0.0017	N/A	<0.003 (<0.063)	0.007	X	X	X
Dieldrin	EPA 8081B	0.0033	0.001	<0.11 (<0.006)	0.002	X	X	X
SEMI-VOLATILES/PAHs								
Acenaphthene (LMW)	EPA 8270D	N/A	0.1	N/A	0.016	X	X	X
Acenaphthylene (LMW)	EPA 8270D	N/A	0.1	N/A	0.044	X	X	X
Anthracene (LMW)	EPA 8270D	N/A	0.1	N/A	0.085	X	X	X
Benz(a)anthracene (HMW)	EPA 8270D	N/A	0.1	N/A	0.261	X	X	X
Benzo(b)fluoranthene (HMW)	EPA 8270D	N/A	0.1	N/A	1.8*	X	X	X
Benzo(k)fluoranthene (HMW)	EPA 8270D	N/A	0.1	N/A	0.24	X	X	X
Benzo(g,h,i)perylene (HMW)	EPA 8270D	N/A	0.1	N/A	0.17	X	X	X
Benzo(a)pyrene (HMW)	EPA 8270D	N/A	0.1	N/A	0.43	X	X	X
2-Chloronaphthalene	EPA 8270D	0.1	0.66	N/A	N/A	X		X
Chrysene (HMW)	EPA 8270D	N/A	0.1	N/A	0.384	X	X	X
Dibenz(a,h)anthracene (HMW)	EPA 8270D	N/A	0.1	N/A	0.063	X	X	X
Fluoranthene (HMW)	EPA 8270D	N/A	0.1	N/A	0.6	X	X	X
Fluorene (LMW)	EPA 8270D	N/A	0.1	N/A	0.019	X	X	X

Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Test Analyte	EPA Method	NYSDEC Required Method Detection Limits (mg/kg, ppm)	NJDEP or USACE Required Reporting Limits[1] (mg/kg, ppm)	NYSDEC No Appreciable Contamination Threshold Values[2] (mg/kg, ppm)	NJDEP Low Effects Range[3] (mg/kg, ppm)	Listed in NYSDEC TOGs 5.1.9	Listed in USACE Reg2 Guidance	Listed in NJDEP 1997 Manual
Indeno(1,2,3-c,d)-pyrene (HMW)	EPA 8270D	N/A	0.1	N/A	0.2	X	X	X
2-Methylnaphthalene	EPA 8270D	0.1	0.66	N/A	0.07	X		X
Naphthalene (LMW)	EPA 8270D	N/A	0.1	N/A	0.16	X	X	X
Phenanthrene (LMW)	EPA 8270D	N/A	0.1	N/A	0.24	X	X	X
Pyrene (HMW)	EPA 8270D	N/A	0.1	N/A	0.665	X	X	X
Total PAH (Sum of Target Compound List above)	EPA 8270D	0.33	N/A	<4 (<4)	4	X		X
Sum of Low-Molecular Weight (LMW) PAHs	EPA 8270D	N/A	0.1	N/A	N/A		X	
Sum of High-Molecular Weight (HMW) PAHs	EPA 8270D	N/A	0.1	N/A	N/A		X	
1,4-Dichlorobenzene	EPA 8270D	N/A	0.1	(<1.2)	0.110*		X	X
Phenol	EPA 8270D	N/A	0.66	N/A	0.130*			X
bis-(2-Chloroethyl)ether	EPA 8270D	N/A	0.66	N/A	N/A			X
2-Chlorophenol	EPA 8270D	N/A	0.66	N/A	0.008*			X
1,3-Dichlorobenzene	EPA 8270D	N/A	0.66	(<2.1)	N/A			X
1,2-Dichlorobenzene	EPA 8270D	N/A	0.66	(<0.85)	0.013*			X
2-Methylphenol	EPA 8270D	N/A	0.66	N/A	N/A			X
2,2'-oxybis(1-Chloropropane)	EPA 8270D	N/A	0.66	N/A	N/A			X
4-Methylphenol	EPA 8270D	N/A	0.66	N/A	N/A			X
N-Nitroso-di-n-propylamine	EPA 8270D	N/A	0.66	N/A	N/A			X
Hexachloroethane	EPA 8270D	N/A	0.66	N/A	0.073*			X
Nitrobenzene	EPA 8270D	N/A	0.66	N/A	N/A			X
Isophorone	EPA 8270D	N/A	0.66	N/A	N/A			X
2-Nitrophenol	EPA 8270D	N/A	0.66	N/A	N/A			X
2,4-Dimethylphenol	EPA 8270D	N/A	0.66	N/A	N/A			X
bis(2-Chloroethoxy)methane	EPA 8270D	N/A	0.66	N/A	N/A			X
2,4-Dichlorophenol	EPA 8270D	N/A	0.66	N/A	0.005*			X
1,2,4-Trichlorobenzene	EPA 8270D	N/A	0.66	(<2.0)	>0.0048			X
4-Chloroaniline	EPA 8270D	N/A	1.3	N/A	N/A			X
Hexachlorobutadiene	EPA 8270D	N/A	0.66	N/A	0.0013*			X
4-Chloro-3-methylphenol	EPA 8270D	N/A	1.3	N/A	N/A			X
Hexachlorocylcopentadiene	EPA 8270D	N/A	0.66	(<0.13)	N/A			X
2,4,6-Trichlorophenol	EPA 8270D	N/A	0.66	N/A	0.006*			X
2,4,5-Trichlorophenol	EPA 8270D	N/A	0.66	N/A	0.003*			X
2-Nitroaniline	EPA 8270D	N/A	3.3	N/A	N/A			X
Dimethylphthalate	EPA 8270D	N/A	0.66	N/A	N/A			X
2,6-Dinitrotoluene	EPA 8270D	N/A	0.66	N/A	N/A			X
3-Nitroaniline	EPA 8270D	N/A	3.3	N/A	N/A			X
2,4-Dinitrophenol	EPA 8270D	N/A	3.3	N/A	N/A			X
4-Nitrophenol	EPA 8270D	N/A	3.3	N/A	N/A			X
Dibenzofuran	EPA 8270D	N/A	0.66	N/A	N/A			X
2,4-Dinitrotoluene	EPA 8270D	N/A	0.66	N/A	N/A			X
Diethylphthalate	EPA 8270D	N/A	0.66	N/A	0.006*			X
4-Chlorophenyl-phenyl ether	EPA 8270D	N/A	0.66	N/A	N/A			X
4-Nitroaniline	EPA 8270D	N/A	0.83	N/A	N/A			X
4,6-Dinitro-2-methylphenol	EPA 8270D	N/A	3.3	N/A	N/A			X
N-Nitroso-diphenylamine	EPA 8270D	N/A	0.66	N/A	N/A			X
4-Bromophenyl-phenylether	EPA 8270D	N/A	0.66	N/A	N/A			X
Hexachlorobenzene	EPA 8270D	N/A	0.66	N/A	0.02			X
Pentachlorophenol	EPA 8270D	N/A	3.3	(<21)	N/A			X
Carbazole	EPA 8270D	N/A	0.33	N/A	N/A			X
Di-n-butylphthalate	EPA 8270D	N/A	0.33	N/A	0.058*			X
Butylbenzylphthalate	EPA 8270D	N/A	0.66	N/A	0.063*			X
3,3'-Dichlorobenzidine	EPA 8270D	N/A	1.3	N/A	N/A			X
bis(2-Ethylhexyl)phthalate	EPA 8270D	N/A	0.66	N/A	0.18216			X
Di-n-octylphthalate	EPA 8270D	N/A	0.66	N/A	N/A			X
VOLATILES								
Benzene	EPA 8260C	0.0003	0.01	<0.59 (<0.46)	0.34	X		X
Ethylbenzene	EPA 8260C	N/A	0.01	(<0.11)	1.4	X		X
Toluene	EPA 8260C	N/A	0.01	(<0.80)	2.5	X		X
Xylenes	EPA 8260C	N/A	0.01	(<0.091)	>0.12	X		X
Total BTEX	EPA 8260C	0.0008	0.01	<0.96	N/A	X		
MISCELLANEOUS								

Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Test Analyte	EPA Method	NYSDEC Required Method Detection Limits (mg/kg, ppm)	NJDEP or USACE Required Reporting Limits[1] (mg/kg, ppm)	NYSDEC No Appreciable Contamination Threshold Values[2] (mg/kg, ppm)	NJDEP Low Effects Range[3] (mg/kg, ppm)	Listed in NYSDEC TOGs 5.1.9	Listed in USACE Reg2 Guidance	Listed in NJDEP 1997 Manual
Total Organic Carbon (TOC)	EPA 9060A	Not established	Not established	N/A	N/A	X	X	X
Cyanide (Total/Amenable)	EPA 9012B	N/A	0.5	N/A	N/A			X

KEY:

N/A = Not available

1. Unshaded cells present reporting limits from USACE/EPA *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal* (2014). Shaded Cells present reporting limits from *The Management*

2. Primary threshold value from NYS TOGS 5.1.9 (2004). Value in parathesis from NYS *Screening and Assessment of Contaminated Sediment* (2014).

3. NJDEP Ecological Screening Criteria (2009). Values denoted with * are Effects Range Medium.

^Use the laboratory reporting limit if the value is less than the method detection limit shown in the table.

Appendix D Chemical Laboratory Standard Operating Procedures

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Percent Solids Determination

References: **SM 2540G,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: This method is applicable to samples such as soils, sediments, sludges separated from water and wastewater treatment processes, sludge cakes, and any tissue.

Definitions: Refer to Alpha Analytical Quality Manual.

Percent solids are the total amount of solid material remaining in the weighing tin, after sample drying at a defined temperature, expressed as a percent. This measurement is used in the calculation of analytical results, both organic and inorganic, to express these results on a “dry” weight basis.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of experienced analysts and in the interpretation of data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

A well-mixed aliquot of solid sample is placed into an aluminum weighing tin. The aliquot dried to a constant weight, generally overnight, in an oven at 103°C - 105°C. The aliquot is then re-weighed, and calculations are applied (Section 11) to determine the percent of solid material.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Method Detection Limit (MDL) studies for the determination of quantitation limits, do not apply to this method or SOP.

4. Interferences

4.1 The temperature at which the sample is dried has an important bearing on the results because weight losses due to volatilization of organic matter, water crystallization, gases from heat

induced chemical decomposition, as well as weight gains due to oxidation, are dependent on temperature and the time of heating. Oven temperature and time must be recorded at the time the samples are placed into the oven and at the time they are removed from the oven.

4.2 It is important that all weighing, pre- and post-sample drying, be performed quickly and at ambient temperatures. Sample cooling must take place in a desiccator, to avoid loss or gain of weight with temperature fluctuations and the re-introduction of moisture to the samples. Tongs must be used when handling weighing tins, as oil residue from hands may contribute to the sample weights. Small differences in weights can significantly affect the final calculation.

4.3 All weights must be recorded on the Top loading balance to the nearest 0.01g or on the Analytical balance to 0.0001g. The balance to be used depends upon the amount of sample being dried. If less than 1g is to be dried, smaller weighing tins, and the Analytical balance, must be used.

4.4 Decanting Standing Water from Sediments Samples

The practice of decanting standing water from sediments and soil samples is **only performed upon Client Request.** The Project Manager must be consulted prior to initiating the following protocol.

- Samples will be inspected upon arrival by the Sample Custodian for standing water.
- If the samples do not have standing water, or have 0.0 - 0.25 inches, or 0.0 – 1.0 centimeters of standing water, this will be considered to be pore water that has settled out of the sample during shipping. This water will be mixed back into the sample prior to the removal of any aliquot for percent solids determination.
- If the sample has more than 0.0 - 0.25 inches, or 0.0 – 1.0 centimeters of standing water, the Project Manager will be contacted by the Sample Custodian to inspect the samples. The Project Manager will give the instruction to decant the water, or to mix the water into the sample. The Project Manager must make this decision either through prior knowledge of the site and the client requirements, or must contact the client for instructions.
- If instructed to decant the water, all samples will be decanted at this time by the Sample Custodian or designee. An “X” will be marked on the top of the sample jar in indelible ink and a notation that the standing water has been decanted from the sample will made in the Percent Solids logbook. The Project Manager will also note this in the project narrative to the client.
- If instructed to mix the standing water back into the sample, this will be done at the time the aliquot for percent solids is removed, and no further notations are needed.
- At no time are any analysts, other than the Sample Custodian or designee, to decant standing water from samples. If samples are removed from refrigerators or freezers and the analyst notes that there is standing water on the sample, this is to be mixed back into the sample, as this water is to be assumed to be due to further settling of the pore water.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From

this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Eating, drinking, smoking, or the application of cosmetics is not permitted in the laboratory area. Horseplay of any kind is prohibited. Pipetting by mouth is not permitted. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and before entering the employee lounge or eating area. Always wash your hands before leaving the laboratory. All relevant Material Safety Data Sheets (MSDSs) are kept alphabetically in the centrally located file storage, in the common area outside of the Information Technology (IT) offices.

5.2 Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled under a hood. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting devise and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

5.3 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to chemicals must be reduced to the lowest possible level by whatever means available. All standards and reagents shall be prepared in a hood while using the proper PPE.

5.4 Spilled samples, solvents, reagents, and water must be cleaned up from bench tops, instruments and autosampler surfaces immediately. A spill is considered a quantity of hazardous material if it is two times greater than the normal working volume. Concentrated solvents, acids or bases present a moderate to extreme hazard to the skin and mucous membranes. If contact with the skin occurs, immediately flush with large volumes of water. In the case of acidic/basic spills, the *Spill Kit* located in each laboratory shall be utilized before attempting to cleanup the spill. Although procedures are designed to minimize the possibility of an accident, all injuries or accidents, regardless of the nature or severity, are to be reported to the Department Manager immediately. If an employee discovers a potentially unsafe condition, this must be reported to the Department Manager immediately. No employee should feel compelled to work in a situation where they do not feel entirely informed, trained, or safe.

5.5 Tongs must always be used when removing hot aluminum weighing tins from the oven and transferring them to the desiccator to avoid burning the skin.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are typically collected in 4oz or 8oz glass jars.

6.2 Sample Preservation

No preservatives are used. See Section 6.4.

6.3 Sample Shipping

No special sample shipping requirements. Typical shipping procedures may be found in the Sample Receipt and Login SOP (SOP 1559).

6.4 Sample Handling

Samples are stored at 4°C ± 2°C. Sediment and tissue samples are stored at -20°C ± 10°C.

Refer to the Sample Receipt and Login SOP (SOP 1559) and the Sample Custody SOP (SOP 1560).

7. Equipment and Supplies

7.1 Analytical balance: Capable of weighing to the nearest 0.0001g, and Top loading balance capable of weighing to the nearest 0.01g.

The balances used for each weighing (both Top loading and Analytical) for this method are checked daily before use with Class S-1 weights, in the weight range of use, by a designated analyst, or appointed alternate. These daily checks are documented in the "Balance Calibration" logbook. All balances are serviced annually by an outside service technician.

7.2 Aluminum weighing tins: "Standard" size from Fisher Scientific (No.: 08-732), or equivalent. "Small" size for weighing less than 1g of material from Perkin-Elmer (No.: N241-1362), or equivalent.

7.3 Drying oven: Set at 103°C – 105°C, vented and/or placed under a fume hood if possible.

7.4 Clean stainless steel spatulas: One for each sample to be weighed.

7.5 Desiccator: With a desiccant containing a color indicator for moisture content.

7.6 Indelible ink Sharpie

7.7 Tongs and tweezers

8. Reagents and Standards

No standards or reagents are required for this method.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Not applicable to this method.

9.2 Laboratory Control Sample (LCS)

Not applicable to this method.

9.3 Initial Calibration Verification (ICV)

Not applicable to this method.

9.4 Continuing Calibration Verification (CCV)

Not applicable to this method.

9.5 Matrix Spike

Not applicable to this method.

9.6 Laboratory Duplicate

Duplicate analyses are performed 1 per 20 samples, or at least 1 per batch, whichever is more frequent. The RPD between the duplicates must be less than 10%. If the RPD between the duplicates exceeds 10% the analyst shall check the calculations for errors. If no calculation errors are found, repeat the analysis for the batch.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Samples 1-20
- Sample Duplicate

10. Procedure

10.1 Equipment Set-up

Samples are obtained from the Sample Custodian. Note removal of the samples in the Internal Tracking COC in the Sample Management office. If samples are removed from the freezer, note the date of removal from, and return to, the freezer, in the freezer logbook for hold time tracking purposes.

10.1.1 If a sample has been selected by the client for the Duplicate analysis, this sample must be weighed in duplicate. Otherwise, select the sample that appears to have the greatest total sample mass for the duplicate.

10.1.2 If samples are those that have been selected for re-extraction, the % Solids are **not** reset. The original % Solids data is used throughout the sample set.

10.2 Initial Calibration

Not applicable to this method.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample aliquots:

NOTE: If there is ever any question about the amount of sample that can be taken for the percent solid determination, see the Department Supervisor for guidance.

10.3.1.1 Soils, Sediments, Sludges and Tissue (homogenized tissue samples that yield greater than 20g of total weight):

- Mix the sample well. See Section 4.4 regarding Client requests for standing water and sediment samples.
- Use the Top loading balance and the *standard* size aluminum weighing tin.
- Label the tins with the dish number for each sample to be weighed using an indelible ink Sharpie.
- Tare the balance to read zero.
- Place the tin on the balance and allow the balance to stabilize. Record the weight of the empty tin in the “mass of dish” column “A” in the Percent Solids logbook to the nearest 0.01g.
- Aliquot approximately 5-10g of sample into the tin.
- Allow the balance to stabilize and record the weight of the sample plus the tin to the nearest 0.01g, in the “mass of dish & wet sample” column “B” in the Percent Solids logbook.

10.3.1.2 Tissue (homogenized tissue samples that yield between 5-20g of total weight) and other low weight solid samples:

- Mix the sample well. See Section 4.4 regarding Client requests for standing water and sediment samples. If weighing a homogenized tissue, use caution to preserve as much tissue sample as possible for future analyses.
- Use the Top loading balance and the *standard* size aluminum weighing tin.
- Label the tins with the dish number for each sample to be weighed using an indelible ink Sharpie.
- Tare the balance to read zero.
- Place the tin on the balance and allow the balance to stabilize. Record the weight of the empty tin in the “mass of dish” column “A” in the Percent Solids logbook to the nearest 0.01g.
- Aliquot approximately 1-2g of sample into the tin.
- Allow the balance to stabilize and record the weight of the sample plus the tin to the nearest 0.01g, in the “mass of dish & wet sample” column “B” in the Percent Solids logbook.

10.3.1.3 Tissue (homogenized tissue samples that yield less than 5g of total weight):

- Carefully mix the homogenate, if possible, using caution to preserve the tissue sample for future analyses.
- Use the Analytical balance and either the *standard* or *small* size aluminum weighing tins.
- Label the tins with the dish number for each sample to be weighed using an indelible ink Sharpie.
- Tare the balance to read zero.
- Place the tin on the balance and allow the balance to stabilize. Record the weight of the empty tin in the “mass of dish” column “A” in the Percent Solids logbook to the nearest 0.0001g.
- Aliquot approximately 200-500mg of sample into the tin.
- Allow the balance to stabilize and record the weight of the sample plus the tin to the nearest 0.0001g, in the “mass of dish & wet sample” column “B” in the Percent Solids logbook.

10.3.2 Place the tins in the drying oven overnight, until a constant weight is achieved (see Section 10.3.5, 10.3.6). Record the oven temperature when samples are put into the oven.

10.3.3 Using tongs or tweezers, quickly and carefully, remove the samples from the oven and place them into the desiccator to cool to room temperature. The samples must stay in the desiccator for at least 20 minutes.

10.3.4 Tare the balance to zero.

10.3.5 Remove the samples one at a time from the desiccator and place the dried sample, in the tin, on the balance and allow the balance to stabilize. Record the weight to the appropriate decimal place in the “mass of dish & dry sample” column “C” of the Percent Solids logbook. All post-drying weighing must be performed as quickly as possible, preferably within 2 minutes, of removing the samples from the desiccator, to prevent any atmospheric moisture from returning to the dried samples.

10.3.6 If a constant weight is not achieved, the sample must be placed back into the oven for approximately one hour. Repeat the cooling and desiccating cycle after this hour. This cycle should be repeated until the weight change is less than 4% or 50mg of the previous weight, whichever is less.

10.4 Continuing Calibration

Not applicable to this method.

10.5 Preventive Maintenance

Ovens are calibrated on an annual basis by an instrument service company. Certificates are kept on file.

Analytical balances are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file. The calibration of the balances is verified on a daily basis and records are kept in a Logbook.

11. Data Evaluation, Calculations and Reporting

11.1 Calculations

11.1.1 Go to the Excel spreadsheet labeled "Percent" on the server.

11.1.2 Transcribe the logbook entries into the correct columns on the spreadsheet template. Save this template as the date the samples were initially weighted (*i.e.,* 081302.xls). The spreadsheet will calculate the % Solid and the RPD for the duplicate analysis, using the following formulas:

$$\textbf{\% Solid} = \frac{\text{Mass of dish \& dry sample "C"} - \text{Mass of dish "A"}}{\text{Mass of dish \& wet sample "B"} - \text{Mass of dish "A"}} \times 100$$

$$\textbf{RPD} = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = result of sample
R2 = result of the duplicate sample

If percent moisture is requested, as opposed to percent solid, apply the following formula:

$$\textbf{\% Moisture} = 100 - \%\ \text{Solid}$$

11.1.3 Go to the LIMS and "batch" and "associate" all samples in this analytical batch.

11.1.4 Enter the results obtained from the Excel spreadsheet.

11.1.5 Forward the hand recorded raw data from the Percent Solid logbook, the Excel spreadsheet and the LIMS batch printout to the Department Manager for secondary review.

11.1.6 Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP 1729. All records shall be stored in such a manner as to be safe and accessible for at least 10 years.

11.1.7 Notebooks: Laboratory notebooks are designed to accommodate the specific analysis. If a sample requires re-analysis or re-extraction for any reason, a notation is made next to the sample entry in that log. Such logbooks shall be archived so as to remain available for at least 10 years. All laboratory notebooks must follow the specifications in the Logbook Usage Work Instructions (WI 1556), and all record keeping and document control practices.

11.2 Data Evaluation and Reporting

All results are reportable without qualification if the QC criteria defined in Section 9 are met. If any QC parameters are not met, all associated samples must be evaluated for re-analysis. See Sections 9 for discussion, including corrective action, for any QC outlier.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to Section 11.2.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

MDL/LOD/LOQ Studies are not applicable to this method.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

The Alpha *Hazardous Waste and Sample Disposal* SOP (1797), must be referenced for disposal of used standards, solvents, acids, reagents or other chemicals.

All solid waste generated during this analysis must be stored in satellite containers in the preparation laboratory labeled “Soil/Solid Waste”.

Once the solid waste satellite containers are full, they must be emptied into a 55-gallon drum marked “Soil/Solid Waste”.

15. Referenced Documents

Sample Receipt and Login SOP (SOP 1559)

Sample Custody and Tracking SOP (SOP 1560)

Hazardous Waste and Sample Disposal SOP (1797)

Logbook Usage Work Instructions (WI 1556)

16. Attachments

None.

Acid Digestion of Solid Samples for Metals Analysis

References: **Method 3050B,** Acid Digestion of Sediments, Sludges and Soils, Test Methods for Evaluating Solid Waste, SW-846, Third Edition (Revision 2) as promulgated in the Final Update, December 1996..

Method 6020A, Inductively Coupled Plasma-Mass Spectrometry, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Draft Update IVA, May 1998.

EPA/625/R-96/110a, Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.1. SELECTION, PREPARATION AND EXTRACTION OF FILTER MATERIAL. U.S. EPA, Cincinnati, OH 45269, June, 1999.

1. Scope and Application

Matrices: The method is applicable to soil and sediment.

Method Instrumentation: The method is applicable to ICP and ICP/MS analysis.

Definitions: Refer to Alpha Analytical Quality Manual.

Two procedures are discussed in this SOP.

Lab Method 3050:1T-**restricted to non-regulatory sediment/tissue projects only**

This acid digestion procedure is used to prepare solid samples for analysis of total metals by ICPM MS. Silver, antimony, and tin can be analyzed by ICP MS using this digestion.

Element	CASRN
Antimony (Sb)	7440-36-0
Silver (Ag)	7440-22-4
Tin (Sn)	7440-31-5

Lab Method 3050:2T

Samples prepared by this method may be analyzed by ICP and ICP-MS for all the metals listed below.
The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Element	CASRN	Element	CASRN	Element	CASRN
Aluminum (Al)	7429-90-5	Cobalt (Co)	7440-48-4	Nickel (Ni)	7440-02-0
Antimony (Sb)	7440-36-0	Copper (Cu)	7440-50-8	Selenium (Se)	7782-49-2
Arsenic (As)	7440-38-2	Iron (Fe)	7439-89-6	Sodium (Na)	7440-23-5
Barium (Ba)	7440-39-3	Potassium (K)	7440-09-7	Silver (Ag)	7440-22-4
Beryllium (Be)	7440-41-7	Lead (Pb)	7439-92-1	Thallium (Tl)	7440-28-0
Cadmium (Cd)	7440-43-9	Magnesium (Mg)	7439-95-4	Tin (Sn)	7440-31-5
Calcium (Ca)	7440-70-2	Manganese (Mn)	7439-96-5	Titanium (Ti)	7440-32-6
Chromium (Cr)	7440-47-3	Molybdenum (Mo)	7439-98-7	Vanadium (V)	7440-62-2
Cobalt (Co)	7440-48-4	Nickel (Ni)	7440-02-0	Zinc (Zn)	7440-66-6

2. Summary of Method

Lab Method 3050:1T

7.5 mL of concentrated hydrochloric acid and 2.5 mL of concentrated nitric acid are added to 1-2g of solid sample measured into a digestion tube. The sample is heated in a block at 90-95°C for 75 minutes After cooling, the sample is diluted up to 50mL with deionized water.

If the sample should go to dryness, the sample must be re-prepared.

All solid samples require filtration due to insoluble silicates and the formation of precipitates during digestion.

Lab Method 3050:2T

5mL of 1:1 nitric acid is added to 1-2g of solid sample measured into a digestion tube. For analysis of an air sampling filter, cut a one inch by eight inch section of filter using high carbon steel scissors and place into a digestion vial. The sample is heated in a block at 90-95°C for 15 minutes. 2.5mL of concentrated nitric acid is added, and the sample is refluxed for 30 minutes. The sample is then cooled and 2.5mL of concentrated nitric acid is added again. The sample is refluxed for an additional 30 minutes. 0.5mL of 30% hydrogen peroxide is added to the cooled sample and the sample is re-heated in a block until the sample no longer is effervescing. 1.0mL of hydrogen peroxide is added to the sample three more times and sample is heated in the block between each aliquot addition of 30% hydrogen peroxide. After cooling, add 2.5 mL HCl and 5 mL H_2O to the sample digest and heat the sample to 95°C ± 5C. Reflux at 95°C ± 5C without boiling for 5 minutes then sample is diluted up to 50mL with deionized water.

If the sample should go to dryness, the sample must be re-prepared.

All solid samples require filtration due to insoluble silicates and the formation of precipitates during digestion.

2.1 Method Modifications from Reference

A final extract volume of 50mL is used instead of 100mL specified in the reference method. All reagent volumes have been reduced proportionately.

3. Reporting Limits

For information regarding the RLs, see the appropriate analytical SOPs. These values are equivalent to the lowest standard in the initial calibration curve, and may be approximately 3-5X the calculated MDL.

4. Interferences

All re-usable glassware or plastic must be acid cleaned prior to use to remove residual trace elements.

Based on historical information, or by sample observation, it may be noted that all solid samples will require filtration after digestion, and prior to analysis, due to insoluble silicates and the formation of precipitates. After sample digestion, the associated method blank and laboratory control sample must be filtered with the associated batch samples.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Eating, drinking, smoking, or the application of cosmetics is not permitted in the laboratory area. Horseplay of any kind is prohibited. Pipetting by mouth is not permitted. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and before entering the employee lounge or eating area. Always wash your hands before leaving the laboratory. All relevant Material Safety Data Sheets (MSDSs) are kept alphabetically in the centrally located file storage, in the common area outside of the Information Technology (IT) offices.

Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled under a hood using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting device and/or a secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

Spilled samples, solvents, reagents, and water must be cleaned up from bench tops, instruments and autosampler surfaces immediately. A spill is considered a quantity of hazardous material if it is two times greater than the normal working volume. Concentrated solvents, acids or bases present a moderate to extreme hazard to the skin and mucous membranes. If contact with the skin occurs, immediately flush with large volumes of water. In the case of acidic/basic spills, the *Spill Kit* located in each laboratory shall be utilized before attempting to cleanup the spill. Although procedures are designed to minimize the possibility of an accident, all injuries or accidents, regardless of the nature

or severity, are to be reported to the Section Head Supervisor immediately. If an employee discovers a potentially unsafe condition, this must be reported to the Section Head Supervisor immediately. No employee should feel compelled to work in a situation where they do not feel entirely informed, trained, or safe.

Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintenance an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

Compressed gas cylinders shall only be moved with the dolly supplied for this specific purpose. The cap must be on the cylinder while it is being moved. The tank must be secured when in its final position. All spent tanks are to be returned in the same manner, and secured until removed by the vendor. Liquid argon or nitrogen represents a potential cryogenic hazard and safe-handling procedures must be used at all times.

All additional company safety practices shall be followed at all times as written in the *Chemical Hygiene Plan*.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

A minimum of 10g of sample must be collected in a pre-cleaned 2oz or 4oz glass jar with a Teflon lined screw cap.

6.2 Sample Preservation

No preservatives are used. See Section 6.4.

6.3 Sample Shipping

No special sample shipping requirements. Typical shipping procedures may be found in the Alpha Sample Receipt & Log-In SOP (1559).

6.4 Sample Handling

Samples are stored at 4°C $\pm$ 2°C. Sediment and soil samples may be stored at -20°C $\pm$ 10°C. The hold time for solid samples for metals digestion is 6 months. Air sampling filters may be stored at room temperature. Tissue samples are stored frozen with a hold time of 1 year.

Refer to the Sample Receipt & Log-In SOP (1559) for Sample Receipt, Login and internal Sample Custody information.

7. Equipment and Supplies

7.1 Digestion Tubes: Pre-cleaned, graduated, disposable, 50 mL volume. The 50mL volume of each Lot of tubes is verified and documented in a logbook (Form No.: 11453).

7.2 Watch covers

7.3 Filter Funnels: Glass or plastic

7.4 Electric Hot Plate: Adjustable and capable of maintaining a temperature of 90-95°C equipped with graphite carbon blocks that each have 36 positions to hold the sample tubes.

NOTE: Hotplate/Block temperatures are monitored and recorded regularly using NIST calibrated and traceable thermometers. If any thermometer is suspected to not be reading the temperatures correctly, see the QAO for a certified replacement thermometer.

7.5 Other: Adjustable Eppendorf pipettes and replacement tips, pre-cleaned Filter Mate from Environmental Express (2.0um filter paper and plunger), 100mL and 200mL Class-A volumetric flasks, high carbon steel scissors.

8. Reagents and Standards

ACS Trace Metal grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

Solutions below expire 6 months from preparation, or expire on the manufacturer's expiration date, whichever comes first. Standards are stored out of direct light at ambient temperature. Reagents are stored at ambient temperature, under a hood if necessary. Acids are kept in a storage crate under a hood. Once opened, initialed and dated, they are kept in a hood. Acid expiration dates are generally provided by the manufacturer and printed on the label.

8.1 Deionized (DI) water: The DI water is ASTM Type II laboratory reagent grade water or better (*i.e.,* Type I). The Barnstead NANO-pure system provides Type I water used in the preparation of samples and standards.

8.2 Hydrochloric acid, concentrated (HCl): ACS reagent grade, Fisher #A508-212, or equivalent. Hydrochloric acid (1:1 HCl) - Add 500mL concentrated HCl to 400mL DI water and dilute to 1L. Store in or under fume hoods. Prior to use, the acid is analyzed to determine levels of impurity. If the method blank is less than the RL, the acid can be used. Results of this analysis are kept in a logbook.

8.3 Nitric acid, concentrated (HNO_3): ACS reagent grade, Fisher #A509-212, or equivalent. Nitric Acid (1:1 HNO_3) - Add 500mL concentrated HNO_3 to 400mL DI water and dilute to 1L. Prepare 1% Nitric Acid by diluting 1mL of concentrated HNO_3 up to 100mL in DI water. Store in or under fume hoods. Prior to use, the acid is analyzed to determine levels of impurity. If the method blank is less than the RL, the acid can be used. Results of this analysis are kept in a logbook

8.4 Spiking solutions:

All standard solutions are stored in cabinets out of direct light.

8.4.1 Laboratory Control Sample (LCS) and Matrix Spiking (MS) solution for 3050:2T: Spike 1mL of custom spike from Ultra Scientific into the LCS and MS samples. The final concentration in the LCS and MS samples are Sn at 1.0 mg/L; Be and Cd at 2.0mg/L; Sb, Ba, B, Cr, Cu, Co, Mn, Zn, Ni, Pb, V, Tl, As, Se and Mo at 4.0mg/L; Al, Ca, Fe, Mg, K, and Na at 20.0mg/L.

8.4.2 Laboratory Control Sample (LCS) and Matrix Spiking (MS) solution for 3050:1T: Spike 0.8mL of S3 into the LCS and MS samples.

8.4.2.1 S3 Solution: Prepare by adding 5mL of concentrated HNO_3 to a 100mL volumetric flask using a 5mL glass pipette. Then add 2.5mL of IQC-026 (Ultra Scientific) Elements solution containing Al, Sb, As, Se, Ba, Be, B, Cd, Cr, Ca, Cu, Co, Fe, Pb, Mg, Mn, Mo, Ni, Ag, Na, Tl, Ti and Zn at a concentration of 100ppm, K at 1000ppm, and Si at 50ppm. This solution is brought to volume with DI water.

8.4.3 LCS and MS spiking for additional targets not in spike mixes for 3050:2T:

Additional target of Ag may be added to the LCS and MS. Add 0.5 mL of a 100 mg/L stock solution to the LCS and MS resulting in a concentration of 1 mg/L (final volume, as noted in Section 10, is 50 mL).

8.5 LCS Solid Matrix Sample: Catalog # 540 from ERA.

8.6 30% Hydrogen Peroxide (H_2O_2): ACS reagent grade, BDH #BDH3742-1, or equivalent. Stored under the hood, kept in the plastic bag it comes in and placed in a "spill" dish pan. Expires according to the manufacturer's date, or 18 months from date received, whichever comes first.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A method blank must be prepared once per every 20 samples or per digestion batch, whichever is more frequent.

Metals elements of interest must not be detectable in the method blank at a concentration greater than the reporting limit.

If required, and a blank solid material of a similar matrix type of high enough purity can be obtained to meet required reporting limits, a solid material may be used in the Method Blank preparation to matrix match QC samples and field samples.

Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. Digestion of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Metals Section Head, Laboratory Director or QAO, if the samples associated with the out of control method blank are non-detect for the elements of interest, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)

Laboratory control sample (LCS) must be prepared once per every 20 samples or per digestion batch, whichever is more frequent, and a solid LCS may be prepared to matrix match QC samples and field samples.

If required, an LCS may be prepared by spiking with a solution prepared with a second source.

The acceptable recovery QC limits are documented in the applicable analytical SOPs. The solid recovery limits are continuously monitored and documented in-house through control charts. The

Control Limit Generation SOP (1734) provides details explaining how control charts are generated and used for quality control.

Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-prepare and re-analyze the LCS and all associated samples. Samples cannot be reported until an acceptable LCS is obtained. Exceptions may be made with approval of the Metals Section Head, Laboratory Director or QAO, if the samples associated with the out of control LCS are also associated with a matrix spike that is in control. This is an acceptable measure of accuracy of the digestion and analytical procedures. An explanation of the out of control LCS recovery must be included in the project narrative to the client and the sample data reported noting the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

Not applicable to this method.

9.4 Continuing Calibration Verification (CCV)

Not applicable to this method.

9.5 Matrix Spike

A matrix spike (MS) sample must be performed once per 20 samples (5% frequency), or per digestion batch, whichever is more frequent. The MS contains all target elements of interest.

The acceptable % recovery QC limits are documented in the applicable analytical SOPs. The aqueous % recovery QC limits are continuously monitored and documented in-house through control charts. The Control Limit Generation SOP (1734) provides details explaining how control charts are generated and used for quality control.

Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits and the LCS is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy of the reported results as evidenced by MS recovery outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses (matrix or sample duplicate) must be performed once per 20 samples 5% frequency), or per digestion batch, whichever is more frequent.

Acceptable relative percent differences (RPD) of duplicates are documented in the applicable analytical SOPs. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = sample Replicate #1
R2 = sample Replicate #2

The RPD limits are continuously monitored and documented in-house through control charts. The Control Limit Generation SOP (1734) provides details explaining how control charts are generated and used for quality control.

Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported metals results as evidenced by the matrix duplicate %RPD exceedance.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

Refer to Analytical SOP 2137 for method sequence.

10. Procedure

10.1 Equipment Set-up

Samples are prioritized by the Metals Section Head or Preparation Group Leader for digestion based on hold time and client due date. Gather all samples for digestion from the Sample Custodian according to the procedures outlined in the Sample Receipt & Log-In SOP (1559). Batch the samples that are being digested in the LIMS. Include the method blank, LCS, MS and Duplicate samples. **Note**: The proportional volume of reagents used for the digestions have been reduced from the volumes cited in the reference methods.

10.2 Initial Calibration

Not applicable to this method.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Digestion according to Lab Method 3050:1T:

10.3.1.1 Turn on the electric hotplates and monitor the temperature to 90-100°C. The hot blocks take approximately 120 minutes to reach the proper temperature. Using a plastic spoon, homogenize sample and weigh 1-2g of sample (to achieve a 1g dry weight) into a digestion tube. Label each tube with the sample batch ID.

10.3.1.2 Add 7.5 mL of concentrated hydrochloric acid and 2.5 mL concentrated nitric acid to each sample and spike the LCS and MS sample using the solutions prepared in Section 8.4.1.

10.3.1.3 Place the tubes into the block. Cover the samples with a watch cover. Heat the samples for a timed 75 minutes using caution not to let the samples go to dryness.

10.3.1.4 Remove the samples and allow them to cool. Remove the watch covers and bring the samples up to the 50mL mark on the digestion tube with DI water. Cap the tubes and shake. There will be precipitate formed during digestion, such as silicates or other insoluble material that may clog the nebulizer, these samples will require filtration. See Section 10.3.3 for filtration procedures.

10.3.2 Sample Digestion according to Lab Method 3050:2T:

10.3.2.1 Turn on the electric hotplates and monitor the temperature to 90-100°C. The hot blocks take approximately 120 minutes to reach the proper temperature. Weigh 1-2g or sample (to achieve a 1g dry weight) into a digestion tube. For analysis of an air sampling filter, cut a one inch by eight inch section of filter using high

carbon steel scissors and place into a digestion vial. Label each tube with the sample batch ID.

10.3.2.2 Spike the LCS and MS sample using the solutions prepared in Section 8.4.2.

10.3.2.3 Add 5mL of 1:1 nitric acid to each sample and place the tubes into the block. Cover the samples with a watch cover. Heat the samples for a timed 15 minutes using caution not to let the samples go to dryness.

10.3.2.4 Remove the samples from the block. When cool, add 2.5mL of concentrated nitric acid to each sample. Cover the samples with a watch cover, and place them back into the block. Reflux the samples for 30 minutes, remove the sample and repeat by adding 2.5mL of concentrated nitric acid to each sample and heating for an additional 30 minutes. Continue and repeat adding nitric acid until NO brown fumes are given off by the sample indicating the complete reaction with the nitric acid.

10.3.2.5 Remove the sample, when cool SLOWLY add 0.5mL of 30% hydrogen peroxide and replace the watch cover, placing sample back into the block. Heat sample until effervescence subsides, then remove, and cool the sample. Once cool, add 1.0mL of 30% hydrogen peroxide, replace the watch cover, and heat in the block until the effervescence subsides. When the sample is removed from the block and cooled, 1.0mL of 30% hydrogen peroxide should be added 2 more times and the sample should effervesce and cool between each 1.0mL aliquot addition. This step may take up to 2 hours to complete.

10.3.2.6 Remove samples and allow them to cool, add 2.5 mL HCl and 5 mL H_2O to the sample digest and heat the sample to 95°C ± 5C, Reflux at 95°C ± 5C without boiling for 5 minutes. Then remove watch covers and bring the samples up to the 50mL mark on the digestion tube with DI water. Cap the tubes and shake. There will be precipitate formed during digestion, such as silicates or other insoluble material that may clog the nebulizer, these samples will require filtration. See Section 10.3.3 for filtration procedures.

10.3.3 Sample filtration after digestion:

10.3.3.1 After digestion, all samples, including the method blank and LCS are filtered with a pre-cleaned Filter Mate and plunger.

10.3.3.2 If, during the filtration step above, the digestion tube inadvertently cracks, have a clean new tube near by. Quickly transfer the remaining sample digestate (minimum of 5mL) to the new tube. If less than 5mL of digestate remains, the sample must be re-digested. *Under no circumstances should any foreign object be placed into the sample tube to retrieve or re-set the filter plunger, as this may cause sample contamination.*

10.4 Continuing Calibration

Not applicable to this method.

10.5 Preventive Maintenance

The Hot Block thermometers are calibrated on an annual basis by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

Procedures for data and record management for organic extraction must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control*

SOP (1729). All records must be stored in such a manner as to be safe and accessible for at least 10 years.

The digestion batch bench sheets and other relevant laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* Work Instruction (1556), and all record keeping and document control practices.

See the appropriate analytical SOPs, for details on sample analysis, data evaluation, calculations and data reporting.
All results for the metals elements of interests are reportable without qualification if digestion and analytical holding times are met, preservation (including cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference is not suspected during digestion or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

QC Parameter	Acceptance Criteria
Method Blank	< reporting limit
Laboratory Control Sample	See the applicable analytical SOP for acceptance criteria
Matrix Duplicate	See the applicable analytical SOP for acceptance criteria
Matrix Spike	See the applicable analytical SOP for acceptance criteria
Matrix Spike Duplicate (if needed)	See the applicable analytical SOP for acceptance criteria

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 and the appropriate analytical SOPs outline sample batch QC acceptance criteria. If non-compliant inorganic element results are to be reported, the Metals Section Head and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The instrument analyst or Section Head performing the secondary analytical review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

The *Hazardous Waste and Sample Disposal* SOP (1797), must be referenced for disposal of used standards, solvents, acids, reagents or other chemicals.

Once sample batches have completed digestion, the sample containers are stored in the metals lab and held for 30 days. If there is no sample remaining in the sample collection bottle, it may be rinsed and thrown away. It must be noted in the *Internal COC* that there is no sample remaining in the bottle, and the bottle was discarded.

Once the samples have been held for 30 days, any aqueous sample remaining must be disposed in a 55-gallon drum labeled “Corrosive Liquid”.

Once satisfactory inorganic element results have been generated, the digestates are held for 30 days, or longer if specified by a client contract, then discarded into a 55-gallon drum labeled “Corrosive Liquid”.

All reagent waste generated during digestion must be stored in satellite containers in the metals preparation laboratory.

Once the reagent waste satellite containers are full, they must be emptied into 55-gallon drums marked “Corrosive Liquid”.

15. Referenced Documents

Chemical Hygiene Plan

Control Limit Generation SOP (1734)

Document Control SOP (1729)

Hazardous Waste and Sample Disposal SOP (1797)

SOP/1559 Sample Receipt & Log-In

SOP/1732 MDL Generation

SOP/1739 IDC Generation

16. Attachments

None.

Inductively Coupled Plasma - Mass Spectrometry

References: **Method 6020A,** Test Methods for Evaluating Solid Waste:
Physical/ChemicalMethods, EPA SW-846, Revision 1, February 2007.

1. Scope and Application

Matrices: Inductively coupled plasma-mass spectroscopy (ICP-MS) is applicable to the determination of multi-element trace metals in water and other aqueous samples (surface and saline waters, groundwaters, domestic and industrial wastes), toxicity characteristic leaching procedure (TCLP) extracts, acid-volatile sulfide – simultaneously extractable metals (AVS-SEM)) and solid samples (soils, sediments, sludges, and tissues).

Definitions: Refer to Alpha Analytical Quality Manual.

Inductively coupled plasma-mass spectroscopy (ICP-MS) determines trace metal concentrations in solution. The method is applicable to all of the metals and matrices listed below. This method is approved for use in compliance monitoring programs such as the Clean Water Act (NPDES). All matrices, with the exception of dissolved metals samples, require extraction and/or digestion prior to analysis. The metals listed in the tables of Section 16 can accurately be determined in the range of 0.2 µg/L to 50,000 µg/L for aqueous samples, and in the range of 0.02 to 5000 mg/Kg for solid samples, for samples that do not require dilution.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the ICPMS and in the interpretation of ICPMS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Parameter	CAS	Parameter	CAS	Parameter	CAS
Aluminum (Al)	7440-36-0	Copper (Cu)	7440-50-8	Strontium (Sr)	7440-24-6
Antimony (Sb)	7440-36-0	Iron (Fe)	7439-89-6	Silver (Ag)	7440-22-4
Arsenic (As)	7440-38-2	Lead (Pb)	7439-92-1	Sodium (Na)	7440-23-5
Barium (Ba)	7440-39-3	Magnesium (Mg)	7439-95-4	Thallium (Tl)	7440-28-0
Beryllium (Be)	7440-41-7	Manganese (Mn)	7439-96-5	Tin (Sn)	7440-31-5
Boron (B)	7440-42-8	Molybdenum (Mo)	7439-98-7	Titanium (Ti)	7440-32-6
Cadmium (Cd)	7440-43-9	Nickel (Ni)	7440-02-0	Vanadium (V)	7440-62-2
Calcium (Ca)	7440-70-2	Potassium (K)	9/7/7440	Zinc (Zn)	7440-66-6
Chromium (Cr)	7440-43-9	Selenium (Se)	7782-49-2		
Cobalt (Co)	7440-48-4				

2. Summary of Method

An aliquot of a well mixed, homogeneous aqueous or solid sample is accurately measured or weighed for sample preparation. Prior to analysis, all samples, with the exception of samples requiring dissolved metal analysis, must be “solubilized” or digested in acid using the appropriate

sample preparation methods as noted in above. Dissolved metals do not require acid digestion if the samples are filtered and acid preserved prior to analysis. Once the samples have been digested, they are ready for analysis by ICP-MS.

This analytical method entails the simultaneous multi-elemental determination of sub-µg/L concentrations of many trace metals by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Samples are nebulized and the resulting aerosol is transported to the plasma torch by argon gas. The ions produced by a radio-frequency inductively coupled plasma are then introduced into a quadrapole mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

2.1 Method Modifications from Reference

The calibration blank is used as a reference to monitor changes in internal standard recoveries in QC samples and client samples. This deviates from the reference method which suggests the use of the initial calibration standard. The instrument software can not be changed to perform this analysis. There is no apparent impact to the quality of the data as indicated by successful analysis of PT samples over the period of operation of the instrument.

3. Reporting Limits

Reporting Limits are listed in Table 1.

4. Interferences

4.1 Isobaric elemental interferences occur when an isotope of one element is at the same nominal mass-to-charge ratio (m/z) as an isotope of another element (*i.e.*, Mo 98 and Ru 98). Corrections for isobaric interferences may be made by measuring the intensity due to the interfering element at another isotope and using its natural abundance ratios to correct for its presence at the analytical mass of interest. Most commonly used corrections for isobaric interferences are already present as default interference equations in the ELAN NT software. A list of the corrections used is given in the listing of the isotopes monitored in the 6020A method in Table 3 of this SOP.

Care should be taken that the isotope measured for correction purposes does not suffer from overlap with other isotopes that may be present in the sample. Extreme caution should be exercised when reporting metal concentrations where the "apparent concentration" from an interfering element accounts for 90% of the measured concentration. This can be estimated by closely monitoring the concentrations of the non-spiked metal concentrations in the daily analysis of the ICSA solution.

4.2 Isobaric molecular and doubly-charged ion interferences are caused by ions consisting of more than one atom or charge. Common molecular interferences include ArCl, ClO, nitrogen dimer, oxygen dimer, and oxide species. Most isobaric interferences have been identified in the literature. Isobaric molecular interferences can often be corrected for in the same manner as isobaric elemental interferences, i.e., measuring the intensity present at another isotope and using isotope ratios to calculate the amount of the interfering species. For example, corrections for interferences of $Ar^{40}Cl^{35}$ on As at mass 75 may be made by measuring the intensity of ArCl present at mass 77 ($Ar^{40}Cl^{37}$) and converting to the apparent intensity of ArCl at mass 75 by using the isotopic ratio of Cl^{37} to Cl^{35}. A list of the

corrections used is given in the listing of the isotopes monitored in the ELAN 6020 method in Table 3 of this SOP.

It may be possible to eliminate or minimize isobaric interferences by using the DRCe ICP /MS equipped with a reaction cell. See Section 11.9.

Care should be taken that the isotope measured for correction purposes does not suffer from overlap with other isotopes that may be present in the sample. Extreme caution should be exercised when reporting metal concentrations where the "apparent concentration" from an interfering element accounts for 90% of the measured concentration. This can be estimated by closely monitoring the concentrations of the non-spiked metal concentrations in the daily analysis of the ICSA solution

4.3 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. Differences in solution volatility can also cause inaccuracies when organic solvents are involved. If physical interferences are present, they must be reduced by diluting the sample. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The argon flow rate is controlled with a mass flow controller. Changing the nebulizer and removing salt buildup at the tip of the torch sample injector is used as an additional measure to control salt buildup when there is an obvious decrease in instrument sensitivity. An internal standard can also be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When the intensity level of an internal standard is outside of the 70% - 120% range for Method 6020A then the sample must be reanalyzed at a 5 X dilution.

4.4 Memory interference results when analytes in a previous sample contribute to the signals measured in a new sample. Sample deposition and buildup on the sampler and skimmer cones can be minimized by flushing the system with rinse blanks between samples. A normal rinse time of 60 seconds can be increased if memory interference is suspected. Any sample suspected of having memory interference must be reanalyzed.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Eating, drinking, smoking, or the application of cosmetics is not permitted in the laboratory area. Horseplay of any kind is prohibited. Pipetting by mouth is not permitted. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and before entering the employee lounge or eating area. Always wash your hands before leaving the laboratory. All relevant Material Safety Data Sheets (MSDSs) are kept AlphaNet.

5.2 Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled under a hood using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting devise and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

5.3 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to chemicals must be reduced to the lowest possible level by whatever means available. All standards and reagents shall be prepared in a hood while using the proper PPE.

5.4 Spilled samples, solvents, reagents, and water must be cleaned up from bench tops, instruments and autosampler surfaces immediately. A spill is considered a quantity of hazardous material if it is two times greater than the normal working volume. Concentrated solvents, acids or bases present a moderate to extreme hazard to the skin and mucous membranes. If contact with the skin occurs, immediately flush with large volumes of water. In the case of acidic/basic spills, the *Spill Kit* located in each laboratory shall be utilized before attempting to cleanup the spill. Although procedures are designed to minimize the possibility of an accident, all injuries or accidents, regardless of the nature or severity, are to be reported to the Section Head Supervisor immediately. If an employee discovers a potentially unsafe condition, this must be reported to the Section Head Supervisor immediately. No employee should feel compelled to work in a situation where they do not feel entirely informed, trained, or safe.

5.5 Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintain an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

5.6 Compressed gas cylinders shall only be moved with the dolly supplied for this specific purpose. The cap must be on the cylinder while it is being moved. The tank must be secured when in its final position. All spent tanks are to be returned in the same manner, and secured until removed by the vendor. Liquid argon or nitrogen represents a potential cryogenic hazard and safe-handling procedures must be used at all times.

5.7 Care must be taken when handling all liquid samples, digestates, and standards since they are preserved to a pH <2.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous Samples for total metal analysis: The volume of sample required is dependent on the analyses requested. Typically, a 500 mL of sample is collected in plastic containers. Samples of this volume should be preserved with 0.5mL of concentrated nitric acid per liter of sample (pH ≤ 2) at time of collection.

Solid samples: A minimum of 10.0 grams of sample must be collected in a glass jar.

6.2 Sample Preservation

Aqueous Samples for total metal analysis: For the determination of total parameters, the sample is preserved with 1:1 HNO_3 to pH < 2, not to exceed 10 ml of 1:1 HNO_3 per liter of sample. Usually 2 ml of 1:1 HNO_3 is sufficient for the preservation of waters Samples must be pH <2 for at least 24 hours prior to digestion if not preserved at the time of collection.
If conditions do not permit field preservation, this must be noted on the COC and in the laboratory case narrative.

Aqueous samples for dissolved metal analysis: an aliquot of the unpreserved sample is filtered through a 0.45μm membrane filter within 24 hours of the collection time, and prior to sample digestion and analysis. The sample is filtered through a nitric acid presoaked glass filtration apparatus. Once a sufficient volume of the filtrate is obtained, the filtrate is preserved with 1:1 Nitric acid (HNO_3) to a pH < 2. Soluble samples must be held at pH < 2 for at least 24 hours prior to digestion if not preserved at the time of filtration. A separate SOP lists the sample preparation technique.

A plastic apparatus must be used when determination of boron or silica is critical.

Sample Shipping

None.

6.3 Sample Handling

Aqueous Samples for total metal analysis: The samples can be stored at room temperature or can be refrigerated and maintained at 4°±2°C until digestion and analysis. All aqueous samples must be analyzed within 6 months from date of collection

Solid samples: The samples must be refrigerated and maintained at 4°±2°C until digestion and analysis. All solid samples must be analyzed within 6 months from the date of collection. The hold time for samples that require AVS/SEM analysis is 14 days.

Note: Although mercury is not typically analyzed by ICP-MS, the hold time for samples that require mercury analysis is 28 days.

7. Equipment and Supplies

7.1 Perkin-Elmer ELAN 6100 or ELAN DRCe inductively coupled argon plasma – mass spectrometer (ICP-MS): Capable of providing resolution better than or equal to 1 amu at 10% peak height. The system has a mass range from 6-240 amu with a data system that allows for correction for interferences and the application of the internal standard technique.

7.1.1 ELAN 6100 and DRCe computer system

7.1.2 ELAN software

7.1.3 Printer
7.1.4 Mixing block manifold for on-line addition of internal standards
7.1.5 Autosampler
7.1.6 Peristaltic pump tubing

7.1.6.1 Sample Introduction; Black/Black - 0.76 mm (0.030") i.d

7.1.6.2 Internal Standard Introduction; Green/Orange - 0.38 mm (0.015") i.d.
7.1.6.3 Drain; Black/White – 3.18 mm (0.125") i.d

7.1.6.4 Rinse; Red/Red - 1.14 mm (0.045") i.d

7.1.6.5 Making Connections; Blue/Blue - 1.65 mm i.d

7.1.6.6 Making Connections; Purple/Purple -2.06 mm i.d

7.2 Liquid Argon - 99.999% purity and regulator.

7.3 Oxygen: High purity gas and regulator

7.4 Ammonia: High purity gas and regulator

7.5 Glassware - Assorted Class-A volumetric flasks, beakers, graduated cylinders and pipettes of appropriate sizes for preparing reagents, standards, and measuring sample volumes

7.6 Air Displacement pipetters: Eppendorf brand or equivalent digital pipettes capable of delivering volumes ranging from 0.1 to 5000 µL with an assortment of high quality disposable pipette tips

7.7 Autosampler tubes: 15-mL plastic AS-91 and/or S10

7.8 Digestion tubes: 50-mL plastic with caps

7.9 Analytical balance: Capable of accurate measurement to the nearest 0.01 g

8. Reagents and Standards

Deionized (DI) water is ASTM Type II laboratory reagent grade water or better (*i.e.,* Type I). The Barnstead NANO-pure system provides Type I water used in the preparation of samples and standards. ACS Trace Metal grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration is less than the RL then the reagent is acceptable.

Solutions below expire six months from preparation unless noted. All stock and working calibration standards expiration dates are based on manufacturer expiration date or one year from date received. All solutions are stored at room temperature

8.1 Nitric acid (HNO_3), concentrated. Trace metal grade or ultra-pure from sub-boiling distillation is preferred. Suggested suppliers include: Seastar ™- Sub-boiling distilled grade, Sidney, BC; J. T. Baker - ULTREX© Grade; and Fisher - Optima Grade. Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.2 Hydrochloric acid (HCL), concentrated. Trace metal grade or ultra-pure from sub-boiling distillation is preferred. Suggested suppliers include: Seastar™ - Sub-boiling distilled grade, Sidney, BC; J. T. Baker - ULTREX® Grade; and Fisher - Optima Grade. Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.3 2% (vol/vol) Nitric acid. Prepare by adding 40 mL of concentrated nitric acid to 2000 mL reagent water in a clean glass bottle or 180 mL of concentrated nitric acid to 9000 mL reagent water in a clean carboy. This acid minimizes the damage to the interface and also minimizes isobaric molecular-ion interferences.

8.4 10% (vol/vol) Nitric acid. Prepare by adding 200 mL of concentrated nitric acid to 2000 mL reagent water in a clean glass bottle or 900 mL of concentrated nitric acid to 9000 mL reagent water in a clean carboy. This acid is the maximum concentration used for calibration standards, samples and rinse acid as recommended by the instrument manufacturer.

8.5 0.1% (vol/vol) Sub-boiling distilled Nitric acid. Prepare by adding 1.0 mL of concentrated sub-boiling distilled nitric acid to 1000 mL reagent water in a clean polyethylene bottle.

8.6 5% (vol/vol) Nitric acid. Prepare by adding 50 ml of concentrated nitric acid to 950 mL reagent water in a clean polyethylene bottle.

8.7 Single element stock solutions for the following elements are typically maintained in the laboratory. These are normally purchased as 1000 mg/L standards from Fisher Scientific or equivalent. The ICP MS is capable of determining concentrations of 73 elements in the Periodic Table. Other calibration standards, not present in Table 4, may be purchased as necessary for non-routine analyses.

8.7.1 Arsenic

8.7.2 Beryllium

8.7.3 Selenium

8.7.4 Strontium

8.7.5 Tin

8.7.6 Zinc

8.8 Tuning Solution Stock standards consist of 1000 mg/L beryllium (Be), cobalt (Co), indium (In), thallium (Tl), and uranium (U).

8.8.1 100 mg/L Tuning Solution Intermediate Standard . Prepare 1:10 dilutions by pipetting 100 µL of each 1000 mg/L single element stock solutions of Co, In, Tl, and U into a 1 mL autosampler cup containing 900 µL 2% nitric acid.

8.8.2 Tuning solution: 150 µg/L Be and 10 µg/L Co, In, Tl, and U. Prepare by pipetting 100 µL of 100 mg/L tuning solution standard in 8.8.1 and 150 µL of 1000 mg/L Be into a 1 liter volumetric flask filled with 200 mL of reagent water and 20 mL of concentrated nitric acid. Dilute to a final volume of 1 liter with reagent water and mix well.

Can also order Perkin Elmer part # N812-2014 for the tuning solution.

8.9 The Internal Standard Solution is prepared from single element standards consisting of 100 mg/L Germanium (Ge), 100 mg/L indium (In), 100 mg/L lithium (Li^6), 100

mg/L scandium (Sc) and 100, mg/L terbium (Tb). Alternately, a custom mix of internal standards may be purchased from Spex.

8.9.1 Add 10 mL of concentrated nitric acid to a 500 ml volumetric flask containing 250 ml of DI water.

8.9.2 Add 5 ml of 100 mg/L Li^6. Li^6 is used routinely when analyzing beryllium and boron or other elements as needed. If these elements are not being analyzed then it is not necessary to add this internal standard.

8.9.3 Add 1.0 mL of a stock solution containing 100 mg/L of Sc.

8.9.4 Add 0.5 mL of 100 mg/L of In, Ge and Tb stock solutions

Note: All solutions including calibration blanks, calibration standards, samples, quality control standards, and quality control samples must be spiked with the same level of the internal standard spiking solution. Use the above solution in conjunction with the on-line mixing block for addition of internal standards to calibration standards and samples. Internal standard intensities should be between 100,000 cps and 150,000 cps. The internal standard solution as made here should be diluted 1:2 for the DRCe ICP MS instrument due to the greater sensitivity of the instrument. The internal standard mass should be no more than 50 AMU removed from an analyte.

Note: Other internal standards are suggested for use such as Y, Ho, Lu and Bi and may be used if poor performance is observed from the above standards

8.10 Calibration Stock Standard and Intermediate Working Standard Solutions

Intermediate Working Standard solutions are prepared from certified vendor Stock Standard solutions and include those analytes listed below. Vendor sources are subject to change and equivalent solutions may be used.

Mixed calibration standard solutions must contain the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates. The acid matrix for all standards is 0.1% HNO_3 or 5%. These concentrations do not damage to the ICP-MS interface and also minimizes isobaric molecular-ion interferences with the analytes. A diluent of the acid matrix preparation is described in 8.3 and 8.4. *Note: Silver and antimony may precipitate out of solution at higher concentrations between 50-500 µg/L. Higher silver concentrations (>500 µg/L) require additional HCl. Caution: Many metal salts are extremely toxic if inhaled or swallowed. The Health and Safety precautions noted in Section 5 must be followed.* All standards are stored in the metals instrument laboratory at room temperature. The expiration dates are monitored from vendor supplied dates. The element or mix of elements that expires first in a solution dictates the expiration date for the entire solution. Standard and spike solutions are labeled using an alpha-numeric system based on the date prepared. All standards or spikes that are prepared are recorded in the metals laboratory logbooks. It is suggested that standards with a final concentration of less than 1 mg/L be prepared daily

8.10.1 **CPI-19:** *Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Se, Tl, Ti, V and Zn* from CPI containing all of the listed elements at 100 mg/L.

8.10.1.1 (**Date)-C1:** Intermediate working standard is prepared by adding 1 mL of CPI-19 (8.10.1) to 9 ml of 0.1% nitric acid. This standard

is prepared approximately every four weeks and is used to prepare the daily calibration standards

8.10.2 **CPI-7:** *K* at 1000 mg/L, Si at 50 µg/ml, and *Al, Ba, B, Ag and Na* at 100 mg/L from CPI at the concentrations noted.

8.10.2.1 (**Date)-C2:** Intermediate working standard is prepared by adding 1 mL of CPI-7 (8.10.2) to 9 ml of 0.1% Acid (8.5). This standard is prepared approximately every four weeks and is used to prepare daily calibration standards.

8.10.3 **AT-3:** *Al, Ca, Fe, Mg, K, and Na* a custom mix from Inorganic Ventures at 500 mg/L for each element.

8.10.3.1 **Date)-C3**: Intermediate working standard is prepared by adding 1 mL of AT-3 (8.10.3) to 9 ml of 0.1% Acid (8.5). This standard is prepared approximately every four weeks and is used to prepare daily calibration standards.

8.10.3.2 **(Date)-C4:** This standard is prepared by pouring AT-3 (8.10.3) into a satellite container for use in preparing daily calibrations standards.

8.10.4 **IQC-026:** *Sb, Ag, Al, As, B, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Na, Ni, Se, Tl, Ti, V and Zn* at 100 mg/L, *Si* at 50 mg/L and *K* at 1000 mg/L from Ultra Scientific.

8.10.4.1 **(Date)-I1:** Intermediate working standard is prepared by adding 1 mL of IQC-026 (8.10.4) to 9 ml of 0.1% Acid (8.5). This standard is prepared approximately every four weeks and is used to prepare daily calibration standards.

8.10.5 **XAQU-15**: *Fe, Al, Mg, Na, Ca and K* a custom mix from SPEX at 500 mg/L for each element listed.

8.10.5.1 (**Date)-I2:** Intermediate working standard is prepared by pouring XAQU-15 (8.10.5) into a satellite container for use in preparing daily calibrations standards

8.10.6 **Single Element Added Analytes:** Sn, and other analytes may be added to the calibration standards, ICV, ICSAB, and LLCV. Two intermediate solutions from two separate sources are prepared. 0.5 ml of each 1000 mg/L stock solution is added to 50 mLs of 10% HNO3 to a concentration of 10 mg/L, one is labelled Calibration intermediate and the second source is labelled ICV intermediate.

8.10.7 **ICUS-3411**: RL check standard stock solution, a custom mix from ULTRA. Ag, Cd, Co, Pb, Tl, at 1 mg/L, Be at 1.5 mg/L, As, Ba, Cu, Cr, Mn, Mo, Ni, Sb, Sr, Ti at 2.5 mg/L, Se, V at 5 mg/L, B, Zn at 25 mg/L, Al, Fe, Mg at 50 mg/L, Ca, K, Na, at 500 mg/L

8.11 Daily Calibration Standards

For USFSER work, an Initial Calibration Curve is prepared for each level and is described below. Every element is *not* in each level of the calibration curve. At minimum, a high level, a low level that confirms the reporting limit (STD1), and a calibration blank (STD0), is used. See Table 4 for a list of the initial calibration concentrations. Standards are prepared in either 0.1% or 5% nitric acid to matrix match standards to samples depending on the how samples and sample digests are diluted for analysis. Standards used to analyze undiluted Dissolved Aqueous samples are prepared in 0.1% nitric acid. Standards used to analyze undiluted Total Aqueous samples prepared by hot plate (2151) are prepared in 5% nitric acid. Soil/Sediment samples prepared by Hot Plate Method 3050 (2148) and.Tissue

samples prepared by microwave oven (2150) and diluted 1:2 are prepared in 5% nitric acid. Standards used to analyze Total Aqueous samples prepared by hot plate (2151), diluted 1:5, and Soil/Sediment samples prepared by Hot Plate Method 3050 (2148), diluted 1:5, are prepared in 5% nitric acid.

8.11.1 **STD0 (Calibration Blank):** Calibration Blank: A solution containing the same acid matrix as samples and standards

8.11.2 **STD1 (Reporting Limit Standard):** Prepared from the RL check standard stock solution(8.10.7). Dilute stock solution to make RL intermediate(add 0.5 mL to 50 mL final volume in 2% HNO3). Add 1 mL of the RL intermediate to 50 ml digestion tube and bring to volume with diluent acid**.** Ag, Cd, Co, Pb, Tl, at 0.2ug/L, Be at 0.3ug/L, As, Ba, Cu, Cr, Mn, Mo, Ni, Sb, Sr, Ti at 0.5 ug/L, Se, V at 1 ug/L, B, Zn at 5 ug/L, Al, Fe, Mg at 10 ug/L, Ca, K, Na, at 100 ug/L. Sn is added as a single element additional target using the Calibration Intermediate (Section 8.10.6). Calibration intermediate is serially diluted, 0.5 mLs added to 50 mLs of 10% HNO3 resulting in a 100µg/L concentration. Add 0.25 mLs of the subsequent dilution and bring to 50 mL final volume with diluent acid**.** Concentrations are at or below the reporting limit.

8.11.3 **STD2:** Add 50 µL of (Date)-C1 (8.10.1.1) and 100 µL of (Date)-C3 (8.10.3.1) to a 50mL digestion tube and bring to 50mL with the diluent acid. The final concentration of each element is: *As, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se , Ti, Tl, V and Zn* at 10 µg/L; *Al, Ca, Fe, and Mg* at 110 µg/L; *K and Na* at 100 µg/L; and *Si* at 5 µg/L. Note: only 16 of the 19 elements in this mix are evaluated at this concentration level. Add 50 µL of Calibration intermediate (8.10.6) (single element additional targets, e.g.,Sn) for a concentration of 10 µg/L.

8.11.4 **STD3:** Add 100 µL of (Date)-C1 (8.10.1.1) and 100 µL of (Date)-C4 (8.10.3.2) to a 50mL digestion tube and bring to 50mL with the diluent acid. The final concentration of each element is: *As, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Ti, Tl, V and Zn* at 20 µg/L; *Al, Ca, Fe, and Mg* at 1020 µg/L; *K and Na* at 1000 µg/L; *Hg* at 5 µg/L, and *Si* at 10 µg/L. Note: only 16 of the 19 elements in this mix are evaluated at this concentration level. Add 100 µL of Calibration intermediate (8.10.6) (single element additional targets, e.g.,Sn) for a concentration of 20 µg/L.

8.11.5 **STD4:** Add 500 µL of (Date)-C1 (8.10.1.1) and 1000 µL of (Date)-C4 (8.10.3.2) to a 50mL digestion tube and bring to 50mL with the diluent acid. The final concentration of each element is: *As, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Ti, Tl, V and Zn* at 100 µg/L; *Al, Ca, Fe, and Mg* at 10100 µg/L; *K and Na* at 10000 µg/L. Note: only 16 of the 19 elements in this mix are evaluated at this concentration level. Add 500 µL of Calibration intermediate (8.10.6) (single element additional targets, e.g.,Sn) for a concentration of 100 µg/L.

8.11.6 **STD5:** Add 500 µL of (Date)-C2 (8.10.2.1) to a 50mL digestion tube and bring to 50mL with the diluent acid. The final concentration of each element is: *Ag, B and Ba* at 100 µg/L. Note: These three metals are only performed with a 3 point calibration rather (including the blank).

8.11.7 For non-USFSER and non-DOD work, a single point calibration is performed. Concentrations up to the linear range may be reported without qualification.

8.12 Initial calibration verification (ICV) and continuing calibration verification (CCV)

The ICV and CCV standards are prepared from sources separate from the initial calibration curve and are used to verify the initial calibration curve throughout the analytical sequence. See Table 4 for a list of the ICV/CCV concentrations. To prepare this solution add 0.25mL of (Date)-I1 (8.10.4.1) and 0.5mL of (Date)-I2 (8.10.5.1) to a 50mL digestion tube and bring to 50mL with the diluent acid. The final concentration of each element is: *As, Ag, Sb, B, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Si, Ti, Tl, V and Zn* at 50 µg/L; *Al, Ca, Fe, and Mg* at 5050 µg/L; *K* at 5500 µg/L; and *Si* at 25 µg/L. Add 250 µL of ICV intermediate (8.10.6) (single element additional targets, e.g.,Sn) for a concentration of 50 µg/L.

8.13 Calibration blank (STD0), initial and continuing calibration blanks (ICB and CCB) are the analysis of the matrix diluent and are analyzed prior to the initial calibration curve and after the ICV and CCVs, respectively.

8.14 Reporting Limit check standard

Used to verify the reporting limit.

8.15 Interference Check Solutions – ICS

Two Interference Check Solutions (ICSA and ICSAB) are prepared to contain known concentrations of interfering elements and analytes that will provide an adequate test of correction factors. Concentrations of the interfering analytes correspond to concentrations in Table 1 of USEPA, "Method 6020A - Inductively Coupled Plasma-Mass Spectrometry," in Test Methods for Evaluating Solid Waste, SW-846, EPA SW-846, Revision 1, February 2007.
Dilution can reduce isobaric and physical interferences as well as instrument exposure to strong acids used in sample preparation.

Digests of water samples may be analyzed without dilution, at 1:5 dilution or higher to bring analyte concentrations within the linear dynamic range of the instrument. Digests of soil/sediment samples and tissue samples must be diluted 1:2 in DI water, at a minimum, to reduce the high acid concentration used in the preparation. The instrument manufacturer suggests a maximum acid concentration of 10% be used during analysis

Three solutions are used to prepare the interference check solutions ICSA and ICSAB.

8.15.1 6020ICS-9A: Supplied by Inorganic Ventures with concentrations of 20,000 ppm Cl^-; 3000 ppm Ca; 2500 ppm Fe and Na;1000 ppm Al, Mg, P, K, and S; 2000 ppm C; 20 ppm Mo and Ti

8.15.2 (**Date)-C1:** See 8.10.1.1

8.15.3 (Date)-C2: See 8.10.2.1

8.15.4 The ICSA Solution is prepared in a 50 ml screw cap digestion tube by adding 5 ml of 6020ICS-9A (8.15.1) to 0.1% nitric acid or 5% nitric depending on the acid being used for analysis (see section 8). This solution can be used for multiple analyses and can be stored until the expiration date of the standard.

8.15.5 The ICSAB Solution is prepared as a 50 ml aliquot in acid used for analysis according to the following table and must be prepared daily as some analytes are

not stable in solution. The final concentrations of analytes in (Date)-C1 are 100 μg/L and 50μg/L for (Date)-C2 with the exception of *Mo* and *Ti* which are present as interferents. Add 500 µL of Calibration intermediate (8.10.6) (single element additional targets, e.g.,Sn) for a concentration of 100 µg/L.

Note: Method 6020A suggests a concentration of 200 μg/L for Cr, Co, Cu, Mn, Ni and V. The reduced concentrations prepared for this standard represent a more rigorous test of isobaric corrections.

Solution	mLs added (final volume 50 mLs)
6020ICS-9A (8.4.1)	5
(Date)-C1	0.5
(Date)-C2	0.25
Tin calibration intermediate	0.5

8.16 Laboratory Control Sample (LCS) or Laboratory Fortified Blank (LFB) for aqueous samples is prepared from a source other than the calibration standard sources. It is prepared and stored for use in the metals preparation laboratory. The LCS for solid samples is a solid LCS purchased from ERA at known and certified values. See the metals preparation SOPs for LCS preparation details. See table 6 for spike levels.

8.17 Matrix Spike (MS) solution for aqueous and solid samples is the same solution as the aqueous LCS/LFB in 8.16. It is prepared and stored for use in the metals preparation laboratory. See the metals preparation SOPs for MS preparation details. See table 6 for spike levels.

8.18 Daily Performance Standard: Prepare by adding 0.1 ml of a 1:10 dilution of 1000 mg/L stock Ba, Ce, Mg, Pb, In, U and Rh standards to 1 L volumetric flask containing 100 ml of Di water with 20 ml of concentrated HNO_3. Mix and bring to volume with DI water.

8.19 Dual Detector Calibration Solution: Add 0.15 ml of CPI-19 stock (or equivalent), 0.15 ml of CPI-7 stock (or equivalent), 0.15 ml of 500 ml/L Al, Ca, Fe, K, Mg and Na stock and 0.1 ml each of 1000 mg/L As, Be, and Zn and 0.2 ml of 1000 mg/L Se stock to 100 ml volumetric flask containing 2% HNO_3. Bring to volume with 2% HNO_3. This solution is required for optimization of the pulse and analog state detectors. Other analytes may be added to this solution as needed.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

9.1.1 A method blank must be analyzed once per every 20 samples or per metals digestion batch, whichever is more frequent.

9.1.2 Metal concentrations must not be detectable in the method blank at values greater than the reporting limit.

9.1.3 Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. Digestion of the method blank and all associated samples must be performed until the method blank for the out of control metal is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Section Head if the samples associated with an out of control method blank are below the reporting limit for the affected metal or if the concentrations of the affected metal are greater than 10x the blank level in the samples. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified in the project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS) / Laboratory Fortified Blank (LFB)

9.2.1 The LCS/LFB is from a second source to verify the accuracy of the digestion and analytical procedures. The LCS/LFB is digested along with the samples. An LCS/LFB must be digested and analyzed once per every 20 samples or per metals digestion batch, whichever is more frequent.

9.2.2 The acceptable recovery QC limits are 80%-120% (Method 6020A) for an aqueous LCS/LFB and within manufacturer's control limits for the solid SRM LCS.

9.2.3 Corrective Action: May repeat analysis once to see if an analytical error has occurred. If the LCS/LFB recovery is still out of control, re-digest and re-analyze the LCS/LFB and all associated samples. Samples cannot be analyzed until an acceptable LCS/LFB is obtained. Exceptions may be made with approval of the Section Head if the samples associated with the out of control LCS/LFB are also associated with a matrix spike that is in control. This is an acceptable measure of accuracy of the digestion and analytical procedures. An explanation of this out of control LCS recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

9.3.1 Initial Calibration

9.3.1.1 A three-point calibration curve must be performed if the CRI standard is not analyzed.

9.3.1.2 The correlation coefficient must be r $\geq$0.998.

9.3.1.3 Corrective Action: Re-calibrate until criteria are met.

9.3.2 Initial Calibration Verification (ICV) Check Standard

9.3.2.1 The initial calibration verification check standard is from a second source to verify the accuracy of the standard curve. The concentration of the ICV is at approximately the mid-level of the calibration curve.

9.3.2.2 The acceptable recovery QC limits for the ICV is 90-110%. In addition, the relative standard deviation between three replicate readings must be less than 5% RSD.

9.3.2.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the ICV still exceeds the control limits, re-calibrate the instrument.

9.3.3 Initial Calibration Blank (ICB)

9.3.3.1 An ICB must be analyzed immediately following the ICV.

9.3.3.2 The acceptance limit for the ICB is < RL

9.3.3.3 Corrective Action: May repeat analysis once to determine if analytical error has occurred. Results may be reported from the analysis if the ICB concentration is less than the reporting limit. If the ICB still exceeds the reporting limits, re-calibrate the instrument and re-analyze a fresh blank.

9.4 Continuing Calibration Verification (CCV)

9.4.1 Continuing Calibration Verification (CCV) Check Standard

9.4.1.1 A CCV must be analyzed at a minimum of every 10 samples and at the close of an analytical sequence. The concentration of the CCV is at approximately the mid-level of the calibration curve. This standard monitors instrument performance throughout the duration of the analytical sequence.

9.4.1.2 The acceptable recovery QC limits for the CCV is 90-110%. In addition, the relative standard deviation between three replicate readings must be less than 5% RSD.

9.4.1.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CCV still exceeds the control limits, re-calibrate and re-analyze all samples since the last acceptable CCV.

9.4.2 Continuing Calibration Blank (CCB)

9.4.2.1 A CCB must be analyzed immediately after every CCV

9.4.2.2 The acceptance limit for the CCB is <RL.

9.4.2.3 Corrective Action: Results may be reported from the analysis if the CCB concentration is less than the reporting limit. If not, repeat analysis once to see if an analytical error occurred. If the CCB still exceeds the reporting limit, re-calibrate the instrument and re-analyze a fresh blank. All samples associated with the out of control metals in the CCB must be re-analyzed (since the last acceptable CCB). Exceptions may be made with approval of the Section Head if the samples associated with the out of control method blank are non-detect for the affected metals or if sample concentrations for the affected metals are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high CCB and the client is notified in a project narrative associated with the sample results.

9.5 Matrix Spike

9.5.1 A matrix spike must be performed once per 20 samples (5% frequency) and is from a second source to verify the accuracy of the digestion and analytical procedures. The matrix spike recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{MS_{recovery} - R1}{MS_{true\ value}} \times 100$$

When project specifications dictate, a Matrix Spike Duplicate (MSD) may also need to be performed at the same frequency as the MS.

9.5.2 The acceptable recovery QC limits for a MS/MSD is 75%-125% for both the solid and aqueous MS/MSD pair. Calculate the %RPD as in 12.3.2 above when analyzing a MS/MSD pair. The acceptable %RPD is ≤ 20%.

9.5.3 Corrective Action: May repeat analysis once to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the control limits and the LCS is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the affected metals results as evidenced by matrix spike recovery or %RPD outside of QC limits.

9.6 Laboratory Duplicate

9.6.1 Duplicate analyses (matrix duplicate) must be performed once per 20 samples (5% frequency).

9.6.2 Acceptable relative percent difference (RPD) for duplicate analysis is ≤ 20 % for both aqueous and solid matrices. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

9.6.3 Corrective Action: May repeat analysis once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the affected metals results as evidenced by the matrix duplicate RPD exceedence

9.7 Method-specific Quality Control Samples

9.7.1 Tuning Solution, Mass Calibration, and Resolution Checks

9.7.1.1 The tuning solution, mass calibration, and resolution checks are required prior to sample analysis. The tuning solution must be analyzed 4 times prior to instrument set up at the beginning of the day.

9.7.1.2 The analytes in the tuning solution must meet ≤5 percent relative standard deviation. The mass calibration must not differ by more than 0.1 amu from the true value. The resolution must be less than 0.9 amu full width at 10 percent peak height.

9.7.1.3 Corrective Action: Adjust the mass calibration to the correct value or correct the problem until criteria are met.

9.7.2 LLCV (RL) Check Sample (Reporting Limit Standard)

This solution is used to verify instrument sensitivity at the reporting limit at the beginning and end of the sequence. The concentration of the LLCV is equivalent to the reporting limit for each element in STD1, see Table 1. See Section 8.11.2 for preparation details.

9.7.2.1 Results for all metals in the LLCV solution must be within the 70-130% recovery criteria. When performing work for the Department of Defense (DoD, Army Corps of Engineers or the US Navy) the CRI acceptable recovery criteria is 80-120%.

9.7.3 Corrective Action: If the LLCV solution exceeds the control limits, sample results which are sufficiently high (10X the RL) can be qualified. Samples that are < 10X the RL are re-analyzed.

9.7.4 Interference Check Samples (ICSA and ICSAB) Solutions

9.7.4.1 The ICSA and ICSAB solutions must be analyzed at the beginning of an analytical run or every 12-hour work shift, whichever is more frequent. These solutions verify the isobaric corrections.

9.7.4.2 Results for all spiked non-interference metals in the ICSAB solution must be within 80-120% recovery criteria. It may be necessary to correct concentrations measured in the ICSAB solution with background concentrations measured in the ICSA solution. Results for aluminum, calcium, iron, sodium, potassium, and magnesium in the ICSA solution should be within 80-120% recovery, however, depending on the sample dilution used, concentrations of the these elements may be close to or exceed the linear range of the instrument which can cause the channel to saturate.

9.7.4.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the ICSA or ICSAB solutions still exceed the control limits, re-calibrate and/or re-analyze a fresh ICSA or ICSAB. If the sample results are accepted without corrective action for the ICSA or ICSAB exceedence(s), the client must be notified in a project narrative associated with the sample results.

Note: If an interference is suspected, use of computerized compensation or comparison with an alternate method such as graphite furnace is recommended.

9.7.5 Internal Standard Recovery

9.7.5.1 Internal standard intensities must be monitored in all solutions.

9.7.5.2 Intensities of internal standards in all subsequent analyses of instrument QC samples (including ICV, ICB, CCV and CCB) solutions must be within 70% -120% of the levels in the original calibration blank. Intensities of internal standards in samples must be within the same limits as the QC samples.

9.7.5.3 Corrective Action: If the instrument QC sample does not meet criteria, may reanalyze once to determine if analytical error has occurred. If QC sample still does not meet criteria, terminate the analysis for analytes associated with the internal standard, correct the problem, re-calibrate, and reanalyze all affected samples since the last in-control CCV/CCB. If a sample does not meet criteria, may reanalyze the sample and/or dilute the sample five-fold and reanalyze. This procedure is followed for the sample until the internal standard intensities fall within the prescribed window.

9.7.6 Serial Dilution Analysis

9.7.6.1 Serial dilution analysis must be performed once per 20 samples (5% frequency).

9.7.6.2 Analysis of a 1:5 dilution must agree within 10% of the original determination, if the metal concentration is sufficiently high (minimally, a factor of 50 above the method detection limit) in the original undiluted analysis.

9.7.6.3 Corrective Action: Analysis may be repeated once to see if an analytical error has occurred. If the %D still exceeds the control limits; include a project narrative with the results to client noting that there may be

potential matrix effects on the accuracy of the affected metals results as evidenced by the serial dilution %D exceedance.

9.7.7 Post Digestion Spike (PDS)

9.7.7.1 Post digestion spike analysis may be performed if the MS or MSD does not meet recovery criteria. A known spike amount is added to a portion of a prepared sample, or its dilution. The spike addition should produce a minimum level of 10 times, and a maximum of 100 times, the reporting limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

9.7.7.2 The post digestion spike recovery is 80%-120% of the known value.

9.7.7.3 Corrective Action: If the post digestion spike exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy of the affected metals results as evidenced by the post digestion spike exceedance.

9.8 Method Sequence

Initial calibration curve
ICV
ICB
LLCV
ICSA
ICSAB
Blank
Method Blank
LCS
Sample analysis
CCV
CCB
Sample analysis
CCV
CCB
Sample analysis
LLCV
CCV
CCB

10. Procedure

10.1 Equipment Set-up

10.1.1 Prior to using the Perkin-Elmer ELAN 6100 and DRCe ICP/MS, the operator must read and become familiar with the operating procedure guidelines specified in the operating manual. The analyst must be trained and familiarized with the instrument software provided by the manufacturer. The instrument must be set up with the proper operating parameters and conditions described in the Perkin-Elmer ELAN 6100 and DRCe ICP/MS operating manual. The criteria for the background correction points, analytical dynamic ranges, method and instrument detection limits and isobaric molecular-ion correction equations must be established and documented prior to initial calibration.

10.1.2 The plasma torch must be aligned before ignition and is done with an alignment tool supplied with the instrument. The procedure centers the torch within the load coil and

adjusts the distance of the torch to the sampler cone surface. This is outlined in the Elan 6100 Hardware Guide. This is not required on a routine basis, only initially upon instrument set up and following any changes, such as removal and cleaning of the plasma torch. The instrument must become thermally stable (usually 30-60 minutes) before beginning operation.

10.1.3 Isobaric corrections must be put into the equations page of any method used to generate quantitative analytical data. The correction equations can be found in Table 3. These corrections are based on natural isotopic abundance's and cannot be altered.

10.1.4 A linear dynamic range (LDR) study must be conducted every six months. The LDR is determined by analyzing increasingly higher standard concentrations of each element until the observed concentrations are no more than ± 10% of the true value of the standard. Sample analyte concentrations that are above the linear dynamic range must be diluted and re-analyzed. The linear dynamic range must be established for each analytical run by analysis of a high level standard or Linear Range Verification (LRV) standard. Results greater than the daily calibration range but within the linear calibration can be reported provided that analyte recovery from the LRV is within 10% of the true value. If the recovery from the LRV is outside of the 10% acceptance limit then samples may be diluted below the concentration of the highest calibration standard or reanalyzed. The linear dynamic range must be checked and verified every six months. See Table 6 for the current linear range.

10.2 Initial Calibration

10.2.1 Prior to daily calibration, inspect the sample introduction system including the nebulizer, torch, injector tube and uptake tubing for salt deposits, dirt or debris that could constrict flow and affect instrument performance. Clean the system when needed following the manufacturer's instructions.

10.2.2 A series of calibration standards are prepared for the initial calibration curve as described in Section 8.0. The preparation date of these standards, the initials of the analyst, the lot number of the source material, stock concentrations, volumes used, final volumes, final concentrations and manufacturer must be recorded in the metals standard preparation logbook. All standards are traceable to NIST via internal and external calibration checks. *Certificates of Analysis* accompany the receipt of standard solutions and are kept on file in the laboratory. The Perkin-Elmer ELAN 6100 and DRCe are calibrated using a multi-point calibration curve consisting of a blank and a two to three standards per analyte. The standards are named, for example, "C" for calibration, plus "Date" for the date prepared, followed by the letter M and a number 1, 2, 3 etc. for first, second or third set of standards prepared that day. "I" is used to determine the ICV standard. Standard "I040402M1" is the first ICV standard for the ICP MS made on 04/04/02. Standard "I040202M2" is the second ICV standard for the ICP MS made on 04/02/02. This nomenclature is for traceability and to distinguish calibration standards from independent check standards and field samples.

10.2.3 A Linear Through Zero curve type is be selected for all analytes.

10.3 Equipment Operation and Sample Processing

Samples are prioritized by the Section Head for analysis based on hold time and client due date. Section 11 outlines the steps for data reporting that will contain the sample analysis final results.

10.3.1 Preliminary treatment or sample preparation of most matrices is necessary because of the complexity and variability of sample matrices. Aqueous dissolved samples that have

been pre-filtered and acidified do not require acid digestion as long **as the samples and standards are matrix matched.** See the sample preparation SOPs.

10.3.2 Ensure that the instrument configuration and operating procedures established in Section 10.1 are selected.

10.3.3 Affix clips to the peristalic pump windings and open the Device window, click on Connect and click on the right pointing arrow (counterclockwise). Check that the fluid is flowing through all tubing and that waste is flowing out of the spray chamber. Initiate the plasma and allow a warm-up of 30-60 minutes.

10.3.4 Conduct mass calibration and resolution check.

Note: Tuning and optimization procedures are performed without the Internal Standard mixing block in-line for The ELAN 6100.

10.3.4.1 Open the Tuning workspace. Aspirate the Tuning Solution (8.8.2) solution containing Be, Co, In, Tl and U. Click on Tune Mass Spec. The measured mass difference must be less than 0.1 amu from the actual value. The resolution must be less than 0.9 amu full width at a 10% peak height (the instrument default setting is 0.65 amu). If the resolution is not achieved for an element, the Resolution Digital to Analog Conversion (Res. DAC) value is changed for that element (increasing the DAC value by 30 units will decrease the peak width by 0.1 amu). If the Measured Mass values are not within 0.1 amu of the exact value then the Mass DAC values must be changed. Increasing the Mass DAC increases the measured mass value. This is done daily before sample analysis. Print the calibration and store with the daily performance check.

Note: Extreme care must be taken when adjusting DAC values. Instrument performance can be severely degraded if tuning parameters are not correct.

10.3.4.2 Open the Daily Performance workspace

10.3.4.2.1 Aspirate the Daily Performance Solution (8.18).

10.3.4.2.2 Click on “Analyze Sample” in the manual sample window.

10.3.4.2.3 Monitor daily performance measures as recommended by Perkin Elmer for Ba, Ce, Mg, Rh, Pb, In and U sensitivity, background, % double charged and % oxide levels.

10.3.4.2.4 See Manufacturer’s recommendations for sensitivity. If criteria are not met then follow optimization procedures specified by the manufacturer.

10.3.4.2.5 The background at mass 220 should be < 30 cps.

10.3.4.2.6 The % double charged ions (Ba^{+}/Ba^{++}) should< 3% and not to exceed 5%.

10.3.4.2.7 The % oxides (Ce/CeO)should be < 3% and not to exceed 5%.

10.3.5 Open the appropriate analytical method. Turn on the autosampler and click on the “Sampling” icon in the Method. Select “Go to Rinse” at which point the sampling probe should go to the rinse station. Click on “OK”. It may not be necessary to analyze for all the metals listed in the 6020A method. The analyte list can be edited down for only those metals required. Go to the Timing Page in the method and highlight the metals to be deleted. Go to the Edit Menu and select “delete rows”. Save the method as a different name. For reporting purposes, the Report File Name on the Report Page of the 6020 Method or edited Method is saved under c:\elandata\dataset\dataset**LIMS WG#. etc.** for each analysis on that date.

10.3.6 Open the "Sample" window and click on batch analysis to update with new sample information.

10.3.7 Open the 6020TEMP sample file or a previously used file and edit the table with sample names, autosampler positions and dilutions. Select the Method for analysis and record all standard ID's and acids used in the Description Column of the Batch Analysis page. Save the file as a different name.

10.3.8 Calibrate the instrument using the calibration standards listed in Section 8.0, Reagents and Standards. The system must be flushed with a rinse blank sample (equivalent to the calibration blank) between each standard to protect against potential carry-over. The average intensity of 3 multiple exposures must be performed for QC samples and field samples to reduce random instrument error. The calibration curve consists of a blank and two to three standards.

10.3.9 To ensure that there is sufficient sample to fill the loop of the 6 port valve, there should be at least 5 mLs in each autosampler tube.

10.3.10 The Calibration action for the first sample for which concentration results are desired must be "Analyze Blank, Standards, and Sample".

10.3.11 The calibration action for all other samples is usually "Analyze Sample", unless periodic re-calibration is desired.

10.3.12 Click on the "Dataset" Icon, go to the "File Menu" and click on "New". The dataset name is designated by **LIMS WG#.**

10.3.13 Select "Analyze Batch"

10.3.14 Flush the system with the rinse blank solution between samples. The rinse blank nitric acid concentration corresponds to the acid concentration in samples and standards. The DRCe, 10% nitric acid or 5% hydrochloric/5% nitric acid (for digests containing hydrochloric acid) is used for the rinse. The same time must be elapsed between CCVs and CCBs as is allowed between samples. Analyze the CCV and the CCB after each 10 samples and at the end of the analytical sequence.

10.3.15 The LLCV (RL check standard) must be analyzed at the beginning and end of an analytical run., ICSA, and ICSAB solutions must be analyzed at the beginning of an analytical run or once every 12 hours, whichever is more frequent. A rinse may be analyzed after ICSA and ICSAB in order to prevent carryover into samples.

The LLCV analysis is not performed if a calibration standard is prepared at the reporting limit and included in the initial calibration curve. The calibration curve must always meet the criteria for linearity (r = 0.998).

10.3.16 Analysis Review

Two forms are used to primary and secondary review the analysis. These are called the "ICP MS Checklist" and the "Blank Reporting Limit Checklist" and are shown below.

10.3.16.1 ICP MS Checklist

This analysis review form is used by the analyst and secondary reviewer to note passing/failing QC standards and to record any other comments as communication for reporting sample results.

10.3.16.2 Blank Reporting Limit Checklist

This analysis review form is used to set the sample reporting limit based on the batch preparation blank and to compare instrument blanks (ICB and CCB's) to instrument detection limits. Instrument blanks are compared to the limit of 3 times

the IDL. Instrument blanks, which exceed the IDL limit, are recorded on the form and may require sample reanalysis or reporting limit elevation. This form is generated annually as IDL studies are completed.

10.3.17 Target analytes detected above the calibration range must be diluted and re-analyzed. See Section 9 for all Quality Control frequencies and criteria.

10.4 Continuing Calibration

See Section 9.4.

10.5 Preventive Maintenance

10.5.1 General cleaning tips for ICP-MS

10.5.1.1 Sampling and skimmer cones should be removed and cleaned on a weekly basis or more often under high usage. Remove the cones with the tool provided with the instrument and clean with 10% HCl, and rinse thoroughly with deionized water. Reinstall cones and perform XY alignment procedures after igniting the plasma and the instrument has warmed up for 30 minutes.

10.5.1.2 Peristaltic pump tubing must be inspected regularly for wear and changed if necessary. Sample and internal standard tubing should be changed weekly or more often if necessary. Rinse and drain tubing less often.

10.5.1.3 The aspiration chamber may be cleaned periodically using the sonication soap solution in the sonication bath for 0.5 hrs and rinsed with 10% HNO_3 and deionized water.

10.5.1.4 To clean the ion lens, the vacuum must be turned off, the cover to the vacuum chamber removed and the autolens taken out. Clean with Softscrub and a toothbrush, rinse with hot water, then DI water.Dry thoroughly and return to the chamber. Replace the cover and evacuate.

10.5.2 Electron multiplier voltages require periodic optimization if Daily Performance Standard criteria in Section 10.3.4.2 are not met (see Operators Manual).

11. Data Evaluation, Calculations and Reporting

11.1 All calculations necessary to convert raw data (ion counts/second) are performed by the ELAN software. The calculated quantities are selected by choosing the desired options in the Report Options screen. The default report option for the ELAN 6020 Method is 6020.rop.

11.2 All calculations performed in the ELAN software are based on the ratio of the analyte intensity (cps) to the internal standard intensity (cps). In all calculations where internal standards are used, the ratio of the analyte intensity to internal standard intensity is taken before any other calculation is performed. *Note: Method 6020 requires the use of internal standards.*

11.3 The metal results are calculated by the following equation:

Aqueous:

$$\text{Metal result in } \mu g/L = \frac{C \times B}{A} \times DF$$

where:

A = Initial sample volume in mL, typically 25 mL
B = Digestate final volume in mL, typically 25 mL

C = Concentration of sample from instrument read-out in µg/L
DF = Dilution Factor

Solid:

$$\text{Metal result in mg/Kg} = \frac{\frac{C \times B}{A} \times DF \times \frac{100}{DW}}{1000}$$

where:

A = Initial sample weight in grams, typically 1 gram
B = Digestate final volume in mL, typically 50 mL
C = Concentration of sample from instrument read-out in µg/L
DF = Dilution Factor
DW = Dry Weight

11.4 To calculate Hardness:

(Ca result (in mg/L) x 2.5) + (Mg result (in mg/L) x 4.12) = Hardness in mg/L

11.5 All metal results should be reported to three significant figures

11.6 The primary analyst does data entry into the LIMS system. The LIMS is "linked" to the instrument, so the analyst must choose the sample(s) and elements to be reported from that analytical sequence. All associated preparation and instrumental QC samples and dilutions are also chosen. The laboratory generates two types of data packages from the LIMS: "Commercial" for routine projects, and "Full Deliverable" or "CLP-like" for fully data validated projects. A Commercial package consists of sample results and the associated method blank and LCS results. A Full Deliverable package includes all sample results, all preparation and instrumental QC results and the associated supporting raw data.

11.7 All solids including soils, sediments, and sludges must be reported on a dry-weight basis. Tissue results may be reported in wet-weight depending upon client request.

11.8 A secondary review is performed on all data.

11.9 If interferences on sample analysis resulting in false positive, false negative or biased results are suspected or have been determined, it may be possible to eliminate or minimize these interferences through use of the DRCe ICP MS equipped with a reaction cell. The reaction cell allows for the introduction of oxygen and ammonia to react with the analyte or the interfering ion.

11.9.1 Arsenic analysis

The ArCl interference (see section 4.2) on As at m/z of 75 can be removed by introducing oxygen into the reaction cell. Oxygen reacts with As to form AsO. This analyte is measured at m/z of 91 and is not affected by the ArCl interference. The internal standard used for this analysis is 300 µg/L In. The following conditions have been found to be suitable for analysis of As in seawater. Seawater is a source of high chloride concentration.

Analyte	Cell Gas	Flow Rate (ml/min)	RP_0	RP_q
As	Oxygen	0.6	0	0.75
In	Oxygen	0.6	0	0.85

Note: These conditions may change to optimize the analysis for a particular sample matrix.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 outlines sample batch QC acceptance criteria. If non-compliant metals results are to be reported, the Department Manager and/or the Laboratory Director must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Section Head performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Instrument Detection Limit

The instrument detection limit (IDL) is the smallest signal above the background noise that an instrument can detect. The IDLs can be calculated by multiplying by 3, the average standard deviations for measurements of a reagent blank analyzed on three analytical runs on three non-consecutive days. Seven consecutive measurements must be taken per day. The IDLs are not required to go through sample digestion. Each measurement must be performed as though it were a separate sample. The current IDLs are available upon request.

13.3 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.3.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.3.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1732 DL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1797 Waste Management and Disposal SOP

16. Attachments

Table 1: Reporting Limits
Table 2: QC Acceptance Criteria
Table 3: Isotopes Monitored and Equations Used
Table 4: Initial Calibration Levels and ICV/CCV Concentrations
Table 5: Initial Calibration Levels and ICV/CCV Concentrations for 1 Point Curve
Table 6: ELAN 6100 and DRCe Linear Ranges for Method 6020A

Table 1 – Reporting Limits

Metal	Aqueous (µg/L)	Solid - Hot Plate (mg/Kg)	Tissue – Microwave (mg/Kg)
Aluminum	100	10	10
Antimony	0.5	0.05	0.05
Arsenic	0.5	0.05	0.1
Barium	0.5	0.3	0.05
Beryllium	0.5	0.03	0.05
Boron	10	0.5	0.5
Cadmium	0.5	0.02	0.02
Calcium	100	50	20
Chromium	1.0	0.2	0.05
Cobalt	0.2	0.05	0.05
Copper	1.0	0.2	0.05
Iron	50	20	10
Lead	1.0	0.06	0.02
Magnesium	70	10	10
Manganese	0.5	0.2	0.1
Molybdenum	0.5	0.05	0.05
Nickel	0.5	0.1	0.05
Potassium	100	10	10
Selenium	5.0	0.2	0.1
Silver	0.2	0.05	0.05
Sodium	100	15	10
Strontium	0.5	0.1	0.05
Thallium	0.2	0.02	0.02
Tin	0.5	0.1	0.10
Titanium	0.5	0.2	0.1
Vanadium	5.0	0.1	0.1
Zinc	10	1.0	0.5

Table 2 – QC Acceptance Criteria

QC Parameter	Acceptance Criteria
Tuning Solution	≤ 5% RSD
Mass Calibration	Must not differ by more than 0.1 amu from true value
Resolution Checks	Less than 0.9 amu at 10% peak height
Initial Calibration Curve	R ≥ 0.998
Method Blank	< reporting limit
Laboratory Control Sample	80-120%R for aqueous and Manufacturer's control limits for solid
Matrix Duplicate	≤ 20%RPD for results >5x reporting limit
Matrix Spike	75-125%R for solid and aqueous
Matrix Spike Duplicate (if requested)	75-125%R for solid and aqueous; and ≤20% RPD
Initial and Continuing Calibration Verification	90-110%R
Initial and Continuing Calibration Blank	<RL
Reporting Limit Check	70-130% R (DoD = 80-120%R)
ICSA and ICSAB Solution	80-120%R for spiked analytes
Internal Standard	70-120%R (Method 6020A) for instrument QC samples and field samples
Serial Dilution Sample	<10% D
Post Digestion Spike	80-120%R

Table 3 - Isotopes Monitored and Equations Used

1.1.1.1 / 1.1.1.2 Analyte	Symbol	Isotopes Monitored (primary in bold)	1.1.1.3 / 1.1.1.4 Correction Equations
Aluminum	Al	27	
Antimony	Sb	**121**,123	Sb 123 = Sb 123 - 0.127189 * Te 125
Arsenic	As	75	As 75 = As 75 - 3.127 * [ArCl 77 - (0.815*Se 82)]
Barium	Ba	135,**137**	
Beryllium	Be	9	
Cadmium	Cd	106,108,111,**114**	Cd 111 = Cd 111 - 1.073 * [MoO 108 -(0.712*Pd 106)] Cd 114 = Cd 114 - 0.026826 * Sn 118
Chromium	Cr	**52**,53	
Calcium	Ca	44	
Cobalt	Co	59	
Copper	Cu	**63**,65	
Iron	Fe	54	Fe 54 = Fe 54 - 0.028226 * Cr 52
Lead	Pb	206,**207**,208	Pb 208 = Pb 208 + 1* Pb 206 + 1* Pb 207
Calcium	Ca	44	
Manganese	Mn	55	
Molybdenum	Mo	95,**97**,98	Mo 98= Mo 98 - 0.110588 * Ru 101
Nickel	Ni	**60**,62	
Potassium	K	39	
Selenium	Se	77,**82**	Se 82 = Se 82 - 1.008696 * Kr 83
Silver	Ag	**107**,109	
Sodium	Na	23	
Thallium	Tl	203,**205**	
Tin	Sn	118	
Vanadium	V	51	V 51 = V 51 - 3.127*[ClO 53 - (0.113*Cr 52)]
Zinc	Zn	**66**,67,68	
1.1.1.4.1 Internal Standards (alternates in parentheses)			
Lithium	Li	6	
Scandium	Sc	45	
Germanium	Ge	74	
(Yttrium)	Y	89	
(Rhodium)	Rh	103	
Indium	In	115	
Terbium	Tb	159	
(Bismuth)	Bi	209	

Table 4 - Initial Calibration Levels and ICV/CCV Concentrations

Analyte	STD1	STD2	STD3	STD4	STD5	ICV,CCV
	(µg/L)	(µg/L)	(µg/L)	(µg/L)	(µg/L)	(µg/L)
Ag	0.20				100	50
Al	10	110	1020	10050		5050
As	0.50	10	20	100		50
B	5.0	10	20	100		50
Ba	0.50				100	50
Be	0.30	10	20	100		50
Ca	100	110	1020	10100		5050
Cd	0.20	10	20	100		50
Co	0.20	10	20	100		50
Cr	0.50	10	20	100		50
Cu	0.50	10	20	100		50
Fe	10	110	1020	10100		5050
K	100	100	1000	10000		5500
Mg	10	110	1020	10100		5050
Mn	0.50	10	20	100		50
Mo	0.50	10	20	100		50
Na	100	100	1000	10000		5050
Ni	0.50	10	20	100		50
Pb	0.20	10	20	100		50
Sb	0.50	10	20	100		50
Se	1.0	10	20	100		50
Si	0.05	5	10	50		25
Sn	0.10	10	20	100		50
Sr	0.50	10	20	100		50
Ti	0.50	10	20	100		50
Tl	0.20	10	20	100		50
V	1.0	10	20	100		50
Zn	5.0	10	20	100		50

Table 5 - Initial Calibration Levels and ICV/CCV Concentrations for 1 Point Curve

Analyte	STD1	STD2	ICV,CCV
	(µg/L)	(µg/L)	(µg/L)
Ag		100	50
Al	10050		5050
As	100		50
B	100		50
Ba		100	50
Be	100		50
Ca	10100		5050
Cd	100		50
Co	100		50
Cr	100		50
Cu	100		50
Fe	10100		5050
K	10000		5500
Mg	10100		5050
Mn	100		50
Mo	100		50
Na	10000		5050
Ni	100		50
Pb	100		50
Sb	100		50
Se	100		50
Si	50		25
Sn	100		50
Sr	100		50
Ti	100		50
Tl	100		50
V	100		50
Zn	100		50

Table 6 - ELAN DRCe Linear Range for Method 6020A.

Analyte	Mass	Linear Range (mg/L)
Ag	107	5
Al	27	50
As	75	5
B	11	5
Ba	138	5
Be	9	5
Ca	44	50
Cd	114	5
Co	59	5
Cr	52	5
Cu	63	5
Fe	57	50
K	39	50
Mg	24	50
Mn	55	5
Mo	98	5
Na	23	50
Ni	60	50
Pb	208	5
Sb	121	5
Se	82	5
Si	28	5
Sn	118	5
Ti	48	5
Tl	205	5
V	51	5
Zn	66	5

Table 6-LCS and Matrix Spike

Analyte	Liquid Concentration (mg/L)	Soil Concentration * (MS spike only) (mg/Kg)
Antimony	0.5	160
Arsenic	0.12	160
Barium	2.00	160
Beryllium	0.05	80
Cadmium	0.051	80
Chromium	0.20	160
Copper	0.25	160
Lead	0.51	160
Nickel	0.50	160
Selenium	0.12	160
Silver	0.05	40
Thallium	0.12	160
Zinc	0.50	160
Iron	1.00	800
Manganese	0.50	160
Calcium	10.0	800
Magnesium	10.0	800
Potassium	10.0	800
Sodium	10.0	800
Aluminum	2.00	800
Cobalt	0.50	160
Vanadium	0.50	160
Boron	1.0	NA
Molybdenum	1.0	NA
Titanium	1.0	NA

*MS spike of a solid based on 1.25g and a final volume of 50 mL; refer to method 3050 for spike amounts.

Note: Solids LCS is an SRM with certified value provided by the vendor on a lot basis.

Mercury Determination in Tissue and Soil/Sediment Samples by Cold Vapor Atomic Fluorescence Technique (CVAF)

References: **Method 7474,** Mercury in Sediment and Tissues by Atomic Fluorescence Spectrometry. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 0, 2007.

Method 7471B, Mercury in Solid or Semisolid Waste (Manual Cold Vapor Technique). SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 2, February 2007.

1. Scope and Application

Matrices: The method is applicable to total mercury in tissue and soil/sediment samples.

Definitions: Refer to Alpha Analytical Quality Manual.

Two procedures for the analysis of mercury by cold vapor atomic fluorescence spectrometry are discussed in this SOP.

Mercury in Tissues - This method utilizes a microwave digestion followed by bromine chloride oxidation. Samples are then decolorized using hydroxylamine hydrochloride and analyzed following reduction with stannous chloride. Elemental mercury is then purged from solution and analyzed by atomic fluorescence spectrometry.

Mercury in Soil/Sediments - This method utilizes a hot plate digestion with aqua regia followed by bromine chloride oxidation. Samples are then decolorized using hydroxylamine hydrochloride and analyzed following reduction with stannous chloride. Elemental mercury is then purged from solution and analyzed by atomic fluorescence spectrometry.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Mercury analyzer and in the interpretation of Mercury data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Parameter	CAS
Mercury	7439-97-6

2. Summary of Method

Mercury in tissue samples is determined by cold vapor atomic fluorescence spectrometry from an aliquot of a microwave digestion (see SOP 2150). A 10 mL aliquot of the digest is added to a 50 mL

screw cap polyethylene digestion tube along with 2 mL of an acidic bromine chloride solution. Bromine chloride is prepared by mixing potassium bromide with potassium bromate solutions with hydrochloric acid producing a yellow solution. Bromine chloride is known to oxidize inorganic and organomercury compounds. If a yellow color does not persist after addition of bromine chloride, then additional solution must be added until a yellow color persists. The same volume of bromine chloride solution is added to all samples and standards. This solution is allowed to stand for at least 12 hours. After digestion is complete, bring to 50 mL, add 0.4 mL of 12% hydroxyl amine hydrochloride solution and shake. Additional hydroxyl amine hydrochloride is added if the yellow color from the bromine chloride solution persists.

Samples and standards are placed in the autosampler rack for analysis following stannous chloride reduction and analysis by cold vapor atomic fluorescence spectrometry at a wavelength of 253.7 nm.

Mercury in soil/sediment samples is determined by cold vapor atomic fluorescence spectrometry from a hot plate digestion utilizing aqua regia. A 0.5 – 1 g wet sediment aliquot is added to a 50 mL screw cap polyethylene digestion tube along with 5 mL of DI water, 3.75 mL concentrated HCl and 1.25 mL of concentrated HNO_3. Samples are heated on the hot block for 2 minutes and allowed to cool. 25 mL of Di water are then added, the samples placed back on the hot block and heated for 30 minutes. The samples are then cooled and brought to 50 mL with DI water. A 10 mL aliquot of the digest is added to a 50 mL digestion tube along with 2 mL of an acidic bromine chloride solution. If a yellow color does not persist after addition of bromine chloride then additional solution must be added until a yellow color persists. The same volume of bromine chloride solution is added to all samples and standards. This solution is allowed to stand for at least 12 hours. After digestion is complete, bring to 50 mL, add 0.4 mL of 12% hydroxyl amine hydrochloride solution and shake. Additional hydroxyl amine hydrochloride is added if the yellow color from the bromine chloride solution persists.

Samples and standards are placed in the autosampler rack for analysis following stannous chloride reduction and analyzed by cold vapor atomic fluorescence spectrometry at a wavelength of 253.7 nm.

2.1 Method Modifications from Reference

Method 7474 calls for a microwave digestion for both tissues and soils/sediments with nitric and hydrochloric acids. The method has been modified for tissues to use an aliquot of a microwave digest utilizing nitric acid only. The method has been modified for soils/sediments to use a hot block digestion similar to that used for Method 7471B but without the addition of potassium permanganate. The change allows for more rapid preparation of samples and yields equivalent data to the reference method as indicated by the performance of PT samples.

Bromate/Bromide solution has been modified from the method reference.

3. Reporting Limit

The Reporting limit for mercury in tissues is 0.05 µg/L (0.0125 mg/kg) and the linear range of the analysis is 5 µg/L (1.25 mg/kg).

The Reporting limit for mercury in soil/sediment is 0.05 µg/L (0.0125 mg/kg) and the linear range of the analysis is 5 µg/L (1.25 mg/kg).

4. Interferences

4.1 Gold, silver and iodide are known interference.

4.1.1 High concentrations of gold and silver in a sample may suppress mercury reduction.

4.1.2 Samples with iodide concentrations greater than 3 mg/L should be pre-reduced with stannous chloride to clarify the brown color. It may be necessary to clean the analytical system with 4N HCl following analysis of a sample with iodide concentrations greater than 30 mg/L.

4.1.3 Sulfide concentrations up to 24 mg/L produce no interference problems.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Eating, drinking, smoking, or the application of cosmetics is not permitted in the laboratory area. Horseplay of any kind is prohibited. Pipetting by mouth is not permitted. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and before entering the employee lounge or eating area. Always wash your hands before leaving the laboratory. All relevant Material Safety Data Sheets (MSDSs) are kept alphabetically in a centrally located file storage.

Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled under a hood using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting device and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

Spilled samples, solvents, reagents, and water must be cleaned up from bench tops, instruments and autosampler surfaces immediately. A spill is considered a quantity of hazardous material if it is moderate to extreme hazard to the skin and mucous membranes. If contact with the skin occurs, immediately flush with large volumes of water. In the case of acidic/basic spills, the *Spill Kit* located in each laboratory shall be utilized before attempting to cleanup the spill. Although procedures are designed to minimize the possibility of an accident, all injuries or accidents, regardless of the nature or severity, are to be reported to the Department Manager Supervisor immediately. If an employee discovers a potentially unsafe condition, this must be reported to the Department Manager Supervisor immediately. No employee should feel compelled to work in a situation where they do not feel entirely informed, trained, or safe.

Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintenance an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

Compressed gas cylinders shall only be moved with the dolly supplied for this specific purpose. The cap must be on the cylinder while it is being moved. The tank must be secured when in its final position. All spent tanks are to be returned in the same manner, and secured until removed by the vendor. Liquid argon or nitrogen represents a potential cryogenic hazard and safe-handling procedures must be used at all times.

All additional company safety practices shall be followed at all times as written in the *Chemical Hygiene Plan.*

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

A minimum of 10g of sample must be collected in a pre-cleaned 2oz or 4oz glass jar with a Teflon lined screw cap.

6.2 Sample Preservation

No preservatives are used. See Section 6.4.

6.3 Sample Shipping

No special sample shipping requirements.

6.4 Sample Handling

Samples are stored at 4°C $\pm$ 2°C. The hold time for soil/sediment and tissue samples is 28 days. Sediment and tissue samples may be stored frozen at -20°C $\pm$ 10°C. The hold time for solid samples stored frozen may be extended unless specifically noted by project or client requirements.

7. Equipment and Supplies

7.1 Instrument: The PSA Millennium Merlin Atomic Fluorescence Analyzer equipped with a PC loaded with instrumental software, gold-amalgamation attachment (Millennium Gallahad) and PSA 20.400 autosampler. The instrument is connected to a source of high-purity, mercury free argon regulated to 50 PSI.

7.2 Santoprene Peristaltic Pump Tubing: Green/Green (PSA part number M025T002 or equivalent) for blank and autosampler rinse, Grey/Grey (PSA part number M055T005 or equivalent) for sample and stannous chloride.

7.3 Electric Hot Plate: Adjustable and capable of maintaining a temperature of 90-95^{o}C equipped with graphite carbon blocks that each have 36 positions to hold the sample tubes.

NOTE: Hotplate/Block temperatures are monitored regularly using NIST calibrated and traceable thermometers. If any thermometer is suspected to not be reading the temperatures correctly, it is replaced with another certified thermometer.

7.4 Milestone Ethos E Microwave Oven: Equipped with temperature control probe and high pressure perfluoropolymer vessels.

7.5 Digestion Tubes: 50mL disposable polyethylene screw cap digestion tubes.

7.6 Other: Adjustable Eppendorf pipettes and replacement tips

8. Reagents and Standards

ACS Trace Metal grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

Solutions below expire six months from preparation unless noted.

Stock standard solutions are stored in a cabinet, out of direct light

8.1 Deionized (DI) water: The Barnstead NANO-pure system provides Type I water used in the preparation of samples and standards.

8.2 Hydrochloric acid, concentrated (HCl): ACS reagent grade, Fisher #A508-212, or equivalent. Hydrochloric acid (1:1 HCl) - Add 500mL concentrated HCl to 400mL DI water and dilute to 1L. 10% HCl – Add 100 mL concentrated HCl to approximately 500 mL DI and bring to 1 L with DI. Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.3 Nitric acid, concentrated (HNO_3): ACS reagent grade, Fisher #A509-212, or equivalent. Nitric Acid (1:1 HNO_3) - Add 500mL concentrated HNO_3 to 400mL DI water and dilute to 1L. Prepare 1% Nitric Acid by diluting 1mL of concentrated HNO_3 up to 100mL in DI water. Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.4 Hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$): ACS reagent grade, VWR #HX0770-1, or equivalent. Prepare 12% solution by adding 12 g per 100 mL of DI.

8.5 Stannous Chloride ($SnCl_2{\cdot}H_20$): ACS reagent grade, Fisher #T142-500, or equivalent. Prepare 2% solution by adding 20 g to 1L of 10% HCl. Prepare daily. Solution may be purged with argon at 2 L/minute for 30 minutes to remove Hg impurities.

8.6 Potassium Bromate ($KBrO_3$): ACS grade, Fisher #P207-250. May be baked in muffle furnace at 250^{o}C for 8 hours to remove mercury impurities.

8.7 Potassium Bromide (KBr): ACS grade, Fisher #P205-500. May be baked in muffle furnace at 250^{o}C for 8 hours to remove mercury impurities.

8.8 Bromine Chloride (BrCl): Prepare in a BOD bottle under a fume hood by adding 5.4 g KBr and 7.6 g $KBrO_3$ to 500mL of concentrated HCl and mix. Final concentration is 0.2N. Solution will turn yellow. **Free Halogens are generated from this solution. Seal the bottle when taking out of fume hood.**

8.9 Stock and Working Standards:

The 1000 µg/L working standards must be prepared in the same manner as samples. These standards are then diluted to make calibration and calibration check standards. After preparation, the 1000 µg/L working standards can be used for one week.

8.9.1 1000 mg/L Stock Calibration Mercury Standard: Ultra Scientific #ICP-080, or equivalent.

8.9.1.1 1000 µg/L Working Standard for Tissues: Prepare by adding 0.05mL of 1000 mg/L stock to a microwave digestion vessel containing 10mL of concentrated HNO_3. This standard is digested along with samples according to Method 3051 (SOP 2150). The standard is then brought to a 50mL final volume with DI water in a screw cap digestion tube. This standard is identified as **C-date-VM** in the Mercury Standards Preparation Log.

8.9.1.2 1000 µg/L Working Standard for Soils/Sediments: Prepare by adding 0.05 mL of 1000 mg/L stock (Section 8.9.1) to a 50 mL screw cap digestion tube containing 5 mL of DI water, 1.25 mL of concentrated HNO_3 and 3.75 mL of concentrated HCl. This standard is digested along with samples. The standard is then brought to a 50 mL final volume with DI water. This standard is identified as **C-date-VH** in the Mercury Standards Preparation Log.

8.9.1.3 100 µg/L Intermediate Standard: Prepare by adding 0.1 mL of the 1000 µg/L working standard (Section 8.9.1.1 or 8.9.1.2) to 0.9 mL DI water. This intermediate standard is used for calibration standards preparation and then discarded.

8.9.1.4 10 µg/L Intermediate Standard: Prepare by adding 0.1 mL of the 100 µg/L working standard (Section 8.9.1.3) to 0.9 mL DI. This intermediate standard is used for calibration standards preparation and then discarded.

8.9.1.5 5 µg/L Intermediate Standard: Prepare by adding 0.25 mL of the 1000 µg/L working standard (Section 8.9.1.1 or 8.9.1.2) to 50 mL graduated digestion tube containing 2% HCl. Bring to 50 mL final volume.

8.9.2 1000 mg/L Stock ICV Mercury Standard: Inorganic Ventures #CGHG-1, or equivalent

8.9.2.1 1000 µg/L ICV Working Standard for Tissues: Prepare by adding 0.05 mL of 1000 mg/L stock to a microwave digestion vessel containing 10 mL of concentrated HNO_3. This standard is digested along with samples according to Method 3051 (SOP-2150). The standard is then brought to a 50 mL final volume with DI water in a screw cap digestion tube. This standard is identified as **I-date-VM** in the Mercury Standards Preparation Log.

8.9.2.2 1000 µg/L ICV Working Standard for Soils/Sediments: Prepare by adding 0.05 mL of 1000 mg/L stock to a 50 mL screw cap digestion tube containing 5 mL of DI water, 1.25 mL of concentrated HNO_3 and 3.75 mL of concentrated HCl. This standard is digested along with samples. The standard is then brought to a 50 mL final volume with DI water. This standard is identified as **I-date-VH** in the Mercury Standards Preparation Log.

8.9.2.3 5 µg/L Intermediate Standard: Prepare by adding 0.25 mL of the 1000 µg/L working standard (Section 8.9.2.1 or 8.9.2.2) to 50 mL graduated digestion tube containing 2% HCl. Bring to 50 mL final volume.

8.9.2.4 1000 µg/L LCS and Matrix Standard for Tissues and Soils/Sediments: Prepare by adding 0.1 mL of 1000 mg/L stock (8.9.2) to a 100 mL volumetric flask containing 2% HCL. This standard is identified as **I-date-V** in the Mercury Standards Preparation Log.

8.10 Calibration and Calibration Check Standards

8.10.1 Standards Preparation for Method 7474 Tissues

To 50 mL screw cap digestion tubes, add 10 mL of DI water and 2 mL of concentrated HNO3. Add the volumes of each standard as indicated in the table below, 2 mL of BrCl solution and bring to 50 mL. The standard set is designated in the Mercury Standards Preparation Log as **C-date-VM1,2,3…** (for calibration standards) and **I-date-VM1,2,3…** (for the ICV) as successive sets of standards are prepared from the stock. The same volume of the 1000 µg/L ICV stock is added to the preparation batch LCS and matrix spike sample.

If more than 2 mL of BrCl solution was added to samples to maintain the yellow color, add the same amount to standards. 0.2 mL of 12% hydroxyl amine hydrochloride is added to each vial and shaken. The yellow color should disappear.

8.10.2 Standards Preparation for Method 7474 Soils/Sediments

To 50 mL screw cap digestion tubes, add 10 mL of DI water, 0.75 mL of concentrated HCl and 0.25 mL of concentrated HNO_3. Add the volumes of each standard as indicated in the table below, 2 mL of BrCl solution and bring to 50 mL. The standard set is designated in the Mercury Standards Preparation Log as **C-date-VH1,2,3…** (for calibration standards) and **I-date-VH1,2,3…** (for the ICV) as successive sets of standards are prepared from the stock. The same volume of the 1000 µg/L ICV stock is added to the preparation batch LCS and matrix spike sample.

If more than 2 mL of BrCl solution was added to samples to maintain the yellow color, add the same amount to standards. 0.2 mL of 12% hydroxyl amine hydrochloride is added to each vial and shaken. The yellow color should disappear.

8.10.3 Calibration, Calibration Check Standards, LCS and Matrix Spike

Calibration and calibration check standards are prepared by adding standard volumes to vials prepared for tissues and soils/sediments as designated in the following table:

Lower detection limits and analytical reporting limits may be required at times for some samples. Lower limits are attained by increasing the instrumental gain from 10 to 100 and by calibrating using lower concentration standards. Calibration standards and the LCS and MS are prepared as in the following table:

Standard ID	Vol. 5 µg/L Standard (Section 8.9.1.5) (mL)	Final Concentration (µg/L)
STD0	-	0.000
STD1	0.05	0.005
STD2	0.10	0.010
STD3	0.20	0.020
STD4	0.50	0.050
STD5	1.0	0.100
STD6	2.0	0.200
ICV,CCV,LCS, MS	1.0 **(Section 8.9.2.3)**	0.100

9. Quality Control

Standard ID	Vol. 1000 µg/L (mL) (Sec. 8.9.1.1 or 8.9.1.2)	Vol. 100 µg/L (mL) (Sec. 8.9.1.3)	Vol. 10 µg/L (mL) (Sec. 8.9.1.4)	Final Concentration (µg/L)
STD0	-	-	-	0.0
STD1	-	-	0.25	0.05
STD2	-	0.05	-	0.1
STD3	-	0.25	-	0.5
STD4	0.1	-	-	2.0
STD5	0.25	-	-	5.0
ICV, CCV	0.125 (Sec. 8.9.2.1 or 8.9.2.2)			2.5
LCS, MS	0.125 (Sec. 8.9.2.4)			2.5

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blanks

9.1.1 Method Blank

A method blank must be prepared once per every 20 samples or per digestion batch, whichever is more frequent.

Mercury must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. Digestion of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control method blank are non-detect for the elements of interest, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

9.1.2 Initial Calibration Blank (ICB)

An initial calibration blank must be analyzed immediately following the ICV (see 9.3).

Mercury must not be detectable in the ICB at a concentration greater than the reporting limit.

Corrective Action: May reanalyze the standard once. If the concentration still exceeds the acceptance limit, stop and recalibrate the instrument. Samples cannot be analyzed until an acceptable ICB analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control blank are non-detect for mercury, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high blank and the client is notified in a project narrative associated with the sample results.

9.1.3 Continuing Calibration Blank

A continuing calibration blank must be analyzed immediately following the CCV (see 9.4).

Mercury must not be detectable in the CCB at a concentration greater than the reporting limit.

Corrective Action: May reanalyze the standard once. If the concentration still exceeds the acceptance limit, stop, recalibrate the instrument and reanalyze all associated samples. Samples cannot be analyzed until an acceptable CCB analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control blank are non-detect for the elements of interest, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high

method blank and the client is notified in a project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)

Laboratory control sample (LCS) must be prepared once per every 20 samples or per digestion batch, whichever is more frequent, and spiked with a solution prepared from a second source or lot number, other than the source used to verify the accuracy of the standard curve for the determinative analytical method. The LCS contains all target elements of interest, and is digested along with the samples as verification of the accuracy of the entire digestion procedure.

The acceptable recovery QC limits are 80 – 120%. The recovery limits are continuously monitored and documented in-house through control charts. The *Control Limit Generation* SOP (1734) provides details explaining how control charts are generated and used for quality control.

Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-prepare and re-analyze the LCS and all associated samples. Samples cannot be reported until an acceptable LCS is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control LCS are also associated with a matrix spike that is in control. This is an acceptable measure of accuracy of the digestion and analytical procedures. An explanation of the out of control LCS recovery must be included in the project narrative to the client and the sample data reported noting the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

An initial calibration verification standard from a source different from that used to calibrate the instrument (see 8.9.2) must be analyzed immediately following calibration as an independent check of instrument performance. Nominal acceptance limits are 90% - 110% of the true value.

Corrective Action: May reanalyze the standard once. If the concentration still exceeds the acceptance limit, stop and recalibrate the instrument. Samples cannot be analyzed until an acceptable ICV analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with a high recovery ICV are non-detect. In such cases, the sample results are accepted without corrective action for the high ICV recovery and the client is notified in a project narrative associated with the sample results.

9.4 Continuing Calibration Verification (CCV)

A continuing calibration verification standard must be analyzed after analysis of no more than 10 samples as an on-going check of instrument performance. Nominal acceptance limits are 90% - 110% of the true value.

Corrective Action: May reanalyze the standard once. If the concentration still exceeds the acceptance limit, stop, recalibrate the instrument and reanalyze all associated samples. Results cannot be accepted until an acceptable CCV analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with a high recovery CCV are non-detect. In such cases, the sample results are accepted without corrective action for the high CCV recovery and the client is notified in a project narrative associated with the sample results.

9.5 Matrix Spike

A matrix spike (MS) sample must be performed once per 20 samples (5% frequency), or per digestion batch, whichever is more frequent. The MS contains all target elements of interest.

The acceptable % recovery QC limits are 80% – 120%. The % recovery QC limits are continuously monitored and documented in-house through control charts. The *Control Limit Generation* SOP (1734) provides details explaining how control charts are generated and used for quality control.

Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits and the LCS is compliant, include a project narrative with the results to the client noting that there may be potential matrix effects on the accuracy of the reported results as evidenced by MS recovery outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses (matrix or sample duplicate) must be performed once per 20 samples 5% frequency), or per digestion batch, whichever is more frequent.

Acceptable relative percent differences (RPD) of duplicates is $\leq$ 20%. This acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = sample Replicate #1
R2 = sample Replicate #2

The RPD limits are continuously monitored and documented in-house through control charts. The *Control Limit Generation* SOP (1734) provides details explaining how control charts are generated and used for quality control.

Corrective Action: Analysis may be repeated once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported metals results as evidenced by the matrix duplicate %RPD exceedance.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- ICV
- ICB
- Method Blank
- Laboratory Control Sample
- Matrix QC Sample

- Matrix Duplicate
- Matrix Spike
- Samples 2-5
- CCV
- CCB
- Samples 6-15
- CCV
- CCB

10. Procedure

10.1 Equipment Set-up

Samples are prioritized by the Metals Department Manager or Preparation Group Leader for digestion based on hold time and client due date. The analyst must be familiar with instrument software and hardware as described in the user manuals supplied with the instrument before attempting to perform an analysis.

Power to the instrument, autosampler , PC and the argon gas supply are turned on. Peristaltic pump windings are inspected for wear and replaced if necessary. The winding clips are relaxed in between usage. The instrument manufacturer suggests that the pump tubing be changed once per month or more frequently as needed.

The stannous chloride solution is attached to the grey/grey pump tubing, 10% HCl is attached to the green/green autosampler rinse and DI is attached to the geen/green rinse blank. The autosampler probe line is attached to a grey/grey pump tubing. The pumps are turned on via the software and the flow is checked to insure that solutions are delivered to the mixing valve.

10.1.1 Sample Preparation

10.1.1.1 Tissues

Tissue samples are prepared by microwave oven assisted acid digestion according to SOP 2150. The LCS and MS are spiked as indicated in the table in 8.10.3.

A 10 mL aliquot of each digest is added to a 50 mL screw cap digestion tube along with 2 mL of BrCl solution. The vial contents should remain a yellow color. If a yellow color does not remain, add more BrCl solution. This solution is allowed to stand for 12 hours. Bring the vial to 50 mL with DI water . The vials are then decolorized with 0.4 mL of 12% hydroxyl amine hydrochloride and shaken. The yellow color should disappear. If the solution remains yellow, add more hydroxyl amine until the color disappears. Add the same amount to all samples and standards.

10.1.1.2 Soils/Sediment

A 0.5 – 1.0 g wet weight well mixed aliquot of soil/sediment is added to a 50 mL screw cap digestion tube with 5 mL of DI water. The LCS and MS are spiked as

indicated in the table in 8.10.3. A 3.75 mL volume of concentrated HCl and 1.25 mL of concentrated HNO_3 are added and the tube placed on the hot block set to 95 ± 3 ^{o}C for 2 minutes. Samples are allowed to cool and then 25 mL of DI water is added to each vial and placed back on the hot block for 30 minutes.

After cooling bring to a final volume of 50 mL with DI water. Then a 10 mL aliquot of each digest is added to a 50 mL screw cap digestion tube along 2 mL of BrCl solution. The vial contents should remain a yellow color. If a yellow color does not remain, add more BrCl solution. Allow samples to react overnight. The vials are then decolorized with 0.4 mL of 12% hydroxyl amine hydrochloride, brought to a final volume of 50 mL and shaken. The yellow color should disappear. If the solution remains yellow, add more hydroxyl amine until the color disappears. Add the same amount to all samples and standards.

10.2 Initial Calibration

The instrument is calibrated prior to sample analysis and at least once every 24 hours. A linear least squares regression calibration curve is selected for Method 7474.

In all cases, a correlation coefficient ≥ 0.995 is required before the analysis of samples can begin. The calibration blank is included as a point in the calibration curve.

An ICV (Section 9.3) and ICB (Section 9.1.2) are analyzed after calibration.

10.3 Equipment Operation and Sample Processing

After initializing the Millennium software, the instrument is turned on via the software and allowed to warm up for 30 minutes. The analysis method is selected and the autosampler template is selected. Method 7474 is set up in the software which specifies delay, read and rinse times as well as pump speeds. An autosampler template is set up specifying calibration standard, QC standards and positions on the autosampler rack. Once sample ID's have been typed in, the file is saved as the Job number.

A Work Group is obtained from LIMS and used as the data file name.

The analysis is initiated by clicking on the start button on the analysis page.

Following analysis, the data file is exported to the LIMS on the O:Metals\Millennium drive for data processing. The instrument and autosampler are then rinsed with DI water and turned off

10.4 Continuing Calibration

Periodically (after no more than 10 samples) and at the end of the analysis, CCV standards (Section 9.4) and CCB standards (Section 9.1.3) are analyzed.

10.5 Preventive Maintenance

Inspect pump tubing for signs of wear and replace monthly or more frequently if necessary.

Clean glass liquid separator when glass appears coated with yellow film.

Replace dryer tube annually.

11. Data Evaluation, Calculations and Reporting

Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP (1729). All records must be stored in such a manner as to be safe and accessible for at least 10 years.

The digestion batch bench sheets and other relevant laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* Work Instruction (1556), and all record keeping and document control practices.

Sample concentrations are calculated as follows:

Sample Concentration = ((Dc*Df)*Dv)/((Md*(S%/100)*1000))

Where:

Dc = digest Concentration in µg/L
Df = Dilution factor
Dv = Digest volume in mL
Md = Mass of sample digested in g
S% = percent solid of the sample (if reported on wet weight basis then percent solid = 100)

All results are reportable without qualification if digestion and analytical holding times are met, preservation (including cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference is not suspected during digestion or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis

QC Parameter	Acceptance Criteria
Method Blank	< reporting limit
Laboratory Control Sample	80%-120% Recovery
Matrix Duplicate	≤ 20% RPD
Matrix Spike	80% - 120% Recovery
Matrix Spike Duplicate (if needed)	≤ 20% RPD, 80% - 120% Recovery

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 outlines sample batch QC acceptance criteria. If non-compliant results are to be reported, the Metals Department Manager and/or the Laboratory Director, and the QA Manager must approve the reporting of the results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The instrument analyst or Department Manager performing the secondary analytical review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method

13.3 Instrument Detection Limits

The instrument detection limit (IDL) is the smallest signal above the background noise that an instrument can detect. The IDL can be calculated taking the average of the standard deviations for three measurements of a reagent blank solution analyzed on three analytical runs on three non-consecutive days. Seven consecutive measurements must be taken per day. The IDLs are

not required to go through sample digestion. Each measurement must be performed as though it were a separate sample.

14. Pollution Prevention and Waste Management

The *Hazardous Waste and Sample Disposal* SOP (1797) must be referenced for disposal of used standards, solvents, acids, reagents or other chemicals.

Once sample batches have completed digestion, the sample containers are stored in the metals lab and held for 30 days. If there is no sample remaining in the sample collection bottle, it may be rinsed and thrown away. It must be noted in the *Internal COC* that there is no sample remaining in the bottle, and the bottle was discarded.

Once satisfactory results have been generated, the digestates are held for 30 days, or longer if specified by a client contract, then discarded into a 55-gallon drum labeled “Corrosive Liquid”.

All reagent waste generated during digestion must be stored in satellite containers in the metals preparation laboratory.

Once the reagent waste satellite containers are full, they must be emptied into 55-gallon drums marked “Corrosive Liquid”.

Refer to Alpha’s Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1559 Sample Receipt & Log-In

SOP/1729 Document Control

SOP/1732 DL/LOD/LOQ Generation

SOP/1734 Control Limit Generation

SOP/1739 Demonstration of Capability Generation

SOP/1797 Hazardous Waste and Sample Disposal

SOP/2150 Microwave Assisted Acid Digestion of Sediments, Soils, Tissues and Waters

WI/1556 Laboratory Notebook Usage

16. Attachments

None.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

THE ANALYSIS OF POLYCHLORINATED BIPHENYLS (PCBs)

BY

HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

(CF-OA-E-003)

APPLICABLE TO METHOD:
EPA Method 1668A and C

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W. M. Larkins

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1.0 STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF POLYCHLORINATED BIPHENYLS (PCB) BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

This standard operating procedure (SOP) covers the analytical determination of PCBs according to EPA Method 1668, Revisions A & C.

3.0 APPLICABLE MATRICES

Applicable matrices include groundwater, wastewater, surface water, leachate, soil, sediment, sludge, oil, and tissue.

4.0 METHOD SCOPE, APPLICABILITY AND DETECTION LIMIT

4.1 Method 1668 may be used to quantify PCBs that are soluble in methylene chloride and/or toluene. The compounds are separated using a gas chromatograph (GC) and detected using a high-resolution double focusing mass spectrometer (HRMS). Appendix 1 lists the analytes currently analyzed using these methods and their practical quantitation limits.

4.2 The practical quantitation limit (PQL) is the lowest level in the calibration curve. The PQL is the lowest level at which compounds may be accurately quantitated and is compound dependent. The calibration curve typically ranges from 0.5 ng/mL to 2000 ng/mL. These ranges reflect instrument readings, which are in ng/mL (ppb). It should be noted that the calibration range may vary between calibrations and instruments.

4.3 Method detection limit studies (MDLs) are performed and/or verified on an annual basis. MDLs are done for aqueous, solid, and tissue matrices. For more information regarding MDLs, refer to The Determination of Method Detection Limits, CF-LB-E-001.

4.4 Qualified analysts must demonstrate proficiency initially and annually thereafter with an IDOC, CDOC or PT study. Acceptability criteria may be found in the analytical method.

4.4.1 To establish the ability to generate acceptable accuracy and precision, the analyst should perform an "analyst validation study" or Initial Demonstration of Capability. Four LCS standards are extracted and analyzed. Calculate the average recovery and the standard deviation of the recovery for each analyte of interest using the four results. Then compare the average and the standard deviation with the corresponding criteria found in Table 6 of Method 1668A or C as appropriate. If the average and the standard deviation for all analytes of interest meet the acceptance criteria, then the analyst may begin work on actual samples. If the validation study fails for one or more of the compounds, then the study must be repeated for those compounds which failed.

5.0 METHOD VARIATIONS

5.1 Sample extracts are stored at room temperature to avoid analyte loss. Many of the target analytes in these methods form a strong cohesive bond with solids such as glass in cold temperatures; this type of analyte loss is not addressed in the method. (This is a variance from the following method recommendations: -10° to -20° C per DoD QSM.)

5.2 The analytical method does not address the reporting of EDL and EMPC. These values are reported only when requested by the client.

5.3 Project specific modifications are allowed to meet the data quality objectives of the Delaware River Basin Commission.

6.0 DEFINITIONS

6.1 Accuracy: The degree of agreement between an observed value and an accepted reference value.

6.2 AlphaLIMS: The Laboratory Information Management System used at CFA, LLC.

6.3 Blank: An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and standard additions that are used with other samples. The LMB (Lab Method Blank) is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Contamination may be derived during sampling, transportation, storage or analysis. The blank may be used to establish a background value.

6.4 Calibration Standard (CAL): An aliquot of a primary standard solution or stock standard solution. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.5 Calibration Verification Standard (CVS, CCAL): A solution of target analytes with a concentration near the mid-point of the calibration range. It should be obtained from a second source vendor and is used to verify the initial calibration on a basis described in the determinative method.

6.6 Cleanup Standards: Isotopes added prior to cleanup that are used to measure the efficiency of the fractionation step alone. Method 1668 uses three compounds as the Cleanup Standards.

6.7 Duplicate Analysis: The analysis or measurement of the variable of interest performed identically on two field subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision of sample, preservation, or storage internal to the laboratory.

6.8 Estimated Detection Limit (EDL): A calculation of the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. The EDL is calculated for each congener that is not identified.

6.9 Estimated Maximum Possible Concentration (EMPC): A calculation for a peak characterized by a response with a signal-to-noise ratio of at least 2.5 for both the quantitation ions, and meeting all identification criteria except ion ratio. EMPC is a worst-case estimate of the concentration.

6.10 Extraction Standards: Isotopes added prior to extraction that serve as internal standards for many PCB congeners. In addition, to measure the overall extraction and fractionation efficiencies. Method 1668 names them Labeled Compounds.

6.11 Injection Standards: Isotopes added prior to injection to determine the recoveries of the Extraction and Cleanup Standards. Method 1668 calls them Internal Standards.

6.12 Internal Standard (ISTD): A known amount of standard added to a test portion of a sample as a reference for evaluating the retention time and concentration of dependent analytes and controlling the precision and bias of the applied analytical method.

6.13 Laboratory Control Standard/Duplicate (LCS/LCSD): Aliquots of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS/LCSD are analyzed exactly like a sample, and the purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

6.14 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

6.15 Matrix Spike and Matrix Spike Duplicate (MS and MSD): Two separate aliquots of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS and MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results. The concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD adjusted. Percent recovery is calculated for both aliquots, and RPD is calculated between the two.

6.16 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

6.17 Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves, a data quality indicator. Precision is usually expressed as standard deviation, variance or range in either absolute or relative terms.

6.18 Quantitation Limits (also PQL, RL): The value at which an instrument can accurately measure an analyte at a specific concentration (i.e., a specific numeric concentration can be quantified). These points are established by the upper and lower limits of the linear calibration range.

7.0 INTERFERENCES/LIMITATIONS

7.1 Contaminants found in extraction glassware, solvents, and other sample processing hardware may jeopardize the integrity of this method.

7.2 Glassware must be scrupulously cleaned as soon as possible after extraction.

7.3 Contamination may also occur in the GC/MS system. High boiling materials tend to build up in the injection port and the front end of the column. The analyst should maintain a thorough working knowledge of keeping the injection port free of contamination, including changing out the septum, injection port liner, O-ring, ferrule, and gold seal.

7.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. If carryover is suspected, potentially impacted samples should be re-analyzed after any needed maintenance, solvent replacement, and/or cleaning has been done.

8.0 SAFETY PRECAUTIONS AND WARNINGS

METHYLENE CHLORIDE IS A SUSPECTED CARCINOGEN AND A KNOWN SKIN IRRITANT.

PCBs HAVE BEEN TENTATIVELY CLASSIFIED AS KNOWN OR SUSPECTED MAMMALIAN CARCINOGENS.

CONTACT WITH OXIDIZERS MAY GENERATE EXPLOSIVE MIXTURES.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS FROM SOLIDS.

8.1 Eye protection should be worn when handling samples, reagents, or standards. NOTE: Contact lenses pose a special problem; soft lenses may absorb irritants and all lenses concentrate them. DO NOT wear contact lenses in the laboratory.

8.2 Treat all chemicals and samples as potential health hazards and reduce exposure to these chemicals to the lowest level possible. CFA maintains a reference file of Material Safety Data Sheets (MSDS) for each chemical and standard. These documents and individual sample MSDS provided by clients are maintained in the laboratory.

8.3 Personal Protective Equipment (PPE)

8.3.1 Gloves and eye protection should be worn when handling reagents, solvents, standards and samples.

8.3.2 Analysts should prepare samples and standards under the hood.

8.4 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.5 Never leave gas cylinders unchained or untied.

8.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, management should be notified of all accidents.

8.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers and fire blankets are located in the lab. Training is available on the proper operation of this equipment.

8.8 The analyst must use care when assembling and operating instrumentation. Check to see that the gas chromatograph equipment is properly assembled and hooked up to the proper gas cylinder and power, referencing the appropriate manual. Analytical equipment must only be operated by qualified personnel.

8.9 For further safety instructions, consult the Safety, Health and Chemical Hygiene Plan, CF-LB-N-001.

9.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION

9.1 Equipment associated with this method includes:

9.1.1 Gas tight syringes

9.1.2 2 mL high recovery (conical) autosampler vials and storage racks

9.1.3 Teflon crimp tops

9.1.4 Crimper/De-crimper

9.1.5 GC Column (SPB-Octyl or equivalent; 30m, 0.25 mm, 0.25 um)

9.1.6 Quartz/Glass injection port liners

9.1.7 Injection port liner O-ring seals

9.1.8 Gold seals

9.1.9 Ferrules

9.1.10 Column cleaving tool

9.1.11 Septa (thermogreen)

9.1.12 0.5-10 uL adjustable air displacement pipette with disposable tips

9.2 Instrumentation

9.2.1 Waters Autospec Premier high resolution mass spectrometer

9.2.2 Agilent 7890 Gas Chromatograph

9.2.2.1 A suggested temperature program follows:

Temperature 1	150° C
Time 1	2.0 min.
Rate	5° C/min.
Temperature 2	180° C
Time 2	1.5°/min
Final Temperature	265° C
Run Time:	55 minutes (may vary due to column length or flow rate)
Solvent Delay:	6.0 min.
Splitless Valve Time:	2.0 min.
Flow:	1.0 mL/min.
Mass Range:	See descriptor definitions (Table 2)

NOTE: These instrument conditions and rates are guidelines which may change.

9.2.3 LEAP Technologies GC PAL Autosampler

9.2.3.1 Suggested parameters:

Sample volume – 1 µL
Air volume – 0.5 µL
Solvent push volume – 1 µL
Number of sample washes - 0
Solvent washes - 30
Sample viscosity wait – 1 second
Number of sample pumps - 0
Injection mode - Fast

10.0 REAGENTS AND STANDARDS

10.1 Reagents and standards

10.1.1 Nonane

10.1.2 Source Standards: Source Standards are purchased directly from vendors and may be diluted to make stock, intermediate, or working standards. These may include extraction standard, matrix spiking standard, cleanup standard, injection standard, as well as others. Source standards expire per the vendor expiration date or after five years from the date opened, whichever is shorter. Please reference CF-LB-E-007 for further information regarding standards and their preparation.

10.1.3 Initial Calibration (ICAL) Standards: Certified calibration standards are purchased from commercial vendors at a minimum of five concentration levels. One of the calibration standards is at a concentration near, but above, the method detection limit; the others should correspond to the expected range of compounds found in samples. Calibration standards expire after a maximum of five years and should be monitored frequently for signs of degradation.

10.1.4 Calibration Verification Standards (CVS, CCAL, CS3): A certified CVS is purchased from a second source commercial vendor at a concentration that is near to the midpoint of the calibration curve.

10.1.5 Window Defining Mix and Column Performance Mix (WDM and CPM): A standard containing the first and last eluters for each homolog group, as well as the isomers used to demonstrate isomer specificity on the GC column in use. Usually contained in the daily 209 injection mix.

11.0 SAMPLE HANDLING AND PRESERVATION

11.1 Samples have a one year holding time from the date of collection, and a one year holding time from the date of extraction.

11.2 Sample extracts are delivered from the prep lab to the instrument lab and are stored in a darkened hood at room temperature. The extracts are usually grouped according to batches and are accompanied by the batch pull sheet and other pertinent paperwork.

11.3 Custody of samples is monitored using the AlphaLIMS sample tracking system. Each analyst should scan the samples planned to run into their custody prior to analysis.

11.4 All sample extracts should be treated with caution as potential health hazards. Refer to Section 8.0 on safety.

12.0 SAMPLE PREPARATION

12.1 Before extracts can be analyzed on the instrument, they must first be evaporated to 18 uL nonane (added after cleanup steps) under nitrogen and then spiked with injection standard to set the final volume nominally at 20 µL. A determination must also be made as to whether the extract should be diluted. The decision to dilute a sample extract is based on a number of factors: sample screening, historical data about the sample or sample site, the appearance of the extract (color, viscosity, incidental odor, turbidity, etc.), or regulatory considerations. The experience of the analyst is invaluable in making this determination.

NOTE: Sample extracts may contain multiple layers or sediment. Samples that contain sediment are returned to cleanup. Multiple layers are treated on a case-by-case basis. If the extract can be homogenized, then a uniform sample is achieved. If the extract remains bi-phasic, the PM and client are contacted for further guidance.

12.2 If a sample is to be analyzed without dilution (‘neat’), 2 nanograms of injection standard solution is added to the extract using a pipette (2 µL of a 1.0 ng/µL = 100 pg/µL extract concentration). A cap is then placed on the vial and secured by crimping before vortexing the sample to ensure complete mixing and vial wall washing.

12.3 If samples require dilution, the dilution is made using nonane or appropriate solvent. If not previously added, 2 nanograms of JS is added to the autosampler vial. Dilution prep may involve the addition of supplemental extraction standard (ES) and is documented in the injection prep logbook.

12.4 Once samples are prepped, they are ready to be injected onto the instrument. An autosampler is used to inject standards and sample extracts on the instrument.

12.5 The need for dilution may also be determined after analysis is performed, and may still be performed as above. Under normal circumstances, a sample would be diluted if any chromatographic peaks saturate the detector.

13.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

Typically a blank (LMB), laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) are extracted and analyzed with each prep batch. Other client requirements may include a matrix spike (MS) and matrix spike duplicate (MSD) or sample duplicate (DUP).

13.1 Blanks

13.1.1 A blank is extracted with each batch of 20 or fewer samples to demonstrate that interferences from glassware, reagents and the analytical system are under control. Blanks are carried through all stages of sample preparation and analysis. An acceptable blank must be below the minimum levels listed in Table 2 of the method for all analytes.

13.1.2 The percent recovery of each labeled standard (extraction and cleanup) is calculated as shown in Sec. 19.1.3. Recoveries must be within the limits in Table 5.

13.2 Laboratory Control Samples and Matrix Spikes

13.2.1 The spiking standard for LCS/LCSDs and MS/MSDs contains all analytes listed in Table 4. For each LCS, LCSD, MS and MSD, the concentration of each analyte and its percent recovery are calculated as shown in Sec.19.1.1 and 19.1.3. Recovered concentrations should be within the limits in Table 5.

13.2.2 If recovery is not within these limits, the data may need to be re-checked for errors, or the samples and QC may need to be re-analyzed. In addition, the instrumentation may need to be checked for performance problems. If the LCS fails to meet acceptance criteria due to low recovery, the associated samples may have to be re-extracted and re-analyzed when possible. If one or more recoveries are high in the LCS and these analytes are not detected in the samples, the event should be documented and data may be reported. If the MS and MSD both fail due to matrix interference and/or dilution, data may be reported provided the associated LCS passes acceptance criteria.

NOTE: Many clients have contract specific criteria that must be considered when evaluating recovery of the Quality Control samples.

13.2.3 The percent recovery of each labeled standard (extraction and cleanup) is calculated as shown in Sec. 19.1.3. Recoveries must be within the limits in Table 5.

13.3 Samples

13.3.1 The percent recovery of each labeled standard (as outlined in SOP CF-OA-E-001) is calculated as shown in Sec. 19.1.3. Recoveries must be within the limits in Table 5.

13.3.2 Calculated EDLs should be below the PQLs in Table 1. Any reported EDLs above the PQLs should be noted in the case narrative.

14.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE

14.1 Mass spectrometer performance

14.1.1 The mass spectrometer is operated in electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Static resolving power checks must be performed at the beginning and at the end of each 12-hr period of operation. Corrective action must be implemented whenever the resolving power does not meet the requirement.

14.1.1.1 Chromatography time for PCBs exceeds the long term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass drift correction is mandatory. A lock-mass ion from the reference compound PFK is used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Lock mass ions may be found in the descriptor table, Table 2. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full scale deflection for a given set of detector parameters. Under these conditions, sensitivity changes that might occur during the analysis can be more effectively monitored. NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in an increase in downtime for source cleaning.

14.1.2 Documentation of the instrument resolving power must be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760) obtained during the above peak matching experiment by using the low mass PFK ion at m/z 304.9824 as a reference. The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10 percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

14.2 System Performance

System performance criteria are presented below. The laboratory may use the recommended GC column described in Sec. 9.1.5. The laboratory must document that all applicable system performance criteria were met before sample analysis begins. Sec. 9.2.2.1 provides recommended GC conditions that may be used to satisfy the required criteria. A GC column performance check is required at the beginning of each

12-hr period during which samples are analyzed. A continuing calibration must be performed at the beginning of the sequence.

14.2.1 Daily 209 injection mix

14.2.1.1 Inject 1 uL of the daily 209 injection mix solution (Sec. 10.1.6) and acquire selected ion monitoring (SIM) data within a total cycle time of $\leq$ 1.5 second. The chromatographic separation between congeners 34-TrCB and 23-TrCB, and between congeners 187-HxCB and 182-HxCB, must be resolved with a valley of $\leq$ 40 percent, where:

Valley percent = (x/y) × 100

x = the height of the valley

y = the peak height of the shorter of the two peaks

Congeners 156-HxCB and 157-HxCB must also co-elute within 2 seconds.

14.2.1.2 The daily 209 injection mix contains all 209 PCB congeners, and may thus be used to identify the first and last PCB eluters in each congener group, and in each analytical descriptor. Their retention times are used to determine the seven homologue retention time windows that are used for qualitative (Sec. 15.4.1.1) and quantitative purposes.

14.3 Initial Calibration

14.3.1 Prior to running a multi-level calibration, take precautions to ensure that the instrument meets system performance criteria. The analyst must document that all system performance criteria are met before analyzing an initial calibration.

14.3.2 Initial calibration is required before any samples are analyzed for PCBs and must meet the acceptance criteria in this section. Initial calibration is also required if any routine calibration does not meet the required criteria listed in Sec. 15.3, and at a minimum, annually.

14.3.3 At a minimum, all five high-resolution concentration calibration solutions listed in Table 4 must be used for the initial calibration. A lower calibration point of 0.5pg/ul is used for DRBC.

14.3.4 Tune the instrument with PFK to meet the above-specified system performance criteria.

14.3.5 Inject the daily 209 injection mix solution and acquire SIM mass spectral data. The laboratory must not perform any further analysis until it is demonstrated and documented that the criteria listed in Sec. 14.2.1 are met.

14.3.6 By using the same conditions (GC and MS) that produced acceptable results with the 209 injection mix solution, analyze each of the five concentration calibration solutions. Each injection must meet the following ion ratio and signal-to-noise (S/N) requirements:

14.3.6.1 The ratio of integrated ion current for the ions appearing in Table 2 (homologous series quantitation ions) must be within the indicated control limits (set for each homologous series) in Table 3. These ion ratio requirements must be within the specified control limits

simultaneously in one run. It is the analyst's responsibility to take corrective action if the ion abundance ratios are outside the limits.

14.3.6.2 For each selected ion current profile (SICP) and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the S/N ratio must be better than or equal to 10. Manual measurement of S/N is required for any GC peak that has an apparent S/N of less than 15:1. The result of the measurement must appear on the SICP above the GC peak in question. NOTE: An interference with PFK m/z 223.9872 may preclude meeting this requirement for DiCB congeners. Suspected column bleed interference may preclude meeting this requirement in other congeners groups. These interferences are noted in the low calibration levels, CS1 and below. If interference occurs, 10:1 S/N must be met at the CS2 level. The lower calibration points are still included in the initial calibration.

14.3.6.3 Manual integrations, if required, are performed and documented according to SOP CF-LB-E-017.

14.3.7 Calculate the relative response factors (RF) for unlabeled target analytes relative to their appropriate internal standards. Also calculate the RFs for the ESs and CSs relative to the appropriate injection standards according to the following formula:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

Ax = Sum of the Areas of the two characteristic ions for the compound being measured.

Ais = Sum of the Areas of the two characteristic ions for the specific internal standard.

Cis = Concentration of the specific internal standard.

Cx = Concentration of the compound being measured.

The RF is a dimensionless quantity; the units used to express Cis and Cx must be the same.

14.3.8 Because more than five calibration levels may be analyzed, the analyst may choose to deactivate one or more levels globally. If a level is not used, it will be deactivated in the method for all analytes in that calibration mixture. In some cases the upper level(s) of the calibration may be deactivated in order to meet method criteria for single compounds. This practice results in a narrower calibration range. Hits detected above the highest calibration level are re-analyzed at a dilution which brings the analytes into the calibration range. The low standard representing the PQL is not dropped. Please note that this practice does not represent "cherry picking," which is acknowledged as an unacceptable laboratory practice.

14.3.9 The average RF must be calculated for each compound as follows:

$$RF_{avg} = \frac{\sum_{i=1}^{n} X}{n}$$

Where:

N = number of calibration levels

X_i; i=1 to n, are the compounds RF values for each calibration point

14.3.10 Criteria for acceptable initial calibration

The criteria listed below for acceptable calibration must be met before sample analyses are performed.

14.3.10.1 The percent relative standard deviations for the mean response factors from the unlabeled standards must not exceed ±20 percent.

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

Where:

RSD = relative standard deviation

$\bar{x}$ = mean of 5 or more initial RFs for a compound

SD = standard deviation of average RFs for a compound

$$SD = \sqrt[2]{\frac{\sum_{i=1}^{n}(X - A)}{n-1}}$$

where:

n = number of calibration levels

X_i; i=1 to n, are the compounds RF values for each calibration point

A = average of the RFs from above

14.3.10.2 The percent relative standard deviations for the mean response factors from the labeled standards must not exceed ±30 percent.

15.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION

15.1 Resolution check

15.1.1 At the beginning and end of each 12-hour window, mass resolution must be tuned and/or verified. A static resolving power of at least 10,000 must be demonstrated at appropriate masses before analysis is performed.

15.1.2 Using a PFK molecular leak, tune the instrument to the minimum required resolving power of 10,000 at m/z 330.9792 (for day to day operations, the instrument may be tuned to approximately 11,000). Verify that the exact mass of m/z 380.9760 is within 5 ppm of the required value. Due to the wide mass ranges required for PCB analysis, resolution may drop to 8000 at either end of the descriptor, but should be >10,000 at the midpoint.

15.2 Daily 209 injection mix

Inject 1 uL of the daily 209 mix. Verify that all column performance and window defining criteria in Section 14.2.1 have been met.

15.3 Continuing Calibration

15.3.1 The initial calibration curve for each compound of interest must be verified once every 12 hours. Inject 1 uL of the CS3 standard. For 1668C, the Daily 209 injection mix is used to satisfy the calibration verification criteria.

Calculate the percent difference using:

$$\% \text{ Difference} = \frac{\left|\overline{RF}_i - RF_c\right|}{\overline{RF}_i} \times 100$$

Where:

$\overline{RF}_i$ = average response factor from initial calibration

RF_C = response factor from current CS3

15.3.1.1 For Method 1668A, if the percent difference for each native analyte is $\leq$ 30%, and for each labeled analyte is $\leq$ 50%, the calibration is assumed to be valid. Method 1668C criteria are listed in Table 7. If the criteria are not met, corrective action should be taken. If no source of the problem can be determined after corrective action has been taken, a new initial calibration may need to be generated.

15.3.1.2 All ion ratios must be within the limits in Table 3.

15.4 Sample Analysis

15.4.1 Data Interpretation

15.4.1.1 Qualitative Analysis

For a peak to be identified as a PCB, it must meet all of the criteria listed below.

15.4.1.1.1 The signals for the two m/z's being monitored must be present and maximize within the same two scans.

15.4.1.1.2 The signal-to-noise ratio between the two m/z's must be $\geq$ 2.5 for each PCB detected in a sample extract, and $\geq$10 for all PCBs in the calibration and verification standards. (DiCBs suffer from PFK interference and are not required to meet 10:1 until the CS2. Other PCBs suffer from suspected column bleed interference and are only required to meet 2.5:1 in calibration points below the CS1.)

15.4.1.1.3 Ion ratios must be within the limits in Table 3.

15.4.1.1.4 The relative retention time of the peak for a PCB must be within the RRT limits calculated as specified in Table 8 based on the RT limits provided.

15.4.1.1.5 Congener overlap, interfering substances, or the loss of chlorine from a higher chlorinated congener may make it difficult to meet all identification criteria. In these cases, an experienced spectrometrist must determine the presence or absence of the congener.

15.4.1.2 Calibration Limit Exceedance

15.4.1.2.1 If a compound in a sample exceeds the upper calibration limit, all subsequent samples must be checked for carryover contamination.

15.4.1.2.2 When a subsequent sample is non-detect for the compound in question, the sequence is again considered acceptable for reporting.

15.4.1.2.3 All affected samples between the exceeding sample and the non-detect sample must be re-analyzed.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

16.1 Preventive maintenance on a HRGC/HRMS system involves the following basic areas:

16.1.1 Vacuum pumps for the inlets, source, and analyzer need a change of oil about every year or when system performance indicates it is needed.

16.1.2 The GC injection port is cleaned as needed, approximately once a week. It is recommended that the septum and injection port liner be replaced at the time of cleaning. Additionally, the gold plated seal should be cleaned or replaced.

16.1.3 Ion source maintenance is usage dependent. The type and quantity of samples that have been injected determine the frequency of ion source cleaning and filament replacement.

16.1.4 Autosampler maintenance is primarily that of cleanliness. Most autosamplers need their moving parts to be clean and lightly lubricated. The most frequent corrective maintenance is that of changing the syringe, usually about once per month.

16.1.5 Instrument maintenance logs are kept with each instrument and serve as a record of all the maintenance that has been done on the instrument.

16.2 Non-Routine Maintenance Procedures (Special, Operational or Failure Mode Maintenance)

16.2.1 Service is provided to the instrument via the analyst, the in-house instrument service engineer, or a technical support specialist from the manufacturer. When instrument failure occurs, different parts of the instrument are isolated to determine the root cause. For example, the injection port may be capped off if a leak is suspected to prove the leak is/is not coming from that source. Instrument maintenance logbooks are kept for each instrument detailing the type of maintenance performed on the instrument and when it was performed. Preventive maintenance visits are scheduled annually for the mass spectrometers.

16.2.2 Analytical GC columns are clipped or replaced when the existing column shows signs of excessive degradation or the inability to properly resolve chromatographic peaks. Excessive peak tailing, poor responses, and baseline disturbances may also indicate that the column needs to be replaced.

17.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS

17.1 Data are evaluated qualitatively and quantitatively using a software program such as Waters MassLynx, or equivalent data system.

17.2 Data are reviewed, and a hard copy is generated. If manual integrations are made, a hard copy of the manual integration is printed and initialed by the analyst and included with the raw data.

17.3 Additional supporting documentation, such as totals pages generated by the software may be included with the data.

17.4 Quantitative Analysis

17.4.1 The concentration (ng/L for aqueous, ng/g for solids) of each identified compound in the sample is calculated as follows:

$$[PCB] = \frac{\left(A_{unk}^{ion1} + A_{unk}^{ion2}\right)}{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)} \times \frac{Q_{ES}}{W_{unk} \times D \times \overline{RF}}$$

Where:

A_{unk} and A_{ES} = the integrated area for each ion monitored.
Q_{ES} = the amount of extraction standard added to the sample in nanograms
W_{unk} = the initial sample aliquot size, in liters for waters and in grams for solids.
D = (% moisture in sample)/100, or 1 for waters
$\overline{RF}$ = Average RF from the ICAL for the compound

17.4.2 The concentration of each extraction and cleanup standard is calculated as follows:

$$[ES_{ng}] = \frac{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)}{\left(A_{JS}^{ion1} + A_{JS}^{ion2}\right)} \times \frac{Q_{JS}}{\overline{RF}}$$

Where:

A_{ES} and A_{JS} = the integrated area for each ion monitored.
Q_{JS} = the amount of injection standard added to the sample in nanograms
$\overline{RF}$ = Average RF from the ICAL for the compound

The cleanup standard concentration is calculated as above, substituting the area of the individual cleanup standard ions for the extraction standard ions.

17.4.3 Percent recovery is calculated as follows:

$$\%R = \frac{R_{ng}}{S_{ng}} \times 100$$

Where:

R_{ng} = the amount of standard recovered in nanograms.
S_{ng} = the amount of standard spiked in nanograms.

18.0 POLLUTION/CONTAMINATION

18.1 Work area should be maintained free of dust and dirt accumulations.

18.2 Fume hoods are utilized to remove fumes and reduce the risk of airborne contaminants to ensure personnel safety. Hoods are monitored in accordance with CF-FC-E-003 for Fume Hood Face Velocity Performance Checks.

18.3 The laboratory area is restricted to authorized personnel.

19.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

19.1 A review process is used to insure the quality of the data. Raw data are reviewed first by the analyst, then by a second (peer) analyst or a data validator. When the analyst is satisfied that the data have been correctly processed and uploaded to the LIMS, a data report is generated from AlphaLIMS. The AlphaLIMS report along with the raw data and supporting documentation, such as a run log and case narrative, are submitted for review to the data validator or another experienced analyst. The reviewer goes through the raw data as if he/she was working it up for the first time and verifies that they are correct. In addition, he/she must make sure that the data have been correctly entered into AlphaLIMS. AlphaLIMS reports may be self-reviewed. If errors are discovered in either the raw data or the AlphaLIMS report, then the two analysts should discuss the differences and how best to resolve them. In some cases, the peer review process may uncover errors that lead to a sample being re-extracted or re-run. In cases such as these, a nonconformance report (NCR) should be completed and submitted to the Quality department. It is recommended that a copy of the NCR be given to the prep analyst if it involves a re-extraction and that a copy be kept with the original data.

19.2 Once the data review has been completed by the reviewer, the batch is returned to the analyst for corrections (if applicable) and the status is updated from REVW to DONE in AlphaLIMS.

19.3 Data may be transmitted automatically to AlphaLIMS. This automatic "upload" procedure may be activated prior to data review or after data review is complete. In either case, the data recorded in AlphaLIMS are checked by the analyst for accuracy and completeness.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data may require instrument maintenance, re-analysis, re-extraction, or a more complex set of actions. When troubleshooting measures fail to bring an analytical process or data into control, a nonconformance report and/or corrective action should be initiated in accordance with CF-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items, and CF-QS-E-002 for Conducting Corrective Action.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting is used to determine the appropriate action to take when an initial or continuing calibration, blank and/or laboratory control sample fails to meet the acceptance criteria defined for the method. Troubleshooting may involve one or more of the following actions:

21.1 If analytes in a multi-point calibration fail to meet specified criteria, additional standards for the failing compounds may need to be reanalyzed. If they still do not meet specifications, instrument maintenance or new standards may be required before work is continued.

21.2 If a continuing calibration fails to meet specified criteria, instrument tuning or inlet maintenance may be required. If these attempts fail, a new initial calibration must be analyzed.

21.3 If a method blank fails to meet defined criteria, the source of contamination should be found and eliminated before proceeding with analysis.

21.4 If a LCS fails to meet specified criteria, the process should be reviewed to identify potential areas for failure. Any problem areas should be corrected before re-extraction or re-analysis is performed.

21.5 If a sample fails to meet recovery or detection limit criteria, the data should be reviewed to determine whether matrix interferences are present, and if further cleanup or a smaller volume extraction might improve recoveries.

22.0 RECORDS MANAGEMENT

22.1 Run logs are generated for each instrument each day that the instrument is run. These run logs serve as records of what is run on the instrument, including samples, QC, calibrations, tunes, etc. Additional information is provided in the run log, including the analyst's initials, run date and time, and file name.

22.2 Raw data are stored in the lab in filing cabinets and/or boxes as long as there is space available. When space runs out, the data are boxed and sent to storage.

22.3 All records generated as a result of this procedure are maintained as quality documents in accordance with CF-QS-E-008 for Quality Records Management and Disposition.

23.0 LABORATORY WASTE HANDLING AND DISPOSAL

Sample extracts that have been run are temporarily stored in case they have to be reanalyzed. Once space is no longer available to keep them in the lab, they are moved to Waste Disposal where they are handled and disposed in accordance with the Laboratory Waste Management Plan, CF-LB-G-001.

24.0 REFERENCES

Method 1668, Revision A, “Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS” with corrections and changes through August 20, 2003

Method 1668C Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS April 2010

DELAWARE RIVER ESTUARY STAGE 2 PCB TMDL Polychlorinated Biphenyls - EPA Method 1668A Project Quality Control Requirements 02/03/05

25.0 HISTORY

Revision 1: Section 15.4.1.2 added.

Revision 2: Calibration upper limit exceedance clarified.

Revision 3: Tables 1, 4, 5, and 6 updated. Table 8 added. 1668A references added. Tridecane use removed. 1668C references and limits added.

Revision 4: Removed references to Version B.

Revision 5: Comments regarding DRBC modifications and references. Additions for DRBC ICAL exceptions.

Revision 6: Added holding time table.

TABLE 1: METHOD ANALYTES AND PQLs

Analyte	Liquid pg/L	Solid pg/g	Tissue pg/g
MoCB 1	20	2	2
MoCB 2	20	2	2
MoCB 3	20	2	2
DiCB 4	100	2	2
DiCB 10	100	2	2
DiCB 9	20	2	2
DiCB 7	20	2	2
DiCB 6	20	2	2
DiCB 5	20	2	2
DiCB 8	20	2	20
DiCB 14	20	2	2
DiCB 11	100	100	100
DiCB 13/12	40	4	8
DiCB 15	20	10	20
TrCB 19	20	2	2
TrCB 18/30	40	4	4
TrCB 17	20	2	2
TrCB 27	20	2	2
TrCB 24	20	2	2
TrCB 16	100	2	2
TrCB 32	20	2	2
TrCB 34	20	2	2
TrCB 23	20	2	2
TrCB 26/29	40	4	4
TrCB 25	20	2	2
TrCB 31	20	10	20
TrCB 20/28	40	20	40
TrCB 21/33	40	20	8
TrCB 22	20	10	20
TrCB 36	20	2	2
TrCB 39	20	2	2
TrCB 38	20	2	2
TrCB 35	20	2	2
TrCB 37	20	10	20
TeCB 54	20	2	2
TeCB 50/53	40	4	4
TeCB 45/51	40	4	4
TeCB 46	20	2	2
TeCB 52	20	2	4
TeCB 73	20	2	2
TeCB 43	20	2	2
TeCB 69/49	40	4	4
TeCB 48	20	2	2

TeCB 44/65/47	60	6	6
TeCB 59/62/75	60	6	6
TeCB 42	20	2	2
TeCB 41	100	100	4
TeCB 40/71	40	4	4
TeCB 64	20	2	4
TeCB 72	20	2	2
TeCB 68	20	2	2
TeCB 57	20	2	2
TeCB 58	20	2	2
TeCB 67	20	2	4
TeCB 63	20	2	2
TeCB 61/76/70/74	80	8	16
TeCB 66	20	10	20
TeCB 55	20	2	2
TeCB 56	20	2	20
TeCB 60	20	2	4
TeCB 80	20	2	2
TeCB 79	20	2	2
TeCB 78	20	2	2
TeCB 81 WHO	20	2	4
TeCB 77 WHO	20	2	4
PeCB 104	20	2	2
PeCB 96	20	2	2
PeCB 103	20	2	2
PeCB 94	20	2	2
PeCB 95	20	2	2
PeCB 93/100	40	4	4
PeCB 102/98	40	4	4
PeCB 88/91	40	4	4
PeCB 84	20	2	2
PeCB 89	20	2	2
PeCB 121	20	2	2
PeCB 92	20	2	2
PeCB 113/90/101	60	6	6
PeCB 83	20	2	2
PeCB 99	100	2	2
PeCB 112	20	2	2
PeCB	120	12	12
PeCB 117/116/85	60	6	6
PeCB 110/115	40	4	4
PeCB 82	20	2	2
PeCB 111	20	2	2
PeCB 120	20	2	2
PeCB 108/124	40	4	4
PeCB 107	20	2	2
PeCB 123 WHO	100	2	4
PeCB 106	20	2	2

PeCB 118 WHO	20	2	4
PeCB 122	20	2	2
PeCB 114 WHO	20	2	2
PeCB 105 WHO *	100	2	4
PeCB 127	20	2	2
PeCB 126 WHO	20	2	2
HxCB 155	20	2	2
HxCB 152	20	2	2
HxCB 150	20	2	2
HxCB 136	20	2	2
HxCB 145	20	2	2
HxCB 148	20	2	2
HxCB 151/135	40	4	4
HxCB 154	20	2	2
HxCB 144	20	2	2
HxCB 147/149	40	4	4
HxCB 134	100	2	2
HxCB 143	20	2	2
HxCB 139/140	40	4	4
HxCB 131	20	2	2
HxCB 142	20	2	2
HxCB 132	20	2	2
HxCB 133	20	2	2
HxCB 165	20	2	2
HxCB 146	20	2	2
HxCB 161	20	2	2
HxCB 153/168	40	4	4
HxCB 141	20	2	2
HxCB 130	20	2	2
HxCB 137	20	2	2
HxCB 164	20	2	2
HxCB 138/163/129	60	6	6
HxCB 160	20	2	2
HxCB 158	20	2	2
HxCB 128/166	40	4	4
HxCB 159	20	2	2
HxCB 162	20	2	2
HxCB 167 WHO	20	2	2
HxCB 156/157 WHO	40	4	4
HxCB 169 WHO	20	2	2
HpCB 188	20	2	2
HpCB 179	20	2	2
HpCB 184	20	2	2
HpCB 176	20	2	2
HpCB 186	20	2	2
HpCB 178	20	2	2
HpCB 175	20	2	2
HpCB 187	20	2	4

HpCB 182	20	2	2
HpCB 183/185	40	4	4
HpCB 174	20	2	4
HpCB 177	20	2	4
HpCB 181	20	2	2
HpCB 173/171	40	4	4
HpCB 172	20	2	4
HpCB 192	20	2	4
HpCB 193/180	40	4	4
HpCB 191	20	2	2
HpCB 170	20	2	2
HpCB 190	20	2	2
HpCB 189 WHO	20	2	2
OcCB 202	20	2	2
OcCB 201	20	2	2
OcCB 204	20	2	2
OcCB 197/200	40	4	8
OcCB 198/199	40	4	4
OcCB 196	20	2	4
OcCB 203	20	2	2
OcCB 195	20	2	2
OcCB 194	20	2	2
OcCB 205	20	2	2
NoCB 208	20	2	2
NoCB 207	20	2	4
NoCB 206	20	2	4
DeCB 209	20	2	2

TABLE 2: MASS DESCRIPTORS

F1	Description*	Mass	Ion
1	13C-MoCB 1	200.0795	M
2	13C-MoCB 2	202.0766	M+2
3	13C-DiCB 1	234.0406	M
4	13C-DiCB 2	236.0376	M+2
5	MoCB 1	188.0393	M
6	MoCB 2	190.0363	M+2
7	DiCB 1	222.0003	M
8	DiCB 2	223.9974	M+2
9	Lock Mass	218.9856	PFK
F2	**Description**	**Mass**	**Ion**
1	13C-DiCB 1	234.0406	M
2	13C-DiCB 2	236.0376	M+2
3	13C-TrCB 1	268.0016	M
4	13C-TrCB 2	269.9986	M+2
5	13C-TeCB 1	301.9626	M
6	13C-TeCB 2	303.9597	M+2
7	DiCB 1	222.0003	M
8	DiCB 2	223.9974	M+2
9	TrCB 1	255.9613	M
10	TrCB 2	257.9584	M+2
11	TeCB 1	289.9224	M
12	TeCB 2	291.9194	M+2
-	DiCB 35 IC	255.9613	M
13	DiCB 35 ICQ	220.9924	M-35Cl
-	TrCB 35 IC	289.9224	M
14	TrCB 35 ICQ	254.9535	M-35Cl
15	Lock Mass	230.9856	PFK
F3	**Description**	**Mass**	**Ion**
1	13C-TrCB 1	268.0016	M
2	13C-TrCB 2	269.9986	M+2
3	13C-TeCB 1	301.9626	M
4	13C-TeCB 2	303.9597	M+2
5	13C-PeCB 1	337.9207	M+2
6	13C-PeCB 2	339.9178	M+4
7	TrCB 1	255.9613	M
8	TrCB 2	257.9584	M+2
9	TeCB 1	289.9224	M
10	TeCB 2	291.9194	M+2
11	PeCB 1	325.8804	M+2
12	PeCB 1	323.8834	M
13	PeCB 2	327.8775	M+4
-	TrCB 35 IC	289.9224	M
14	TrCB 35 ICQ	254.9535	M-35Cl
-	TeCB 35 IC	325.8804	M+2
15	TeCB 35 ICQ	288.9145	M-35Cl
-	TrCB 70 IC	325.8804	M+2
16	TrCB 70 ICQ	253.9457	M-35Cl2
17	Lock Mass	330.9792	PFK
F4	**Description**	**Mass**	**Ion**
1	13C-TeCB 1	301.9626	M
2	13C-TeCB 2	303.9597	M+2
3	13C-PeCB 1	337.9207	M+2
4	13C-PeCB 2	339.9178	M+4
5	13C-HxCB 1	371.8817	M+2
6	13C-HxCB 2	373.8788	M+4
7	TeCB 1	289.9224	M
8	TeCB 2	291.9194	M+2
9	PeCB 1	323.8834	M
10	PeCB2	325.8804	M+2
11	PeCB 3	327.8775	M+4
12	HxCB 1	359.8415	M+2
13	HxCB 2	361.8385	M+4
-	TeCB 35 IC	325.8804	M
14	TeCB 35 ICQ	288.9145	M-35Cl
-	PeCB 35 IC	359.8415	M+2
15	PeCB 35 ICQ	322.8756	M-35Cl
-	TeCB 70 IC	359.8415	M+2
16	TeCB 70 ICQ	287.9067	M-35Cl2
17	Lock Mass	330.9792	PFK
F5	**Description**	**Mass**	**Ion**
1	13C-PeCB 1	337.9207	M+2
2	13C-PeCB 2	339.9178	M+4
3	13C-HxCB 1	371.8817	M+2
4	13C-HxCB 2	373.8788	M+4
5	13C-HpCB 1	405.8428	M+2
6	13C-HpCB 2	407.8398	M+4
7	PeCB 1	323.8834	M
8	PeCB 2	325.8804	M+2
9	PeCB 3	327.8775	M+4
10	HxCB 1	359.8415	M+2
11	HxCB 2	361.8385	M+4
12	HpCB 1	393.8025	M+2
13	HpCB 2	395.7995	M+4
-	PeCB 35 IC	359.8415	M+2
14	PeCB 35 ICQ	322.8756	M-1
-	HxCB 35 IC	393.8025	M+2

15	HxCB 35 ICQ	356.8366	M-35Cl
-	PeCB 70 IC	393.8025	M+2
16	PeCB 70 ICQ	321.8677	M-35Cl2
17	Lock Mass	330.9792	PFK
F6	**Description**	**Mass**	**Ion**
1	13C-HxCB 1	371.8817	M+2
2	13C-HxCB 2	373.8788	M+4
3	13C-HpCB 1	405.8428	M+2
4	13C-HpCB 2	407.8398	M+4
5	13C-OcCB 1	439.8038	M+2
6	13C-OcCB 2	441.8008	M+4
7	HxCB 1	359.8415	M+2
8	HxCB 2	361.8385	M+4
9	HpCB 1	393.8025	M+2
10	HpCB 2	395.7995	M+4
11	OcCB 1	427.7635	M+2
12	OcCB 2	429.7606	M+4
-	HxCB 35 IC	393.8025	M+2
13	HxCB 35 ICQ	356.8366	M-35Cl
-	HpCB 35 IC	427.7635	M+2
14	HpCB 35 ICQ	390.7976	M-35Cl
-	HxCB 70 IC	427.7635	M+2
15	HxCB 70 ICQ	355.8288	M-35Cl2
16	Lock Mass	380.9760	PFK
F7	**Description**	**Mass**	**Ion**
1	13C-HpCB 1	405.8428	M+2
2	13C-HpCB 2	407.8398	M+4
3	13C-OcCB 1	439.8038	M+2
4	13C-OcCB 2	441.8008	M+4
5	13C-NoCB 1	473.7648	M+2
6	13C-NoCB 2	475.7619	M+4
7	13C-DeCB 1	509.7229	M+4
8	13C-DeCB 2	511.7199	M+6
9	HpCB 1	393.8025	M+2
10	HpCB 2	395.7995	M+4
11	OcCB 1	427.7635	M+2
12	OcCB 2	429.7606	M+4
13	NoCB 1	461.7246	M+2
14	NoCB 2	463.7216	M+4
15	DeCB 1	497.6826	M+4
16	DeCB 2	499.6797	M+6
17	Lock Mass	430.9728	PFK

* IC – Interference Check ion
ICQ – Interference Check Quantitation ion

TABLE 3: THEORETICAL ION RATIOS AND CONTROL LIMITS

Homolog Group	M/Z Forming Ratio	Theoretical Ratio	Lower Limit	Upper Limit
Mono	M/M+2	3.13	2.66	3.60
Di	M/M+2	1.56	1.33	1.79
Tri	M/M+2	1.04	0.88	1.20
Tetra	M/M+2	0.77	0.65	0.89
Penta	M/M+2	0.61	0.52	0.71
Penta	M+2/M+4	1.55	1.32	1.78
Hexa	M+2/M+4	1.24	1.05	1.43
Hepta	M+2/M+4	1.05	0.89	1.21
Octa	M+2/M+4	0.89	0.76	1.02
Nona	M+2/M+4	0.77	0.65	0.89
Deca	M+4/M+6	1.16	0.99	1.33

TABLE 4: INITIAL CALIBRATION CONCENTRATIONS

Congener (#)	CS0.5	CS1	CS2	CS3	CS4	CS5
Native Analytes						
2-MoCB (1)	0.5	1	5	50	400	2000
4-MoCB (3)	0.5	1	5	50	400	2000
2,2'-DiCB (4)	0.5	1	5	50	400	2000
4,4'-DiCB (15)	0.5	1	5	50	400	2000
2,2',6'-TrCB (19)	0.5	1	5	50	400	2000
3,4,4'-TrCB (37)	0.5	1	5	50	400	2000
2,2',6,6'-TeCB (54)	0.5	1	5	50	400	2000
3,3',4,4'-TeCB (77)	0.5	1	5	50	400	2000
3,4,4',5-TeCB (81)	0.5	1	5	50	400	2000
2,2',4,6,6'-PeCB (104)	0.5	1	5	50	400	2000
2,3,3',4,4'-PeCB (105)	0.5	1	5	50	400	2000
2,3,4,4',5-PeCB (114)	0.5	1	5	50	400	2000
2,3',4,4',5-PeCB (118)	0.5	1	5	50	400	2000
2',3,4,4',5-PeCB (123)	0.5	1	5	50	400	2000
3,3',4,4',5-PeCB (126)	0.5	1	5	50	400	2000
2,2',4,4',6,6'-HxCB (155)	0.5	1	5	50	400	2000
2,3,3',4,4',5-HxCB (156)	0.5	1	5	50	400	2000
2,3,3',4,4',5'-HxCB (157)	0.5	1	5	50	400	2000
2,3',4,4',5,5'-HxCB (167)	0.5	1	5	50	400	2000
3,3',4,4',5,5'-HxCB (169)	0.5	1	5	50	400	2000
2,2',3,4',5,6,6'-HpCB (188)	0.5	1	5	50	400	2000
2,3,3',4,4',5,5'-HpCB (189)	0.5	1	5	50	400	2000
2,2',3,3',5,5',6,6'-OcCB (202)	0.5	1	5	50	400	2000
2,3,3',4,4',5,5',6-OcCB (205)	0.5	1	5	50	400	2000
2,2',3,3',4,4',5,5',6-NoCB (206)	0.5	1	5	50	400	2000
2,2',3,3',4',5,5',6,6'-NoCB (208)	0.5	1	5	50	400	2000
DeCB (209)	0.5	1	5	50	400	2000
Extraction Standards						
13C12-2-MoCB (1L)	100	100	100	100	100	100
13C12-4-MoCB (3L)	100	100	100	100	100	100
13C12-2,2'-DiCB (4L)	100	100	100	100	100	100
13C12-4,4'-DiCB (15L)	100	100	100	100	100	100
13C12-2,2',6'-TrCB (19L)	100	100	100	100	100	100
13C12-3,4,4'-TrCB (37L)	100	100	100	100	100	100
13C12-2,2',6,6'-TeCB (54L)	100	100	100	100	100	100
13C12-3,3',4,4'-TeCB (77L)	100	100	100	100	100	100
13C12-3,4,4',5-TeCB (81L)	100	100	100	100	100	100
13C12-2,2',4,6,6'-PeCB (104L)	100	100	100	100	100	100
13C12-2,3,3',4,4'-PeCB (105L)	100	100	100	100	100	100
13C12-2,3,4,4',5-PeCB (114L)	100	100	100	100	100	100
13C12-2,3',4,4',5-PeCB (118L)	100	100	100	100	100	100
13C12-2',3,4,4',5-PeCB (123L)	100	100	100	100	100	100
13C12-3,3',4,4',5-PeCB (126L)	100	100	100	100	100	100
13C12-2,2',4,4',6,6'-HxCB (155L)	100	100	100	100	100	100
13C12-2,3,3',4,4',5-HxCB (156L)	100	100	100	100	100	100
13C12-2,3,3',4,4',5'-HxCB (157L)	100	100	100	100	100	100
13C12-2,3',4,4',5,5'-HxCB (167L)	100	100	100	100	100	100
13C12-3,3',4,4',5,5'-HxCB (169L)	100	100	100	100	100	100
13C12-2,2',3,4',5,6,6'-HpCB (188L)	100	100	100	100	100	100
13C12-2,3,3',4,4',5,5'-HpCB (189L)	100	100	100	100	100	100
13C12-2,2',3,3',5,5',6,6'-OcCB (202L)	100	100	100	100	100	100

(CONTD)

13C12-2,3,3',4,4',5,5',6-OcCB (205L)	100	100	100	100	100	100
13C12-2,2',3,3',4,4',5,5',6-NoCB (206L)	100	100	100	100	100	100
13C12-2,2',3,3',4',5,5',6,6'-NoCB (208L)	100	100	100	100	100	100
13C12-DeCB (209L)	100	100	100	100	100	100
Cleanup Standards						
13C12-2,4,4'-TrCB (28L)	100	100	100	100	100	100
13C12-2,3,3',5,5'-PeCB (111L)	100	100	100	100	100	100
13C12-2,2',3,3',5,5',6-HpCB (178L)	100	100	100	100	100	100
Injection Standards						
13C12-2,5-DiCB (9L)	100	100	100	100	100	100
13C12-2,2',5,5'-TeCB (52L)	100	100	100	100	100	100
13C12-2,2',4',5,5'-PeCB (101L)	100	100	100	100	100	100
13C12-2,2',3',4,4',5'-HxCB (138L)	100	100	100	100	100	100
13C12-2,2',3,3',4,4',5,5'-OcCB (194L)	100	100	100	100	100	100

TABLE 5: LCS LIMITS

Method 1668A

Native Analytes	Lower %	Upper %
2-MoCB (1)	50	150
4-MoCB (3)	50	150
2,2'-DiCB (4)	50	150
4,4'-DiCB (15)	50	150
2,2',6'-TrCB (19)	50	150
3,4,4'-TrCB (37)	50	150
2,2',6,6'-TeCB (54)	50	150
3,3',4,4'-TeCB (77)	50	150
3,4,4',5-TeCB (81)	50	150
2,2',4,6,6'-PeCB (104)	50	150
2,3,3',4,4'-PeCB (105)	50	150
2,3,4,4',5-PeCB (114)	50	150
2,3',4,4',5-PeCB (118)	50	150
2',3,4,4',5-PeCB (123)	50	150
3,3',4,4',5-PeCB (126)	50	150
2,2',4,4',6,6'-HxCB (155)	50	150
2,3,3',4,4',5-HxCB (156)	50	150
2,3,3',4,4',5'-HxCB (157)	50	150
2,3',4,4',5,5'-HxCB (167)	50	150
3,3',4,4',5,5'-HxCB (169)	50	150
2,2',3,4',5,6,6'-HpCB (188)	50	150
2,3,3',4,4',5,5'-HpCB (189)	50	150
2,2',3,3',5,5',6,6'-OcCB (202)	50	150
2,3,3',4,4',5,5',6-OcCB (205)	50	150
2,2',3,3',4,4',5,5',6-NoCB (206)	50	150
2,2',3,3',4',5,5',6,6'-NoCB (208)	50	150
DeCB (209)	50	150

Extraction Standards	Lower %	Upper %
13C12-2-MoCB (1L)	15	140
13C12-4-MoCB (3L)	15	140
13C12-2,2'-DiCB (4L)	30	140
13C12-4,4'-DiCB (15L)	30	140
13C12-2,2',6'-TrCB (19L)	30	140
13C12-3,4,4'-TrCB (37L)	30	140
13C12-2,2',6,6'-TeCB (54L)	30	140
13C12-3,3',4,4'-TeCB (77L)	30	140
13C12-3,4,4',5-TeCB (81L)	30	140
13C12-2,2',4,6,6'-PeCB (104L)	30	140
13C12-2,3,3',4,4'-PeCB (105L)	30	140
13C12-2,3,4,4',5-PeCB (114L)	30	140
13C12-2,3',4,4',5-PeCB (118L)	30	140
13C12-2',3,4,4',5-PeCB (123L)	30	140
13C12-3,3',4,4',5-PeCB (126L)	30	140
13C12-2,2',4,4',6,6'-HxCB (155L)	30	140
13C12-2,3,3',4,4',5-HxCB (156L)	30	140
13C12-2,3,3',4,4',5'-HxCB (157L)	30	140
13C12-2,3',4,4',5,5'-HxCB (167L)	30	140
13C12-3,3',4,4',5,5'-HxCB (169L)	30	140
13C12-2,2',3,4',5,6,6'-HpCB (188L)	30	140
13C12-2,3,3',4,4',5,5'-HpCB (189L)	30	140
13C12-2,2',3,3',5,5',6,6'-OcCB (202L)	30	140
13C12-2,3,3',4,4',5,5',6-OcCB (205L)	30	140
13C12-2,2',3,3',4,4',5,5',6-NoCB (206L)	30	140
13C12-2,2',3,3',4',5,5',6,6'-NoCB (208L)	30	140
13C12-DeCB (209L)	30	140
Cleanup Standards		
13C12-2,4,4'-TrCB (28L)	40	125
13C12-2,3,3',5,5'-PeCB (111L)	40	125
13C12-2,2',3,3',5,5',6-HpCB (178L)	40	125

Method 1668C (LCS Limits)

Native Analytes	Lower %	Upper %
2-MoCB (1)	60	135
4-MoCB (3)	60	135
2,2'-DiCB (4)	60	135
4,4'-DiCB (15)	60	135
2,2',6'-TrCB (19)	60	135
3,4,4'-TrCB (37)	60	135
2,2',6,6'-TeCB (54)	60	135
3,3',4,4'-TeCB (77)	60	135
3,4,4',5-TeCB (81)	60	135
2,2',4,6,6'-PeCB (104)	60	135
2,3,3',4,4'-PeCB (105)	60	135
2,3,4,4',5-PeCB (114)	60	135
2,3',4,4',5-PeCB (118)	60	135
2',3,4,4',5-PeCB (123)	60	135
3,3',4,4',5-PeCB (126)	60	135
2,2',4,4',6,6'-HxCB (155)	60	135
2,3,3',4,4',5-HxCB (156)	60	135
2,3,3',4,4',5'-HxCB (157)	60	135
2,3',4,4',5,5'-HxCB (167)	60	135
3,3',4,4',5,5'-HxCB (169)	60	135
2,2',3,4',5,6,6'-HpCB (188)	60	135
2,3,3',4,4',5,5'-HpCB (189)	60	135
2,2',3,3',5,5',6,6'-OcCB (202)	60	135
2,3,3',4,4',5,5',6-OcCB (205)	60	135
2,2',3,3',4,4',5,5',6-NoCB (206)	60	135
2,2',3,3',4',5,5',6,6'-NoCB (208)	60	135
DeCB (209)	60	135

Extraction Standards	Lower %	Upper %
13C12-2-MoCB (1L)	15	145
13C12-4-MoCB (3L)	15	145
13C12-2,2'-DiCB (4L)	15	145
13C12-4,4'-DiCB (15L)	15	145
13C12-2,2',6'-TrCB (19L)	15	145
13C12-3,4,4'-TrCB (37L)	15	145
13C12-2,2',6,6'-TeCB (54L)	15	145
13C12-3,3',4,4'-TeCB (77L)	40	145
13C12-3,4,4',5-TeCB (81L)	40	145
13C12-2,2',4,6,6'-PeCB (104L)	40	145
13C12-2,3,3',4,4'-PeCB (105L)	40	145
13C12-2,3,4,4',5-PeCB (114L)	40	145
13C12-2,3',4,4',5-PeCB (118L)	40	145
13C12-2',3,4,4',5-PeCB (123L)	40	145
13C12-3,3',4,4',5-PeCB (126L)	40	145
13C12-2,2',4,4',6,6'-HxCB (155L)	40	145
13C12-2,3,3',4,4',5-HxCB (156L)	40	145
13C12-2,3,3',4,4',5'-HxCB (157L)	40	145
13C12-2,3',4,4',5,5'-HxCB (167L)	40	145
13C12-3,3',4,4',5,5'-HxCB (169L)	40	145
13C12-2,2',3,4',5,6,6'-HpCB (188L)	40	145
13C12-2,3,3',4,4',5,5'-HpCB (189L)	40	145
13C12-2,2',3,3',5,5',6,6'-OcCB (202L)	40	145
13C12-2,3,3',4,4',5,5',6-OcCB (205L)	40	145
13C12-2,2',3,3',4,4',5,5',6-NoCB (206L)	40	145
13C12-2,2',3,3',4',5,5',6,6'-NoCB (208L)	40	145
13C12-DeCB (209L)	40	145
Cleanup Standards		
13C12-2,4,4'-TrCB (28L)	15	145
13C12-2,3,3',5,5'-PeCB (111L)	40	145
13C12-2,2',3,3',5,5',6-HpCB (178L)	40	145

TABLE 6: SAMPLE AND LMB RECOVERY LIMITS

Method 1668A

Extraction Standards	Lower %	Upper %
13C12-2-MoCB (1L)	15	150
13C12-4-MoCB (3L)	15	150
13C12-2,2'-DiCB (4L)	25	150
13C12-4,4'-DiCB (15L)	25	150
13C12-2,2',6'-TrCB (19L)	25	150
13C12-3,4,4'-TrCB (37L)	25	150
13C12-2,2',6,6'-TeCB (54L)	25	150
13C12-3,3',4,4'-TeCB (77L)	25	150
13C12-3,4,4',5-TeCB (81L)	25	150
13C12-2,2',4,6,6'-PeCB (104L)	25	150
13C12-2,3,3',4,4'-PeCB (105L)	25	150
13C12-2,3,4,4',5-PeCB (114L)	25	150
13C12-2,3',4,4',5-PeCB (118L)	25	150
13C12-2',3,4,4',5-PeCB (123L)	25	150
13C12-3,3',4,4',5-PeCB (126L)	25	150
13C12-2,2',4,4',6,6'-HxCB (155L)	25	150
13C12-2,3,3',4,4',5-HxCB (156L)	25	150
13C12-2,3,3',4,4',5'-HxCB (157L)	25	150
13C12-2,3',4,4',5,5'-HxCB (167L)	25	150
13C12-3,3',4,4',5,5'-HxCB (169L)	25	150
13C12-2,2',3,4',5,6,6'-HpCB (188L)	25	150
13C12-2,3,3',4,4',5,5'-HpCB (189L)	25	150
13C12-2,2',3,3',5,5',6,6'-OcCB (202L)	25	150
13C12-2,3,3',4,4',5,5',6-OcCB (205L)	25	150
13C12-2,2',3,3',4,4',5,5',6-NoCB (206L)	25	150
13C12-2,2',3,3',4',5,5',6,6'-NoCB (208L)	25	150
13C12-DeCB (209L)	25	150
Cleanup Standards		
13C12-2,4,4'-TrCB (28L)	30	135
13C12-2,3,3',5,5'-PeCB (111L)	30	135
13C12-2,2',3,3',5,5',6-HpCB (178L)	30	135

Method 1668C (Sample and LMB Recovery Limits)

Extraction Standards	Lower %	Upper %
13C12-2-MoCB (1L)	5	145
13C12-4-MoCB (3L)	5	145
13C12-2,2'-DiCB (4L)	5	145
13C12-4,4'-DiCB (15L)	5	145
13C12-2,2',6'-TrCB (19L)	5	145
13C12-3,4,4'-TrCB (37L)	5	145
13C12-2,2',6,6'-TeCB (54L)	5	145
13C12-3,3',4,4'-TeCB (77L)	10	145
13C12-3,4,4',5-TeCB (81L)	10	145
13C12-2,2',4,6,6'-PeCB (104L)	10	145
13C12-2,3,3',4,4'-PeCB (105L)	10	145
13C12-2,3,4,4',5-PeCB (114L)	10	145
13C12-2,3',4,4',5-PeCB (118L)	10	145
13C12-2',3,4,4',5-PeCB (123L)	10	145
13C12-3,3',4,4',5-PeCB (126L)	10	145
13C12-2,2',4,4',6,6'-HxCB (155L)	10	145
13C12-2,3,3',4,4',5-HxCB (156L)	10	145
13C12-2,3,3',4,4',5'-HxCB (157L)	10	145
13C12-2,3',4,4',5,5'-HxCB (167L)	10	145
13C12-3,3',4,4',5,5'-HxCB (169L)	10	145
13C12-2,2',3,4',5,6,6'-HpCB (188L)	10	145
13C12-2,3,3',4,4',5,5'-HpCB (189L)	10	145
13C12-2,2',3,3',5,5',6,6'-OcCB (202L)	10	145
13C12-2,3,3',4,4',5,5',6-OcCB (205L)	10	145
13C12-2,2',3,3',4,4',5,5',6-NoCB (206L)	10	145
13C12-2,2',3,3',4',5,5',6,6'-NoCB (208L)	10	145
13C12-DeCB (209L)	10	145
Cleanup Standards		
13C12-2,4,4'-TrCB (28L)	5	145
13C12-2,3,3',5,5'-PeCB (111L)	10	145
13C12-2,2',3,3',5,5',6-HpCB (178L)	10	145

TABLE 7: CONTINUING CALIBRATION LIMITS (VER)

Methods 1668A

Native Analytes	Lower %	Upper %
2-MoCB (1)	70	130
4-MoCB (3)	70	130
2,2'-DiCB (4)	70	130
4,4'-DiCB (15)	70	130
2,2',6'-TrCB (19)	70	130
3,4,4'-TrCB (37)	70	130
2,2',6,6'-TeCB (54)	70	130
3,3',4,4'-TeCB (77)	70	130
3,4,4',5-TeCB (81)	70	130
2,2',4,6,6'-PeCB (104)	70	130
2,3,3',4,4'-PeCB (105)	70	130
2,3,4,4',5-PeCB (114)	70	130
2,3',4,4',5-PeCB (118)	70	130
2',3,4,4',5-PeCB (123)	70	130
3,3',4,4',5-PeCB (126)	70	130
2,2',4,4',6,6'-HxCB (155)	70	130
2,3,3',4,4',5-HxCB (156)	70	130
2,3,3',4,4',5'-HxCB (157)	70	130
2,3',4,4',5,5'-HxCB (167)	70	130
3,3',4,4',5,5'-HxCB (169)	70	130
2,2',3,4',5,6,6'-HpCB (188)	70	130
2,3,3',4,4',5,5'-HpCB (189)	70	130
2,2',3,3',5,5',6,6'-OcCB (202)	70	130
2,3,3',4,4',5,5',6-OcCB (205)	70	130
2,2',3,3',4,4',5,5',6-NoCB (206)	70	130
2,2',3,3',4',5,5',6,6'-NoCB (208)	70	130
DeCB (209)	70	130

Extraction Standards	Lower %	Upper %
13C12-2-MoCB (1L)	50	150
13C12-4-MoCB (3L)	50	150
13C12-2,2'-DiCB (4L)	50	150
13C12-4,4'-DiCB (15L)	50	150
13C12-2,2',6'-TrCB (19L)	50	150
13C12-3,4,4'-TrCB (37L)	50	150
13C12-2,2',6,6'-TeCB (54L)	50	150
13C12-3,3',4,4'-TeCB (77L)	50	150
13C12-3,4,4',5-TeCB (81L)	50	150
13C12-2,2',4,6,6'-PeCB (104L)	50	150
13C12-2,3,3',4,4'-PeCB (105L)	50	150
13C12-2,3,4,4',5-PeCB (114L)	50	150
13C12-2,3',4,4',5-PeCB (118L)	50	150
13C12-2',3,4,4',5-PeCB (123L)	50	150
13C12-3,3',4,4',5-PeCB (126L)	50	150
13C12-2,2',4,4',6,6'-HxCB (155L)	50	150
13C12-2,3,3',4,4',5-HxCB (156L)	50	150
13C12-2,3,3',4,4',5'-HxCB (157L)	50	150
13C12-2,3',4,4',5,5'-HxCB (167L)	50	150
13C12-3,3',4,4',5,5'-HxCB (169L)	50	150
13C12-2,2',3,4',5,6,6'-HpCB (188L)	50	150
13C12-2,3,3',4,4',5,5'-HpCB (189L)	50	150
13C12-2,2',3,3',5,5',6,6'-OcCB (202L)	50	150
13C12-2,3,3',4,4',5,5',6-OcCB (205L)	50	150
13C12-2,2',3,3',4,4',5,5',6-NoCB (206L)	50	150
13C12-2,2',3,3',4',5,5',6,6'-NoCB (208L)	50	150
13C12-DeCB (209L)	50	150
Cleanup Standards		
13C12-2,4,4'-TrCB (28L)	60	130
13C12-2,3,3',5,5'-PeCB (111L)	60	130
13C12-2,2',3,3',5,5',6-HpCB (178L)	60	130

Method 1668C Continuing Calibration Limit (VER)

Native Analytes	Lower %	Upper %
2-MoCB (1)	75	125
4-MoCB (3)	75	125
2,2'-DiCB (4)	75	125
4,4'-DiCB (15)	75	125
2,2',6'-TrCB (19)	75	125
3,4,4'-TrCB (37)	75	125
2,2',6,6'-TeCB (54)	75	125
3,3',4,4'-TeCB (77)	75	125
3,4,4',5-TeCB (81)	75	125
2,2',4,6,6'-PeCB (104)	75	125
2,3,3',4,4'-PeCB (105)	75	125
2,3,4,4',5-PeCB (114)	75	125
2,3',4,4',5-PeCB (118)	75	125
2',3,4,4',5-PeCB (123)	75	125
3,3',4,4',5-PeCB (126)	75	125
2,2',4,4',6,6'-HxCB (155)	75	125
2,3,3',4,4',5-HxCB (156)	75	125
2,3,3',4,4',5'-HxCB (157)	75	125
2,3',4,4',5,5'-HxCB (167)	75	125
3,3',4,4',5,5'-HxCB (169)	75	125
2,2',3,4',5,6,6'-HpCB (188)	75	125
2,3,3',4,4',5,5'-HpCB (189)	75	125
2,2',3,3',5,5',6,6'-OcCB (202)	75	125
2,3,3',4,4',5,5',6-OcCB (205)	75	125
2,2',3,3',4,4',5,5',6-NoCB (206)	75	125
2,2',3,3',4',5,5',6,6'-NoCB (208)	75	125
DeCB (209)	75	125

Extraction Standards	Lower %	Upper %
13C12-2-MoCB (1L)	50	145
13C12-4-MoCB (3L)	50	145
13C12-2,2'-DiCB (4L)	50	145
13C12-4,4'-DiCB (15L)	50	145
13C12-2,2',6'-TrCB (19L)	50	145
13C12-3,4,4'-TrCB (37L)	50	145
13C12-2,2',6,6'-TeCB (54L)	50	145
13C12-3,3',4,4'-TeCB (77L)	50	145
13C12-3,4,4',5-TeCB (81L)	50	145
13C12-2,2',4,6,6'-PeCB (104L)	50	145
13C12-2,3,3',4,4'-PeCB (105L)	50	145
13C12-2,3,4,4',5-PeCB (114L)	50	145
13C12-2,3',4,4',5-PeCB (118L)	50	145
13C12-2',3,4,4',5-PeCB (123L)	50	145
13C12-3,3',4,4',5-PeCB (126L)	50	145
13C12-2,2',4,4',6,6'-HxCB (155L)	50	145
13C12-2,3,3',4,4',5-HxCB (156L)	50	145
13C12-2,3,3',4,4',5'-HxCB (157L)	50	145
13C12-2,3',4,4',5,5'-HxCB (167L)	50	145
13C12-3,3',4,4',5,5'-HxCB (169L)	50	145
13C12-2,2',3,4',5,6,6'-HpCB (188L)	50	145
13C12-2,3,3',4,4',5,5'-HpCB (189L)	50	145
13C12-2,2',3,3',5,5',6,6'-OcCB (202L)	50	145
13C12-2,3,3',4,4',5,5',6-OcCB (205L)	50	145
13C12-2,2',3,3',4,4',5,5',6-NoCB (206L)	50	145
13C12-2,2',3,3',4',5,5',6,6'-NoCB (208L)	50	145
13C12-DeCB (209L)	50	145
Cleanup Standards		
13C12-2,4,4'-TrCB (28L)	65	135
13C12-2,3,3',5,5'-PeCB (111L)	75	125
13C12-2,2',3,3',5,5',6-HpCB (178L)	75	125

TABLE 8: RETENTION TIME LIMITS (and example relative retention time limits)

Reference 209 Mix Analyte	Example RT (min)	RT Ref	Example RT Ref (min)	Window (sec) low	Window (sec) high	Example Resulting RRT Limits
MoCB-1	6.99	1L	6.98	-3	3	0.994 - 1.009
MoCB-2	8.64	3L	8.77	-3	3	0.979 - 0.991
MoCB-3	8.78	3L	8.77	-3	3	0.995 - 1.007
DiCB-4	8.99	4L	8.97	-3	3	0.997 - 1.008
DiCB-10	9.13	4L	8.97	-3	3	1.012 - 1.023
DiCB-9	10.65	4L	8.97	-3	3	1.182 - 1.193
DiCB-7	10.80	4L	8.97	-3	3	1.198 - 1.210
DiCB-6	11.01	4L	8.97	-3	3	1.222 - 1.233
DiCB-5	11.27	4L	8.97	-3	3	1.251 - 1.262
DiCB-8	11.38	4L	8.97	-3	3	1.263 - 1.274
DiCB-14	12.87	15L	14.25	-3	3	0.900 - 0.907
DiCB-11	13.66	15L	14.25	-3	3	0.955 - 0.962
DiCB-13/12	13.98	15L	14.25	-3	3	0.978 - 0.985
DiCB-15	14.26	15L	14.25	-3	3	0.997 - 1.004
TrCB-19	11.66	19L	11.65	-3	3	0.997 - 1.005
TrCB-18/30	13.35	19L	11.65	-3	3	1.142 - 1.150
TrCB-17	13.77	19L	11.65	-3	3	1.178 - 1.186
TrCB-27	13.96	19L	11.65	-3	3	1.194 - 1.203
TrCB-24	14.11	19L	11.65	-3	3	1.207 - 1.215
TrCB-16	14.22	19L	11.65	-3	3	1.216 - 1.225
TrCB-32	14.72	19L	11.65	-3	3	1.259 - 1.268
TrCB-34	15.98	19L	11.65	-3	3	1.367 - 1.376
TrCB-23	16.16	19L	11.65	-3	3	1.383 - 1.391
TrCB-26/29	16.49	19L	11.65	-5	5	1.408 - 1.423
TrCB-25	16.71	37L	21.93	-3	3	0.760 - 0.764
TrCB-31	17.04	37L	21.93	-3	3	0.775 - 0.779
TrCB-20/28	17.38	37L	21.93	-5	5	0.789 - 0.796
TrCB-21/33	17.61	37L	21.93	-5	5	0.799 - 0.807
TrCB-22	18.06	37L	21.93	-3	3	0.821 - 0.826
TrCB-36	19.81	37L	21.93	-3	3	0.901 - 0.906
TrCB-39	20.23	37L	21.93	-3	3	0.920 - 0.925
TrCB-38	20.93	37L	21.93	-3	3	0.952 - 0.957
TrCB-35	21.47	37L	21.93	-3	3	0.977 - 0.981
TrCB-37	21.96	37L	21.93	-3	3	0.999 - 1.004
TeCB-54	14.58	54L	14.55	-3	3	0.999 - 1.005
TeCB-50/53	16.77	54L	14.55	-5	5	1.147 - 1.158
TeCB-45/51	17.56	54L	14.55	-5	5	1.201 - 1.213

TeCB-46	17.82	54L	14.55	-3	3	1.221	-	1.228
TeCB-52	19.38	54L	14.55	-3	3	1.329	-	1.335
TeCB-73	19.54	54L	14.55	-3	3	1.340	-	1.346
TeCB-43	19.67	54L	14.55	-3	3	1.348	-	1.355
TeCB-69/49	19.92	54L	14.55	-5	5	1.363	-	1.375
TeCB-48	20.29	54L	14.55	-3	3	1.391	-	1.398
TeCB-44/65/47	20.59	54L	14.55	-5	5	1.409	-	1.421
TeCB-59/62/75	20.93	54L	14.55	-5	5	1.433	-	1.444
TeCB-42	21.17	54L	14.55	-3	3	1.452	-	1.458
TeCB-41	21.63	54L	14.55	-5	5	1.481	-	1.492
TeCB-40/71	21.76	54L	14.55	-5	5	1.490	-	1.501
TeCB-64	22.03	54L	14.55	-3	3	1.511	-	1.518
TeCB-72	23.04	81L	30.73	-3	3	0.748	-	0.751
TeCB-68	23.40	81L	30.73	-3	3	0.760	-	0.763
TeCB-57	23.93	81L	30.73	-3	3	0.777	-	0.780
TeCB-58	24.23	81L	30.73	-3	3	0.787	-	0.790
TeCB-67	24.45	81L	30.73	-3	3	0.794	-	0.797
TeCB-63	24.79	81L	30.73	-3	3	0.805	-	0.808
TeCB-61/76/70/74	25.20	81L	30.73	-6	6	0.817	-	0.823
TeCB-66	25.63	81L	30.73	-3	3	0.832	-	0.836
TeCB-55	25.84	81L	30.73	-3	3	0.839	-	0.842
TeCB-56	26.49	81L	30.73	-3	3	0.860	-	0.864
TeCB-60	26.78	81L	30.73	-3	3	0.870	-	0.873
TeCB-80	27.34	81L	30.73	-3	3	0.888	-	0.891
TeCB-79	29.39	81L	30.73	-3	3	0.955	-	0.958
TeCB-78	30.16	81L	30.73	-3	3	0.980	-	0.983
TeCB-81 WHO	30.76	81L	30.73	-3	3	0.999	-	1.003
TeCB-77 WHO	31.54	77L	31.51	-3	3	0.999	-	1.003
PeCB-104	20.51	104L	20.48	-3	3	0.999	-	1.004
PeCB-96	20.93	104L	20.48	-5	5	1.018	-	1.026
PeCB-103	23.28	104L	20.48	-3	3	1.134	-	1.139
PeCB-94	23.55	104L	20.48	-3	3	1.147	-	1.152
PeCB-95	24.09	104L	20.48	-5	5	1.172	-	1.180
PeCB-93/100	24.39	104L	20.48	-5	5	1.187	-	1.195
PeCB-102/98	24.57	104L	20.48	-5	5	1.196	-	1.204
PeCB-88/91	25.19	104L	20.48	-6	6	1.225	-	1.235
PeCB-84	25.47	104L	20.48	-3	3	1.241	-	1.246
PeCB-89	26.10	104L	20.48	-3	3	1.272	-	1.277
PeCB-121	26.67	104L	20.48	-3	3	1.300	-	1.305
PeCB-92	27.15	123L	34.37	-3	3	0.788	-	0.791
PeCB-113/90/101	27.91	123L	34.37	-5	5	0.810	-	0.814
PeCB-83	28.55	123L	34.37	-6	6	0.828	-	0.834
PeCB-99	28.72	123L	34.37	-5	5	0.833	-	0.838

PeCB-112	28.87	123L	34.37	-3	3	0.839	-	0.841
PeCB-86/87/97/109/119/125	29.38	123L	34.37	-8	8	0.851	-	0.859
PeCB-117/116/85	30.38	123L	34.37	-5	5	0.881	-	0.886
PeCB-110/115	30.73	123L	34.37	-5	5	0.892	-	0.897
PeCB-82	31.05	123L	34.37	-3	3	0.902	-	0.905
PeCB-111	31.62	123L	34.37	-3	3	0.919	-	0.921
PeCB-120	32.28	123L	34.37	-3	3	0.938	-	0.941
PeCB-108/124	33.88	123L	34.37	-8	8	0.982	-	0.990
PeCB-107	34.23	123L	34.37	-5	5	0.994	-	0.998
PeCB-123 WHO	34.40	123L	34.37	-3	3	0.999	-	1.002
PeCB-106	34.56	123L	34.37	-3	3	1.004	-	1.007
PeCB-118 WHO	34.86	118L	34.85	-3	3	0.999	-	1.002
PeCB-122	35.36	118L	34.85	-3	3	1.013	-	1.016
PeCB-114 WHO	35.64	114L	35.60	-3	3	1.000	-	1.003
PeCB-105 WHO	36.57	105L	36.54	-3	3	0.999	-	1.002
PeCB-127	38.78	105L	36.54	-3	3	1.060	-	1.063
PeCB-126 WHO	41.17	126L	41.14	-3	3	1.000	-	1.002
HxCB-155	27.64	155L	27.61	-3	3	0.999	-	1.003
HxCB-152	27.88	155L	27.61	-3	3	1.008	-	1.012
HxCB-150	28.12	155L	27.61	-3	3	1.017	-	1.020
HxCB-136	28.58	155L	27.61	-3	3	1.033	-	1.037
HxCB-145	29.00	155L	27.61	-3	3	1.049	-	1.052
HxCB-148	31.10	155L	27.61	-3	3	1.125	-	1.128
HxCB-151/135	31.94	155L	27.61	-5	5	1.154	-	1.160
HxCB-154	32.29	155L	27.61	-5	5	1.166	-	1.173
HxCB-144	32.72	155L	27.61	-3	3	1.183	-	1.187
HxCB-147/149	33.22	155L	27.61	-5	5	1.200	-	1.206
HxCB-134	33.48	155L	27.61	-5	5	1.210	-	1.216
HxCB-143	33.64	155L	27.61	-5	5	1.215	-	1.221
HxCB-139/140	34.08	155L	27.61	-5	5	1.231	-	1.237
HxCB-131	34.35	155L	27.61	-3	3	1.242	-	1.246
HxCB-142	34.58	155L	27.61	-3	3	1.251	-	1.254
HxCB-132	35.01	155L	27.61	-5	5	1.265	-	1.271
HxCB-133	35.77	155L	27.61	-3	3	1.294	-	1.297
HxCB-165	36.36	167L	44.03	-3	3	0.825	-	0.827
HxCB-146	36.73	167L	44.03	-3	3	0.833	-	0.835
HxCB-161	36.92	167L	44.03	-3	3	0.837	-	0.840
HxCB-153/168	37.68	167L	44.03	-5	5	0.854	-	0.858
HxCB-141	37.89	167L	44.03	-3	3	0.859	-	0.862
HxCB-130	38.49	167L	44.03	-3	3	0.873	-	0.875
HxCB-137	38.85	167L	44.03	-3	3	0.881	-	0.883
HxCB-164	39.00	167L	44.03	-3	3	0.885	-	0.887

HxCB-138/163/129	39.48	167L	44.03	-7	7	0.894	-	0.899
HxCB-160	39.71	167L	44.03	-5	5	0.900	-	0.904
HxCB-158	40.06	167L	44.03	-3	3	0.909	-	0.911
HxCB-128/166	41.31	167L	44.03	-5	5	0.936	-	0.940
HxCB-159	42.90	167L	44.03	-3	3	0.973	-	0.975
HxCB-162	43.33	167L	44.03	-3	3	0.983	-	0.985
HxCB-167 WHO	44.06	167L	44.03	-3	3	1.000	-	1.002
HxCB-156/157 WHO	45.81	156L	45.76	-3	3	1.000	-	1.002
HxCB-169 WHO	50.88	169L	50.83	-3	3	1.000	-	1.002
HpCB-188	35.64	188L	35.61	-3	3	0.999	-	1.002
HpCB-179	36.11	188L	35.61	-3	3	1.013	-	1.015
HpCB-184	36.92	188L	35.61	-3	3	1.035	-	1.038
HpCB-176	37.41	188L	35.61	-3	3	1.049	-	1.052
HpCB-186	38.08	188L	35.61	-3	3	1.068	-	1.071
HpCB-178	40.17	188L	35.61	-3	3	1.127	-	1.129
HpCB-175	41.12	188L	35.61	-3	3	1.153	-	1.156
HpCB-187	41.54	188L	35.61	-3	3	1.165	-	1.168
HpCB-182	41.86	188L	35.61	-3	3	1.174	-	1.177
HpCB-183/185	42.48	188L	35.61	-3	3	1.192	-	1.194
HpCB-174	42.80	188L	35.61	-3	3	1.201	-	1.203
HpCB-177	43.47	188L	35.61	-3	3	1.219	-	1.222
HpCB-181	44.09	188L	35.61	-3	3	1.237	-	1.240
HpCB-173/171	44.41	188L	35.61	-5	5	1.245	-	1.249
HpCB-172	46.99	189L	54.86	-3	3	0.856	-	0.857
HpCB-192	47.42	189L	54.86	-3	3	0.863	-	0.865
HpCB-193/180	47.93	189L	54.86	-3	3	0.873	-	0.875
HpCB-191	48.55	189L	54.86	-3	3	0.884	-	0.886
HpCB-170	49.94	189L	54.86	-3	3	0.909	-	0.911
HpCB-190	50.77	189L	54.86	-3	3	0.925	-	0.926
HpCB-189 WHO	54.89	189L	54.86	-3	3	1.000	-	1.001
OcCB-202	43.68	202L	43.65	-3	3	1.000	-	1.002
OcCB-201	45.11	202L	43.65	-5	5	1.032	-	1.035
OcCB-204	46.16	202L	43.65	-3	3	1.056	-	1.059
OcCB-197/200	46.51	202L	43.65	-3	3	1.064	-	1.067
OcCB-198/199	51.05	202L	43.65	-5	5	1.168	-	1.171
OcCB-196	52.12	205L	58.97	-3	3	0.883	-	0.885
OcCB-203	52.44	205L	58.97	-3	3	0.888	-	0.890
OcCB-195	54.48	205L	58.97	-3	3	0.923	-	0.925
OcCB-194	58.26	205L	58.97	-3	3	0.987	-	0.989
OcCB-205	59.00	205L	58.97	-3	3	1.000	-	1.001
NoCB-208	54.14	208L	54.11	-3	3	1.000	-	1.001
NoCB-207	55.63	208L	54.11	-3	3	1.027	-	1.029
NoCB-206	61.88	206L	61.85	-3	3	1.000	-	1.001

DeCB-209	64.86	209L	64.81	-3	3	1.000	-	1.002
MoCB-1L	6.98	9L	10.63	-15	15	0.633	-	0.680
MoCB-3L	8.77	9L	10.63	-15	15	0.802	-	0.849
DiCB-4L	8.97	9L	10.63	-15	15	0.820	-	0.867
DiCB-15L	14.25	9L	10.63	-10	10	1.325	-	1.356
TrCB-19L	11.65	9L	10.63	-15	15	1.072	-	1.119
TrCB-37L	21.93	52L	19.37	-15	15	1.119	-	1.145
TeCB-54L	14.55	52L	19.37	-10	10	0.743	-	0.760
TeCB-81L	30.73	52L	19.37	-10	10	1.578	-	1.595
TeCB-77L	31.51	52L	19.37	-10	10	1.618	-	1.635
PeCB-104L	20.48	101L	27.90	-10	10	0.728	-	0.740
PeCB-123L	34.37	101L	27.90	-10	10	1.226	-	1.238
PeCB-118L	34.85	101L	27.90	-10	10	1.243	-	1.255
PeCB-114L	35.60	101L	27.90	-10	10	1.270	-	1.282
PeCB-105L	36.54	101L	27.90	-10	10	1.304	-	1.316
PeCB-126L	41.14	101L	27.90	-10	10	1.469	-	1.481
HxCB-155L	27.61	138L	39.43	-10	10	0.696	-	0.704
HxCB-167L	44.03	138L	39.43	-10	10	1.112	-	1.121
HxCB-156L/157L	45.76	138L	39.43	-10	10	1.156	-	1.165
HxCB-169L	50.83	138L	39.43	-10	10	1.285	-	1.293
HpCB-188L	35.61	194L	58.23	-10	10	0.609	-	0.614
HpCB-189L	54.86	194L	58.23	-10	10	0.939	-	0.945
OcCB-202L	43.65	194L	58.23	-10	10	0.747	-	0.752
OcCB-205L	58.97	194L	58.23	-15	15	1.008	-	1.017
NoCB-208L	54.11	194L	58.23	-10	10	0.926	-	0.932
NoCB-206L	61.85	194L	58.23	-15	15	1.058	-	1.066
DeCB-209L	64.81	194L	58.23	-15	15	1.109	-	1.117
TrCB-28L CU	17.35	9L	19.37	-10	10	0.887	-	0.904
PeCB-111L CU	31.59	101L	27.90	-10	10	1.126	-	1.138
HpCB-178L CU	40.12	138L	39.43	-10	10	1.013	-	1.022
DiCB-9L IS	10.63	138L	39.43	-12.5	12.5	0.264	-	0.275
TeCB-52L IS	19.37	138L	39.43	-12.5	12.5	0.486	-	0.497
PeCB-101L IS	27.90	138L	39.43	-12.5	12.5	0.702	-	0.713
HxCB-138L IS	39.43	138L	39.43	-50	50	0.979	-	1.021
OcCB-194 IS	58.23	138L	39.43	-12.5	12.5	1.472	-	1.482

The retention time limits are applied to the daily 209 mix resulting in RRT limits for calibrations and samples, including QC samples.
OcCB-194 IS upper limit example: (58.23+(12.5/60))/39.43=1.482

TABLE 9: METHOD HOLDING TIMES

Method	Collection to Extraction	Extraction to Analysis
8290A *	30 days	45 days
1613B	365 days	365 days
DLM02.2	365 days	365 days
M23	30 days	45 days
TO-9a	7 days	40 days
CBC01.2	35 days collect to analysis	
1668A/C	365 days	365 days

* NOTE: The holding times listed in method 8290 are recommendations. PCDDs and PCDFs are very stable in a variety of matrices, and holding times under the conditions listed in this section may be as long as a year for certain matrices.

Determination of Polychlorinated Biphenyls (PCBs) as Aroclors or Congeners by Gas Chromatography/Electron Capture Detection (GC/ECD)

References: Method 8082A, Rev. 1, February 2007 and Method 8000C Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final update III, December 1996 (USEPA, Office of Solid Waste and Emergency Response, Washington, DC).

Quality Assurance and Quality Control for SW-846 Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC) for the Massachusetts Contingency Plan (MCP)

Compendium Method TO10A – Determination of Pesticides and PCBs in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by GC/ECD Detection

1. Scope and Application

Matrices: Water, soil, sediment, sludge, product, PUF, and tissue (either animal or vegetable)

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the analysis and quantification of sample extracts for Polychlorinated Biphenyls (PCBs) as Aroclors or Congeners as well as Total PCB by gas chromatography/electron capture detector. See Table I for the target compound list of PCB as Aroclors and PCB Congeners with CAS Registry Number included. The sensitivity of the method usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, the method may also be performed on samples that have undergone cleanup.

Quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment. Therefore, the 8082 method provides procedures for the determination of selected individual PCB Congeners and should be used depending on regulatory requirements and project needs. The PCB Congener approach potentially affords greater quantitative accuracy when PCBs are known to be present. The PCB Congener method is of particular value in determining weathered Aroclors. Caution should be used when using the PCB Congener method where regulatory requirements are based on Aroclor concentrations. The base list of components can be found in Table I. Additional congeners may be added to this base list of targets. Additional targets must be run under the same GC conditions to document retention times on both analytical columns. Congeners may be added without additional method proficiency demonstration.

The following extraction and cleanup methods may apply, prior to sample analysis:

- *Extraction of Water Samples by Separatory Funnel* (2156),
- *Microscale Solvent Extraction - 3570* (2172)
- *Soxhlet Extraction (2173)*
- *Soxhlet Extraction of PUFs (*2167*)*
- *Tissue Preparation and Homogenization* (2166),
- *Shaker Table Extraction* (2261),
- *Sulfur Cleanup* (2168),
- *Sulfuric Acid Cleanup* (2169),
- *Silica Gel cleanup* (2170)
- *GPC Cleanup by HPLC* (2167)

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC-ECD and in the interpretation of GC-ECD data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Sample extracts are analyzed on a gas chromatograph which is fitted with two capillary columns of differing polarities each employing separate detectors. The extracts of PCB Congeners are spiked with internal standard prior to analysis. The target analytes are resolved on each column and detected using an electron capture detector (ECD). Analytes are introduced into the GC/ECD by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC which is temperature and flow programmed to separate the analytes. Identification of target analytes is accomplished by confirming a target hit on two dissimilar columns using Retention Time (RT) and Pattern Recognition (PR). Concentrations are calculated from the ECD response using either external (for PCB Aroclors) or internal (PCB Congeners) standard techniques. For all PCB Aroclors, the concentrations are derived from a multi-level calibration curve, either linear using a least square regression, or non-linear.. For PCB Congeners, the concentrations are determined using mean Relative Response Factor derived from multi-level calibration curve. Alternatively, different calibration models may be employed for either Congeners or Aroclors.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Generally, the Reporting Limit for aqueous samples is 0.02 ug/L for individual Aroclors and 0.001 ug/L for individual Congeners. This value is based on a 1mL final volume and 500 or 1000 mL of sample extracted. The Reporting Limit for solid/tissue samples is 4 ug/Kg for individual Aroclors and 0.2 ug/Kg or for individual Congeners, based on 1 mL final volume and 5 g of the sample extracted (wet weight) using extraction method 3570 (MSE). Utilizing extraction method 3540C (Soxhlets) the achievable RL for client samples at a 30g extraction weight is 1.33 ug/Kg or for individual Congeners, based on 4 mL final volume. The Reporting Limit for Aroclors in PUF air samples is 20-100ng/PUF cartridge, based on the Soxhlet extraction of one low volume PUF and a final volume of 1mL. Detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed.

4. Interferences

4.1 Contaminants in solvents, reagents, glassware, syringes, and other sample processing hardware may cause inferences that lead to discrete artifacts, biases, and/or elevated baselines. Demonstrate that all of these materials are free from interferences under the conditions of the preparation and analysis by extracting and analyzing a laboratory method blank with each batch of up to 20 samples. Also new solvent lots are prescreened and approved by GC/ECD analyst prior to use. All glassware and equipment should be properly cleaned and solvent rinsed prior to use. Never use any glassware, or other implements such as syringes, on multiple samples within the batch unless there has been thorough cleaning and decontamination between samples.

4.2 Contaminants co-extracted from the sample may cause matrix interferences. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the environment and matrix being investigated. In some cases depending on the severity of the observed interferences, a target hit on one column may be biased high when interfered with. Skilled analyst judgment is imperative in determining if a chromatographic peak appears interfered with. In these cases the non-interfered, non-biased hit can be forced for reporting purposes providing a more suitable value. In extreme cases of interference additional cleanups can be employed, or it may be necessary to analytically re-run the sample at a dilution.

4.3 The ECD is theoretically a halogen specific detector. However, phthalate esters can be a major source of contamination in that they do respond to the ECD and can interfere with accurate determination of the analytes of interest. No forms of plastic or any material containing plasticizers (phthalates) should be used in conjunction with any part of this analysis.

4.4 Elemental sulfur is readily extracted from soil samples and may cause chromatographic interferences in the determination of pesticides. Sulfur can be removed through the use of Method 3660B. In addition, all samples for PCB analysis are acid cleaned following Method 3665A. Associated SOPs are noted in Section 1.0, above.

4.5 Care must be taken to ensure a complete hexane exchange is performed on the extract, because residual methylene chloride would be an interferent in the analysis. Residual methylene chloride can also damage the electron capture detectors.

4.6 The injector system of the gas chromatograph can become contaminated with non-volatile components from samples. This can act to decrease the resolution and responsiveness of the system. Maintenance of the analytical system should be performed when system performance degrades (liner changes, injection port cleaning, etc.) to avoid prolonged down time.

4.7 Aldol condensation products (4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) can arise during soil extraction when using methylene chloride:acetone. These products may interfere with detection of analytes and should be monitored.

4.8 Raw GC data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

4.9 Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized in the oven and spread to other glassware. Therefore, exercise caution, and do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analyses, unless glassware has undergone meticulous cleaning.

4.10 Per method 8082A each analytical column analyzing for PCB congeners must be evaluated for the presence of coelutions amongst PCB congeners with pesticides. An aliquot of PCB congeners/pesticide LCS solution is prepared and analyzed routinely on each analytical column in the laboratory. The subsequent coelutions of these compounds are documented and stored internally in the “ECD Standard Verification Logbook” within the facility.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in 500mL, 1L or 2L amber glass bottles and stored without preservative at 4°C

1-5g of tissue sample are required.

Soil/sediment samples are collected in 4oz. glass soil jars,

Air PUF samples are collected following the guidance in reference method TO-10A Compendium.

6.2 Sample Preservation

Aqueous and Air PUF samples are stored without preservative at 4°C

Tissue Samples are stored at 4°C, or if desired, frozen.

Soil/sediment samples are stored at 4°C, or if desired, frozen.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

The hold time for this method is 7 days for the extraction of aqueous and Air PUF samples, and 14 days for the extraction of solid and tissue samples. If a sediment or tissue samples are frozen, this suspends the holding time until removal from the freezer. All extracts must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph: The instrumentation is a temperature-programmable gas chromatograph with heating range from 40°C to 325°C and splitless-type inlet system with electron capture detectors (Hewlett-Packard HP 5890 Series II GC, HP 6890 Plus, or similar).

7.1.1 The injection port is designed for splitless injection onto a capillary column. The injection port utilizes a uniliner for direct injection, to decrease the potential for breakdown of compounds in the injection port and to increase sensitivity. These uniliners are used in place of the standard splitless liners. The 6890 model requires a drilled uniliner due to Electronic pressure control. The injection port will require maintenance on an as needed basis if degradation or contamination is apparent.

7.1.2 Columns and supplies:

7.1.2.1 Restek's proprietary "RTX-5" Stationary Phase, 60m length x 0.25mm ID, 0.25μm film thickness - Restek Corporation, PN 10226, or equivalent.

7.1.2.2 Restek's proprietary "RTX-CLPesticide II" Stationary Phase, 60m length x 0.25mm ID, 0.2μm film thickness - Restek Corporation, PN 11326, or equivalent.

7.1.2.3 Guard column, deactivated fuse silica phenyl-methyl, 5 m x 0.32 mm ID. Restek PN 10027 or equivalent.

7.1.2.4 The flow from the guard column can be split between the two analytical columns using a Siltek MXT-Y connecter Restek PN 20396

7.1.3 The GC/ECD utilizes one injector with guard column sealed to the uniliner inside the injection port. Connector splits the guard column into the two dissimilar columns listed above. Each column is then attached to a separate detector. Rtx-5 is always the front detector column and the CLP II is the back detector column.

7.2 Autosampler: Adapted onto the Gas Chromatograph. Model is HP 6890 series autosampler with a GC autosampler controller and HP 5890 series autosampler with controller, or equivalent.

7.3 Computer: With Windows NT operating software utilizing HP Enviroquant G1701BA Version B.01.00 software.

7.4 Gases: Hydrogen, ultra high purity grade (99.9999% pure) and Nitrogen of equivalent purity. Hydrogen is generally supplied by a Proton Hogen GC 600 Hydrogen generator.

7.5 Glassware: Assorted Class-A volumetric flasks consisting of 5mL through 200mL flasks with ground-glass stoppers for preparing reagents, standards, and measuring sample volumes for sample requiring dilution beyond 1:100.

7.6 Pipettes: Disposable Pasteur brand, or equivalent.

7.7 Cahn balance: Capable of accurate measurement to the nearest 0.001 mg.

7.8 Analytical balance: Capable of accurate measurement to the nearest 0.0001 g.

7.9 Top-loading analytical balance: Capable of accurate measurement to the nearest 0.1g.

7.10 Micro syringes: 10, 25, 50, 100, 250, 500, and 1000 µL.

7.11 VOA vials: Non-preserved, certified, pre-cleaned VOA vials, 40 mL, with Teflon-lined screw caps.

7.12 Vials: 2 mL, with Teflon-lined crew caps for standard solutions (amber) and sample extracts (clear).

7.13 Spatula: Stainless steel.

8. Reagents and Standards

8.1 Solvents: All solvent expirations determined as indicated by manufacturer guidelines.

8.1.1 Methylene Chloride, ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (1816) for additional details regarding solvent purity.

8.1.2 **Acetone,** ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (1816) for additional details regarding solvent purity.

8.1.3 **Hexane,** ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (1816) for additional details regarding solvent purity.

8.1.4 **Methanol,** Purge and Trap grade, see SOP *Reagent, Solvent, and Standard Control* (1816) for additional details regarding solvent purity.

8.2 Primary Standard Solutions: All Primary standard solutions are purchased from commercial vendors as flame sealed ampule certified solutions. When a flame sealed ampule primary solution is opened, it is transferred to a labeled amber 2mL vial.

Primary Standards are stored as indicated by the vendor, away from light when not in use. Primary standards are discarded as indicated by the vendor expiration. If breakdown of a solution is observed, the solution will be discarded.

8.2.1 **Aroclor 1016 Solution:** Obtained from Accustandard at a concentration of 100ug/mL (catalog # C-216S-H-10X-PAK) – for the calibration curve and LCS and obtained from Restek at a concentration of 1000 ug/mL (catalog # 32006) – for ICV.

8.2.2 **Aroclor 1260 Solution:** Obtained from Accustandard at a concentration of 100ug/mL (catalog # C-260S-H-10X-PAK) – for the calibration curve and LCS and obtained from Restek at a concentration of 1000ug/mL (catalog # C32012) – for LCS and ICV.

8.2.3 **Aroclor Solution:** Obtained from Accustandard at a concentration of 100ug/mL or 1000ug/mL for the calibration curve.

Compound	Part Number
Aroclor 1221	APP-9-159
Aroclor 1232	APP-9-160
Aroclor 1242	C-242S-H-10X
Aroclor 1248	C-248S-H-10X
Aroclor 1254	C-254S-H-10X
Aroclor 1262	APP-9-165
Aroclor 1268	APP-9-166

8.2.4 **Aroclor Solution:** Obtained from Ultra at a concentration of 100ug/mL for ICV

Compound	Part Number
Aroclor 1221	PP-291
Aroclor 1232	PP-301
Aroclor 1242	PP-311
Aroclor 1248	PP-341
Aroclor 1254	PP-351
Aroclor 1262	PP-371
Aroclor 1268	PP-381

8.2.5 **Pesticides Surrogate Standard Spiking Solution:** Obtained from Ultra at a concentration of 200ug/mL (catalog # ISM-320) – for PCB Aroclors analysis.

8.2.6 4,4'-Dibromooctafluorobiphenyl Solution-DBOB: Obtained from Ultra at a concentration of 5000ug/mL (catalog # PPS-172) – for PCB Congeners analysis.

8.2.7 2,2',3,3',4,5,5',6-Octachlorobiphenyl Solution-BZ#198: Obtained from Ultra at a concentration of 100ug/mL (catalog # RPC-075S) – for PCB Congeners analysis.

8.2.8 Internal Standard Solution: 2,3,3',4,5,5',6 Heptachlorobiphenyl Solution-BZ#192 –obtained from Ultra at a concentration of 100ug/mL (catalog # RPC-165S) – for PCB Congeners analysis.

8.2.9 Custom PCB Standard: Obtained from Accustandard at a concentration of 100ug/mL (catalog # S-7911-2X) – for the calibration curve, for PCB Congeners analysis.

8.2.10 EPA PCB Congener Calibration Check Solution: Obtained from Ultra at a concentration of 0.2ug/mL (catalog # RPC-EPA) – for ICV, for PCB Congeners analysis.

8.3 Stock Standard Solutions: All Stock Standard Solutions are prepared volumetrically by diluting and mixing the appropriate primary standard solutions in hexane. Stock standards expire 1 year from the date of preparation or sooner if routine QC tests indicate a problem. All stock solutions are stored in the freezer (-20°C -10°C) in 10-40mL vials depending on the amount prepared. If breakdown of a solution is observed, the solution will be discarded.

8.3.1 Aroclor 1660 Calibration Stock Solution: Consists of the Aroclor 1016 and 1260 from Accustandard or equivalent, mixed with the extraction surrogates (TMX and DCB), at a stock concentration of 20,000ug/L for AR1660 and 1000ug/L for surrogates, in hexane. To prepare calibration stock solution, add 1mL of *Aroclor 1016/1260 Primary Solution* at 1000ug/mL and 0.5mL of *Pesticides Surrogate Standard Spiking Solution* at 200ug/mL to a 50mL volumetric flask. Dilute to the volume with hexane.

8.3.2 Aroclor 1660 ICV Stock Solution: Consists of the Aroclor 1016 and 1260 from Restek or equivalent, at a stock concentration of 20,000ug/L in hexane. To prepare calibration stock solution, add 1mL of an individual *Aroclor Primary Solution* at 1000ug/mL to a 50mL volumetric flask. Dilute to the volume with hexane.

8.3.3 Other Aroclors Calibration Stock Solution: Consists of the individual Aroclor from Accustandard or equivalent at a stock concentration of 20,000ug/L, in hexane. To prepare calibration stock solution, add 1mL of an *Aroclor Primary Solution* at 1000ug/mL to a 50mL volumetric flask. Dilute to the volume with hexane.

8.3.4 Other Aroclors ICV Stock Solution: Consists of the individual Aroclor from Ultra or equivalent at a stock concentration of 10,000ug/L, in hexane. To prepare calibration stock solution, add 1mL of an *Aroclor Primary Solution* at 100ug/mL to a 10mL volumetric flask. Dilute to the volume with hexane.

8.3.5 NOAA 22 Congeners Stock Solution: Consists of the NOAA 22 Congeners from Accustandard or equivalent, at the concentration of 5000ug/L in hexane. To prepare congeners stock solution, add 1.25mL of *Custom PCB Standard* at 100ug/mL to a 25mL volumetric flask. Dilute to the volume with hexane.

8.3.6 Pesticides Surrogate Stock Solution: Consists of Pesticides Surrogates (TMX and DCB) from Ultra or equivalent, at a stock concentration of 5000ug/L. To prepare pest surrogate stock solution add 0.625 mL of *Pesticides Surrogate*

Standard Spiking Solution at 200ug/L to a 25mL volumetric flask. Dilute to the volume with hexane.

8.3.7 DBOB and BZ 198 Surrogate Stock Solution: Consists of DBOB and BZ198 from Ultra or equivalent, at a stock concentration of 5000ug/L, in hexane. To prepare surrogate stock solution add 25uL of *DBOB Primary Solution* at 5000ug/mL and 1.25mL of *BZ#198 Primary Solution* at 100ug/mL to a 25mL volumetric flask. Dilute to the volume with hexane.

8.4 Working/Spiking Standard Solutions: All working/spiking standard solutions are prepared volumetrically by diluting and mixing appropriate primary or stock standard solutions in acetone, hexane or methylene chloride. Working/Spiking standards expire 6 months from the date of preparation or sooner if routine QC tests indicate the problem. All working/spiking solutions are stored in the freezer (-20°C -10 °C) in 10-40mL vials depending on the amount prepared. All spiking solutions must be assayed for use by analysis before release to the preparation lab. All compounds must be within 20% of their true value. If breakdown of a solution is observed, the solution will be discarded.

Aroclor 1660 calibration standard solution: Consists of Aroclor 1016 and 1260 from our "Aroclor 1660 **calibration stock solution"** combined into one standard called Aroclor 1660 and the extraction surrogates (TMX and DCB) through dilution of Aroclor 1660 Calibration Stock solution. The extraction surrogates will only be combined with the 1660 calibration. An ICAL is run for each of the nine Aroclors before analysis. A 7 pt calibration is analyzed for the 1660 while 1pt calibration is allowed per method for the other 7 aroclors.

7-Level Curve Preparation for Aroclors

Calibration Level	Aroclor Conc.	Surrogate Conc.	Volume of Std Added	Final Volume in Hexane
Stock	20 00µg/L	2000µg/L		
Level1	20µg/L	2.0µg/mL	0.1mL of Level7.	10mL
Level 2	50µg/L	5.0µg/L	0.25mL of Level7	10mL
Level 3	100µg/L	10µg/mL	0.5mL of Level7	10mL
Level 4	250µg/L	25µg/mL	1.25mL of Level7	10mL
Level 5**	500µg/L	50µg/mL	1.25mL of Stock	50mL
Level 6	1000µg/L	100µg/mL	0.5mL of Stock	10mL
Level 7	2000µg/L	200µg/mL	1mL of stock	10mL

** CCV Level

8.4.1 Congener Calibration standard solution: Consist of 22 NOAA Congeners and the extraction surrogates (DBOB and BZ#198) through dilution of *NOAA 22 Congeners Stock Solution* listed in *8.3.2* and *DBOB and BZ 198 Surrogate Stock Solution* listed in *8.3.4.* 8 levels are described, however a minimum 5 levels are required for initial calibration.

Multi-Level Calibration Solution Preparation for Congeners and Surrogates

Calibration Level	Analyte Conc. ug/L	Surrogate Conc. ug/L	Volume of Std Added	Volume in Hexane
Level 1	1	1	0.05mL of Level 8	10mL
Level 2	5	5	0.25mL of Level 8	10mL
Level 3	10	10	0.50mL of Level 8	10mL

Level 4	20	20	0.04mL of Cong stock and 0.04mL of Surr. stock	10mL
Level 5	50	50	0.1mL of Cong stock and 0.1mL of Surr. stock	10mL
Level 6**	100	100	0.5mL of Cong stock and 0.5mL of Surr. stock	25mL
Level 7	150	150	0.3mL of Cong stock and 0.3mL of Surr. stock	10mL
Level 8	200	200	0.4 mL of Cong stock and 0.4 mL of Surr. stock	10 mL

** CCV Level

8.4.2 PEST/PCB Surrogate Spike Solution: TMX and DCB obtained from Ultra Scientific or equivalent, at a concentration of 1000ug/L in Acetone. To prepare:

- Surrogate spiking solutions add 1mL of *Pesticides Surrogate Standard Spiking Solution* (at 200ug/mL) into a 200mL volumetric flask and dilute to the volume with acetone.

All samples, blank and matrix spike are spiked with 1mL of the surrogate solution prior to extraction to monitor efficiency of sample extraction, chromatographic and calibration system. This amount may be adjusted to meet project specific concentrations, as needed.

8.4.3 PEST/CONG Surrogate Spike Solution: DBOB and BZ 198 obtained from Ultra or equivalent, at a concentration of 100ug/L in Acetone. To prepare surrogate spiking solutions add 5mL of DBOB and BZ 198 Surrogate Stock Solution (at 5000ug/L) into a 250mL volumetric flask and dilute to the volume with acetone. All samples, blank and matrix spike are spiked with 1mL of the surrogate solution prior to extraction to monitor efficiency of sample extraction, chromatographic and calibration system. This amount may be adjusted to meet project specific concentrations, as needed.

8.4.4 PCB Aroclors LCS/LCSD/MS/MSD: Aroclor 1660 commercially obtained from Accustandard at the concentration of 10,000ug/L in Acetone. To prepare:

Spiking solution add 1mL of each primary solution at 1000μg/mL to a 100mL volumetric flask and bring it to volume with Acetone.

1mL is spiked into each QC and field QC sample. This amount may be adjusted to meet project specific concentrations, as needed.

8.4.5 NOAA 22 Cong LCS/LCSD/MS/MSD: NOAA 22 Congeners obtained from Ultra or equivalent, at the concentration of 100ug/L in Acetone. To prepare spiking solution add 1mL of NOAA 22 Congeners Stock Solution at 5000μg/L to a 50mL volumetric flask and bring it to volume with Acetone. 1mL is spiked into each QC and field QC sample. This amount may be adjusted to meet project specific concentrations, as needed.

8.4.6 Independent Verification Standard (ICV) for Aroclors: This is a source separate from the calibration curve containing one of the nine Aroclors, obtained from Ultra or Restek. Prepare from Stock Solution by diluting in hexane to the concentration of 500ug/L

8.4.7 Independent Verification Standard (ICV) for Congeners: This is a source separate from the calibration curve obtained from Ultra, at the concentration of 100ug/mL in hexane. This standard must be analyzed with the appropriate concentration of the internal standard.

8.4.8 **Internal Standards (IS): BZ#192:** Prepared so that a 20-uL spiked into the sample extract prior to analysis, will produce a mid-calibration range response at 50ng/mL in final sample extract. To prepare add 0.625mL of primary solution at 100ug/mL to a 25mL volumetric flask and bring it to volume with hexane. Final concentration is 2.5ug/mL.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Quality Control (QC) samples are necessary to monitor both the sample extraction and instrument analysis procedures. The Quality Control samples described below are considered the method defaults, and are the minimum requirements, except where noted. Client and Project specific Data Quality Objectives (DQOs) supersede the requirements in this section where applicable. Client or Project specified DQOs shall be included, or referenced, in the final report to the client.

9.1 Blank(s)

9.1.1 A method blank must be extracted (spiked with surrogates and/or internal standards) and analyzed once per every 20 samples or per extraction batch, whichever is more frequent. The analysis of the method blank extract will demonstrate the background contamination.

9.1.2 An *acceptable* method blank should not contain any individual compound at the concentration of reporting limit, or above. All efforts must be made to identify and eliminate the source of contamination. The presence of analytes at concentrations at or above the reporting limit will warrant application of a “B” qualifier to that target compound(s) on all associated report forms, and perhaps re-extraction of all associated samples. The results are qualified with a “B” for any associated sample concentrations that are less then 10x the blank concentration for the analyte. Surrogate and internal standard recoveries must meet the QC limits for the method blank, see Sections 12.5 and 12.6. Re-extraction must be initiated immediately so that the minimum of time is wasted before re-extraction can occur - if at all possible, this re-extraction should take place within holding time. Re-extraction *corrective action* that would exceed the sample holding time criteria should be discussed with the client, Laboratory Director, QA Manager, and/or Section Supervisor prior to implementation. Exceptions may be made with approval of the Section Supervisor if the samples associated with an out of control method blank are non-detect for the affected compound(s) or if the concentrations of the affected compound(s) are greater than 5x the blank level in the samples. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified, via the project narrative, of any method blank non-compliance associated with the sample results. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

9.2 Laboratory Control Sample / Laboratory Control Sample (LCS / LCSD)

9.2.1 The LCS/ LCSD are extracted along with the samples as a measure of precision and accuracy. At a minimum, one LCS/LCSD sample set must be analyzed at a frequency of once per 20 samples, or per analytical batch, whichever is more frequent. The LCS contains Aroclor 1660 for PCB's as Aroclors analysis and all 22 NOAA Congeners for PCB Congeners analysis.

9.2.2 The acceptable recovery QC limits are found in Section 12 for an aqueous, solid, and tissue LCS.

9.2.3 Acceptable relative percent difference (RPD) for LCS/LCSD analysis is ± 50 % for both aqueous and solid matrices. See section 12 for QA acceptance criteria. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

9.2.4 Corrective Action: Repeat analysis or check to see if an analytical error has occurred. If the LCS recovery is still out of control, re-extract and re-analyze the LCS and all samples associated with that LCS/LCSD. If the LCS/LCSD do not meet quality control criteria for recovery of some of the spike and surrogate compounds, the results for the other samples and quality control samples within the batch must be evaluated to determine if this is an isolated problem for the LCS/LCSD and whether the data should be reported with the affected LCS/LCSD. If the recoveries for all components in the LCS/LCSD are outside of criteria, the entire analytical batch should be re-extracted and re-analyzed. Exceptions may be made with approval of the Department Manager if the samples associated with the out of control LCS/LCSD are also associated with a matrix spike and matrix spike duplicate that is in control which demonstrates an isolated problem pertaining to the LCS/LCSD only. An explanation of this out of control LCS/LCSD recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS/MSD results as batch QC. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

9.3 Initial Calibration Verification (ICV)

Refer to section 10.2

9.4 Continuing Calibration Verification (CCV)

Refer to section 10.4

9.5 Matrix Spike / Matrix Spike Duplicate (MS / MSD)

9.5.1 MS/MSD analyses are performed once per 20 samples (5% frequency) only per client request. It is preferable to extract samples that have been selected specifically by the client. If none have been assigned then the laboratory analyst must choose a representative sample for each type of matrix prepared. The MS/MSD must be matrix specific. Aqueous and air PUF MS/MSD cannot be done unless client supplies sufficient sample. These samples are also spiked with extraction surrogate. MS/MSD are spiked with the LCS spiking solution (Aroclor 1660 or NOAA 22 Congeners) at the same level as LCS/LCSD.

9.5.2 The acceptable recovery and RPD QC limits are found in Section 12 for an aqueous, solid, tissue and product MS/MSDs.

9.5.3 Acceptable relative percent difference (RPD) for matrix spike analysis is ± 50 % for both aqueous and solid matrices. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

9.5.4 Corrective Action: The recovery of the matrix spikes are evaluated relative to the what was in the unspiked sample to indicate how well the methods worked on extraction of the analytes of interest from the sample matrix. The results for the unspiked and MS/MSD samples need to be compared carefully to determine how well the spiking study worked and if there was any indication of matrix-related problems that might affect the analytical batch. Re-extraction of the sample based on the MS results is rare. Repeat analysis or check to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the control limits and the associated LCS is within control, include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the affected results as evidenced by the matrix spike and matrix spike duplicate exceedance. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

9.6 Laboratory Duplicate

9.6.1 Laboratory matrix or sample duplicates are analyzed if requested by the client. The client must supply sufficient additional sample volume for this duplicate. Duplicates are used to evaluate the precision of the method. The QC limit is 50% RPD for target compounds found above 5 times the reporting limit.

9.6.2 Corrective Action: If the %RPD exceeds the 50% control limit and the associated MS/MSD %RPD is within 50%, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results isolated to this sample, as evidenced by the matrix duplicate exceedance and the MS/MSD acceptance. If both the sample/duplicate and the MS/MSD exceed the control limits, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results as evidenced by the sample/duplicate and the MS/MSD exceedences. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

9.7.1.1 Surrogates are monitored for recovery for all matrices. Two surrogates are used, one that elutes at the beginning and one at the end of GC run: TMX/DCB for Aroclors and DBOB/BZ#198 for Congeners. Every sample, blank, and quality control sample (LCS, MS, MSD, Dup) must be spiked

with the surrogates prior to extraction. The recovery limits are found in Section 12.

9.7.1.2 Corrective Action: Check to see if an analytical, spiking, or dilution error occurred and re-calculate. If only one surrogate falls below the recovery limit and all QC samples have acceptable recovery indicating a matrix effect isolated to the sample, the exceedance is noted, with approval of the Section Supervisor, and the results are reported to the client with a notation in the case narrative. If all surrogates are recovered below the limit or there are outliers in any QC, re-extract the sample. If re-extraction occurred beyond the holding time and the re-extract surrogates are within the QC limits, report the re-extract results along with the original results. If the surrogates are recovered below the limit in the re-extract, this confirms suspected matrix interference on the surrogates, and only the original analysis needs to be reported unless a specific QAPP requires otherwise. If the chromatogram shows obvious matrix interference, no re-analysis or re-extraction is necessary. *This decision must be made with approval of the Section Supervisor and/or Project Manager.* Surrogate outliers and sample re-extracts must be noted in the case narrative to the client.

9.7.2 Internal Standards

9.7.2.1 Internal Standards will be evaluated for relative retention time drift and relative area counts compared to the associated opening CCV for PCB Congeners *only.* Internal standards are added to every field sample, QC sample, standard, and method blank. The internal standard retention times for associated samples should be ± 0.05 minutes relative to the retention times from the associated opening CCV. The area counts for the internal standards in the associated samples should be +/-50% of the area of the internal standard calculated during initial calibration. The daily opening CCV IS areas are also compared to the ICAL.

9.7.2.2 Corrective Action: Check to see if an analytical, dilution or spiking error occurred. If the chromatogram shows obvious matrix interference, no re-analysis is necessary. *This decision must be made with approval of the Department Manager.* Note the exceedance in the case narrative to the client. If no obvious interference is present, re-analyze the extract. If internal standards are now within the acceptance limits, report only the re-analysis, as long as the re-analysis occurred within the 40-day analytical hold time. If the re-analysis occurred outside of the 40-day analytical hold time, both the original and re-analysis must be reported. If the internal standards again are outside the acceptance limits, after re-analysis, either within or outside of the 40-day hold time, report only the original analysis, and include a narrative to the client that the suspected matrix interference on the internal standards was confirmed by sample re-analysis. If specified by the client or project data quality objectives (DQOs) or other specifications, both results of both analysis might have to be reported.

9.7.3 Standard Reference Materials (SRMs)

9.7.3.1 Standard reference materials (SRMs) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples on a project specific basis. For sediment and

tissue sample batches, a certified SRM will be analyzed at the rate of one per batch of up to 20 samples per matrix for PCB Congeners *only.* These are not used as controls, but to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

9.7.3.2 Measured results will be compared against certified values for those target analytes that are certified in these SRMs as follows. Acceptance criteria for SRM analysis will vary from project to project depending upon client data quality objectives (DQOs). Generally, ± 50% difference (%D) based on the true certified values of the target compounds of interest, or 40% - 140% recovery, serve as advisory acceptance criteria.

9.7.3.3 Corrective Action: Repeat analysis and/or check to see if an analytical error has occurred. If the % recovery or %D still exceeds the control limits and the associated LCS/LCSD and/or MS/MSD are within control, include a project narrative with the results to client noting that the observed recovered of the SRM are isolated to this sample as evidenced by the LCS/LCSD and/or MS/MSD acceptance. Individual analyte exceedances will be narrated and may be used to assess potential interferences or system bias for individual analytes.

9.8 Method Sequence

An example sequence for 8082 Aroclor analysis, created 12/04 on ECD4. CCV identifiers and/or nomenclature can be associated according to date/instrument or a specifically generated WG (workgroup) reference number assigned by the company LIMS system.

1.	Primer	
2.	Hexane	
3.	C4120401STD1242	AR1242 Level 5 @ 500ug/L *
4.	C4120401STD1248	AR1248 Level 5 @ 500ug/L *
5.	C4120401STD1254	AR1254 Level 5 @ 500ug/L *
6.	C4120401STD1660	AR1660 Level 5 @ 500ug/L
7.	C4120401STD2162	AR1262 Level 5 @ 500ug/L *
8.	C4120401STD3268	AR1268 Level 5 @ 500ug/L *
9.	Method Blank	
10.	Method LCS	
11.	Method LCSD	
12.	7 client samples	
13.	C4120402STD1242	AR1242 Level 5 @ 500ug/L *
14.	C4120402STD1248	AR1248 Level 5 @ 500ug/L *
15.	C4120402STD1254	AR1254 Level 5 @ 500ug/L *
16.	C4120402STD1660	AR1660 Level 5 @ 500ug/L
17.	C4120402STD2162	AR1262 Level 5 @ 500ug/L *
18.	C4120402STD3268	AR1268 Level 5 @ 500ug/L *
19.	about 10 client samples	
20.	C4120403STD1242	AR1242 Level 5 @ 500ug/L *
21.	C4120403STD1248	AR1248 Level 5 @ 500ug/L *
22.	C4120403STD1254	AR1254 Level 5 @ 500ug/L *
23.	C4120403STD1660	AR1660 Level 5 @ 500ug/L
24.	C4120403STD2162	AR1262 Level 5 @ 500ug/L *
25.	C4120403STD3268	AR1268 Level 5 @ 500ug/L *

*optional CCV levels may be employed to account for the presence of variable Aroclor patterns within client samples.

An example sequence for 8082 Congener analysis, created 12/04 on ECD3. CCV identifiers and/or nomenclature can be associated according to date/instrument or a specifically generated WG (workgroup) reference number assigned by the company LIMS system.

Primer
Hexane
C3120401STD50
Method Blank
Method LCS
Method LCSD
7 client samples
C3120402STD50
10 client samples
C3120403STD50

10. Procedure

10.1 Equipment Set-up

10.1.1 Prior to performing GC/ECD analysis, the operator must read and become familiar with the operating procedure guidelines specified in the instrument operating manuals. The analyst must be trained and familiarized with the instrument software

provided by the manufacturer. The instrument must be set up with the proper operating parameters (data acquisition and processing) and conditions described in the operating manual. The criteria for the analytical calibration ranges and method and instrument detection limits must be established and documented prior to initial calibration. All analytical equipment is traceable to NIST via internal and external calibration checks and *Certificates of Analysis* that are received with the calibration standards.

10.1.2 GC Instrumental Conditions

10.1.2.1 Inject an aliquot of 1uL into the capillary column of the gas chromatograph at the following conditions.

Parameters	Settings for Aroclors Analysis	Settings for Congeners Analysis
Injector A Temp	220°C	220°C
Detector Temp	300°C (HP)	300°C (HP)
Oven Temp	125°C	100°C
Initial Value	125°C	100°C
Initial Time	0 minute	0 minute
Rate	25°/minute to 197°C	25°/minute to 197°C
Hold. Time	0 minutes	2.00 minutes
Rate	5°/minute to 280°C	1°/minute to 210°C
Hold. Time	8.50 minutes	0 minutes
Rate	-	5°/minute to 210°C
Hold. Time	-	7.12 minutes
Purge/Valve A ON	OFF	OFF
Run Time	27.98 minutes	40.00 minutes
Mode	Splitless	Splitless

10.1.2.2 Establish daily retention time windows as the retention time of the component in the opening verification standard ± 0.05. Retention time windows are calculated each time a new GC column is installed. It has been found that this window works well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results. The window listed above is used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms.

10.2 Initial Calibration

10.2.1 Before analysis of sample extracts, establish a multi-point calibration curve showing the linear range of the analysis for all nine Aroclors. The ICAL for 1660 will have extraction surrogates added in the solution. Each other Aroclor will have an individual curve. The calibration curve for 1660 and associated surrogates are generated during initial calibration through the analysis of, at minimum, 5-level calibration standards, which define the working range of the method. A 1pt calibration curve is employed for the other 7 aroclors. See Section 8 for the preparation of the standard solutions for the initial calibration curve.

- The method of external standard calibration is used ***for Aroclors*** analysis. The response of each individual peak and the total response of the PCB in the sample are compared to a calibration curve to determine the analyte concentration in the sample.

  * Using the GC system software, the analyst must choose 3-5 peaks from the pattern which are characteristic of the Aroclor and are the least subject to degradation and weathering to obtain the response for the component of interest. Choose peaks that are at least 25% of the height of the largest Aroclor peak, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. Use at least 5 peaks for the AR1660, none of which should be found in both. Tabulate the peak area for each analyte against the mass injected of the analyte in the standard analyzed to obtain a calibration curve for each analyte across the working range of the method.

- Calibration Factors or Response Factor are determined for individual peaks. Choose 3 to 5 peaks in the standard to calculate calibration factors for each Aroclor. The calibration factors are then used to calculate the concentration of each corresponding peak in the sample. The 3 to 5 resulting concentrations are averaged to provide the final result for the Aroclor for the sample.

- The method of internal standard calibration is used ***for Congener*** analysis. The response of each individual peak relative to the internal standard respond is compared to a calibration curve to determine the analyte concentration in the sample.

10.2.2 The following analytical sequence order should be followed for initial calibration of the system. The primer is used at the beginning of the analysis sequence to ensure all sites of activation in the system have been covered or deactivated prior to performing trace level analysis. The sequence below is a general sequence for determination of PCBs as Aroclors and uses a minimum of 5 levels for AR1660 and a single-point initial calibration for the remaining Aroclors near the mid-point of the expected calibration range. Typically, a 7 point calibration is employed for AR1660. Construct an analytical sequence using the HP Enviroquant software:

- Click on the “Instrument” icon to open the Chem Station
- *Go into the “GC Top Environmental” screen*
- *Go into “Sequence”*
- Edit the “Sample Table Log” by entering the “Vial” number starting at position 1, the “Data File ID” same as the “Sample Name” (for the initial calibration curve the nomenclature is “I” for initial calibration “4” for instrument ID ECD4, the date, and the standard number “01, 02, etc.” such as, I4120401, I4120402, etc.), the

acquisition method (A412041660.m), which indicates the type of method and the date the method was created), and finally the "Miscellaneous Information" (such as, "PW120407A 20ug/L", for the first standard ID and concentration level, etc.). When complete, click "OK".

- Go back into "Sequence" and "Save" the sequence as the date, such as, C4120401.s. The ending "01" indicates the first sequence created on 12/04.
- Go back in to "Sequence" and "Print" the sequence that was just saved. This will become part of the instrument run log. See Section 15.0 for additional instrument run log details.
- Go back into "Sequence" and "Load and Run" the sequence that was just saved.

Example initial calibration sequence for Aroclor analysis, created on 11/16 on ECD4:

1. Primer
2. Hexane
3. I4111611011660L1 AR1660 (PW111611O @ 20ug/L)
4. I4111611011660L2 AR1660 (PW111611N @ 50ug/L) - optional
5. I4111611011660L3 AR1660 (PW111611M @ 100ug/L)
6. I4111611011660L4 AR1660 (PW111611L @ 250ug/L)
7. I4111611011660L5 AR1660 (PW111611K @ 500ug/L)
8. I4111611011660L6 AR1660 (PW111611J @ 1000ug/L)
9. I4111611011660L7 AR1660 (PW11616I @ 2000ug/L)
10. HEXANE
11. I411611STD500 AR1660 (PW092711A @ 500ug/L)
12. HEXANE
13. I4111611011221/1262L5 AR1221/1262 (PW040814R@ 500ug/L)
14. I4111611011221/1262ICV AR1221/1262 (PW032814D @500ug/L)
15. I4111611011232/1268L5 AR1232/1268 (PW031314A@500ug/L)
16. I4111611011232/1268ICV AR1232/1268 (PW032814E @500ug/L)
17. I4111611011242L5 AR1242 (PW040714@500ug/L)
18. I4111611011242ICV AR1242 (PW032814A @500ug/L)
19. I4111611011248L5 AR1248 (PW040714C@500ug/L)
20. I4111611011248ICV AR1248 (PW032814B @500ug/L)
21. I4111611011254L5 AR1254 (PW030614 @500ug/L)
22. I4111611011254ICV AR1254 (PW030614H @500ug/L)

Example initial calibration sequence for Congener analysis, created on 02/04 on ECD3:

1. Primer
2. Hexane
3. I3020401STD1 Level 1 (PW020407A @ 1ug/L)
4. I3020402STD5 Level 2 (PW020407B @ 5 ug/L)
5. I3020403STD10 Level 3 (PW020407C @ 10ug/L)
6. I3020404STD20 Level 4 (PW020407D @ 20ug/L)
7. I3020405STD50 Level 5 (PW020407E @ 50ug/L)
8. I3020406STD100 Level 6 (PW020407F @ 100ug/L)
9. I3020407STD150 Level 7 (PW020407G @ 150ug/L)
10. I3020408STD200 Level 8 (PW020407H @ 200ug/L)
11. HEXANE
12. I3020409STD100 ICV (PW120407J @ 100ug/L)

10.2.3 When the sequence has finished running, the Enviroquant software will generate "Not Reviewed" quantitation reports. All reports must be "Quant Reviewed" before they can become part of the initial calibration acquisition method for sample analysis.

- Enter into the "Environmental Data Analysis" (off-line) screen.
- Go to "File" and under method select the method (A302041660.m) that the initial calibration standards were analyzed under, then select the first data file.
- Go into "Quant" and select "QEdit Quant Results" to process the data files. Refer to SOP 2160 for details and processing of PCB Aroclor/Congener standards.
- When processing is complete for the first standard, "Save" the changes and "Exit." Re-print the re-processed data file by "Generating Quant Report," and save the hard copy for each level of the initial calibration.
- Repeat these steps for all initial calibration standards analyzed within the sequence.
- When all levels have been processed, go into "IntiCal," and select "Update Levels," and enter all levels for the initial calibration at the proper concentrations.
- After all responses are entered, "Save" the completed method and print the resulting response factor summary by selecting "Response Factors to Printer."

10.2.4 Acceptance Criteria:

- If average calibration factor or average relative response factor used, % RSD $\leq$ 20% over the working range of the curve for all analytes.
- If calibration curve used, the correlation coefficient must be $\geq$ 0.99 – for linear model or the COD or ^{r2} must be $\geq$ 0.99 – for non-linear model**.** Statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approach. A quadratic model requires six standards, and a third order polynomial requires seven standards. In setting model parameters, do not force the line through the origin. The COD or r^2 must be $\geq$ 0.99. The analyst should select the regression order that introduces the least calibration error into the quantitation.

10.2.5 The following *corrective actions* are recommended for failing initial calibrations:

- Check the calibration of the standard preparation that was performed. If the problem appears to be isolated to a single standard, the standard may be reanalyzed within twelve hours.
- Perform instrument maintenance and repeat the initial calibration.

The choice of corrective action must be made in consultation with the Department Manager, QA Manager, Project Manager, and/or the client.

10.2.6 Complete the initial calibration by filling out the *Initial Calibration Checklist*. The initial calibration, along with any corresponding continuing calibration data and sample data, is then forwarded for secondary review.

10.2.7 Initial Calibration Verification - ICV (separate source)

The analysis of separate source standard must follow the initial calibration curve. All target analytes in this verification standard are at the mid-point concentration. Note: If this standard was analyzed immediately following the initial calibration curve, it must be re-"Calculated and Generated" in the "QEdit" field, against the completed initial

calibration curve method and then re-processed, before it can be reviewed for acceptability.

Acceptance Criteria: ICV has to be analyzed from the separate source and % recoveries must be +/-20% for all targets. Aroclors must use the average of the 5 chosen/representative peaks to be +/-20%.

10.3 Equipment Operation and Sample Processing

Samples are prioritized by analyst for analysis based on hold time and client due date. All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

10.3.1 Samples are retrieved from the sample storage refrigerator, spiked with 20uL of the chosen internal standard solution per 1mL extract – for congeners only, sulfuric acid cleaned and loaded into the instrument autosampler trays following the generalized sequence below.

10.3.2 Analyze extracts using the same experimental conditions used for the analysis of the calibration standards. Ensure that calibration verification standards are interspersed, at least, every 10-20 samples or every 12 hour period. Area measurements should all be to baseline unless an unresolved complex mixture (UCM) is observed, in which case, the areas measured should skim the top of the UCM.

1. Primer

2. Hexane

3. AR1660/ Congener CCV – C1060501 @ 500ug/L/50ug/L

3*. AR1242/1248/1254 1-point ICAL levels @ 500ug/L (optional)

4. 10 samples + QC's

5. AR1660/ Congener CCV – C1060501 @ 500ug/L/50ug/L

6*. AR1242/1248/1254 CCV @ 500ug/L (optional)

7. 10 samples

8. AR1660/ Congener CCV – C1060501 @ 500ug/L/50ug/L

9*. AR1242/1248/1254 CCV @ 500ug/L (optional)

10.3.3 After sample analysis, "Not Reviewed" quantitation reports are generated by the software system. Samples are processed from "Not Reviewed" data files, to "Quant Reviewed" data files in a similar way the standards were previously processed. See Section 11 for details on sample processing. If a CCV fails the criteria outlined in Section 10.4.4, all samples since the last acceptable CCV must be re-analyzed (refer to Section 10.4.5).

10.3.4 If the on-column concentration of any compound exceeds the calibration range of 2000ug/L – for Aroclors and 200ug/L – for Congeners, the sample must be diluted, re-spiked with the appropriate amount of internal standard and re-analyzed. Assuming all samples are at a 1mL final volume, the following example dilutions would apply. Adjust the volumes accordingly for other sample final volume amounts and other desired dilutions.

1:2 dilution = 500uL of sample : 500uL Hexane and 10uL of IS
1:4 dilution = 250uL of sample : 750uL Hexane and 15uL of IS
1:5 dilution = 200uL of sample : 800uL Hexane and 16uL of IS
1:10 dilution = 100uL of sample : 900uL Hexane and 18uL of IS, etc.

10.4 Continuing Calibration

The systems performance and calibration for each compound of interest must be checked and verified before sample analysis. A continuing calibration verification (CCV) standard, at the concentration of the mid-level of the initial calibration curve, must be analyzed at the beginning and end of every analytical sequence, and every 12 hours or at the minimum of every 20 samples whichever is more frequent. However, it is recommended that the CCV is run every ten samples to minimize the number of samples requiring reanalysis.

10.4.1 Create the sequence: "Edit" the "Sample Table Log" to include the CCV standard (C1060501, where "C" is for CCV, "1" is for instrument number 1, followed by the date, and "01" for the first CCV analyzed this day), and acquire the CCV against the correct initial calibration method. "Save," then "Load and Run" the sequence.

10.4.2 When the sequence has finished running, the Enviroquant software will generate a "Not Reviewed" quantitation report. All reports must be "Quant Reviewed" against the acquisition method for sample analysis.

- Enter into the "Environmental Data Analysis" (off-line) screen.
- Go to "File" and under method, select the method that the CCV was analyzed under, then select the CCV data file.
- Go into "Quant" and select "QEdit Quant Results" to process the CCV file. please refer to SOP 2160 for manual integration details and processing of PCB standards
- When processing is complete, go into "ConCal," and select "Evaluate Data File as Continuing Calibration".
- Establish daily retention time windows as the retention time of the component in the opening verification standard ±0.05 min window.

10.4.3 Acceptance Criteria: Compare the CCV resulting response against the average response for the initial calibration for each calibrated analyte and calculate the % difference (%D). See Section 11 for the calculations. If the %D $\leq \pm$ 20%, sample analysis may take place following this verification standard. For arochlors the average of the 5 representative peaks is used to calculate the %D. If a noncompliant analyte exhibits an increase in response in the noncompliant CCV and is not detected in the associated samples, the analyses may be reported and the CCV noncompliance narrated. In this case CCV data or a detailed narrative must be provided to the client. Otherwise, corrective action must take place.

10.4.4 If the CCV does not meet the criteria for each calibrated analyte, the following *corrective actions* are recommended:

- Check the standard preparation. If the problem appears to be isolated to a single standard reanalyzed the standard
- If the verification is the beginning standard of the sequence, corrective action may consist of prepping the injection port (replace injection liner, clip column, gold seal, and septa) and re-analysis of the standard. If, after re-analyzing the standards, the acceptance criteria are still not met, a new initial calibration must be performed. The system must be in-control with all calibration criteria before sample analysis may proceed.
- If the verification is the ending standard, corrective action depends upon the severity of the verification results. For Congeners the closing CCV is not required if all samples are acquired within 12hr and IS areas met the criteria in section

9.7.2.1. Aroclors still need closing CCVs to meet +/-20%D, with the average of the 5 representative peaks. The closing CCV can be re-analyze once to verify the results (e.g. if there are high responses for the CCV that could mean that re-concentration took place and fresh aliquot should be analyzed).

- If the CCV is a closing CCV, the data for the samples run before this CCV may be evaluated for hits. If there are no hits for the compounds that did not meet %D criteria, those samples do not need to be reanalyzed provided that the response of the analyte had increased and would have been detected in the sample if it were present.
- Both columns should meet QC criteria, however, they may be evaluated with the above corrective actions in mind.
- The choice of corrective action must be made in consultation with the Department Manager, QA Manager, Project Manager, and/or the client. The reasoning for choosing one of the above options must be documented in the project narrative to the client.

10.5 Preventive Maintenance

10.5.1 Preventive maintenance may or may not include the following; replacing system uniliner, o-ring, merlin septum, MXTY connector or clipping a length of guard column.

10.5.2 Additionally, preventive or routine maintenance for GC/ECD systems may involve baking out the ECD detectors, replacing guard, RTX-5 or CLPII columns.

11. Data Evaluation, Calculations and Reporting

11.1 Qualitative identification of multicomponent analytes (Aroclors) requires pattern matching between the calibration standards and the response observed in the sample on both columns. Retention time windows should be used as a gauge; however, pattern recognition for the multicomponent analytes is most important. Qualitative identification for Congeners is made when a peak in a sample is observed within the retention time window for a calibrated analyte on both columns. In other words, an analyte is not considered present in a sample unless the analyte is detected on both columns within the retention time windows for that analyte established during calibration.

11.2 For samples with PCB Aroclors positively identified on both columns, compare the responses of the 3 to 5 major peaks in the single point calibration standard for that Aroclor with the responses of the peaks observed in the sample extract. For Aroclor 1660 use calibration curve to determine concentration of the detected Aroclor. The relative peaks and number of peaks in the sample should be similar to that observed in the standard; however, degradation, weathering and interferences may cause the sample pattern to differ from that observed from the standard. The peaks chosen for quantitation must be free from interferences. Calculate the concentration of each corresponding peak in the sample chromatogram and the 3 to 5 resulting concentrations are averaged to provide the final result for the sample.

11.3 If Total PCB is requested the following rule will apply:

- The Reporting Limit for Total PCB will be set to match the lowest Reporting Limit for individual Aroclor
- All Aroclors detected above RL will be sum together to provide Total PCB

- Total PCB will be calculated based on the amount of Aroclors requested by the client
- Total PCB can only be reported if individual Aroclors are reported as well
- If individual Aroclor is detected above working range of the calibration curve the E qualified results will not be included in the summation for Total PCB

11.4 Calculate the relative percent difference (RPD) between compound concentrations on both columns. If the calculated RPD is >40%, check the chromatograms to see if an overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine baseline parameters established by the data system (or operator) during peak integration.

- *If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result. This approach is conservative relative to protection of the environment. The disparity between the two columns should be noted by a "P" qualifier.
- *If the high RPD is clearly the result of an interference on one of the columns, the lower result is reported with an "I" qualifier on the report.
- If one of the two results for a target appears to have a biased response due to interference the analyst can force the quantitation from the column providing the more accurate value. Any target hit forced off of the compliant column due to interference on the alternate column will be flagged with an "I" on the report only if the RPD between the two columns is greater than 40%.
- In the instance that a Congener co-elutes with another Congener or other target compound on one column, the compound will be "C" flagged and reported off the unbiased conformant column (i.e.: on the RTX-5 column, BZ#184 and BZ#153 co-elute). Studies of independent targets have been conducted for reference of co-eluting pesticides and congeners.

11.5 Calculate the recovery for the surrogates TMX and DCB for Aroclors, and DBOB and BZ 198 for Congeners, in each field, blank, and quality control sample extract on both columns. Calculate the recovery of the matrix spike and LCS compounds in the appropriate samples on both columns.

11.6 Evaluate whether or not a dilution of an extract is needed by ensuring that all target PCBs areas measured fall within the calibration range of the instrument. If the range is exceeded, a dilution analysis is required. Dilute the extract to bring the out-of-range component(s) to within 50-100% of the full calibration range. Also evaluate the sample chromatogram for possible saturation of the detector.

11.7 If a dilution analysis is made, compare this run to the initial analysis to ensure that the chromatography is similar, that compound detection is similar, and that the reported values for detected compounds make sense (i.e., that the final value for any compound detected in both analyses is about the same value). If the dilution run is not comparable to the initial run, corrective action should take place.

11.7.1 *Corrective Action:* Re-evaluate both analyses (un-diluted and diluted) ensuring that integration of all peaks was performed properly and that the correct calibration curves were used for quantitation. Determine whether or not the results are off by a systematic bias which might be indicative of a poor injection or of an inaccurate dilution. If the initial run's injection is determined to have been poor and if this analysis does not need to be reported to the client (based no project specific requirements) then the results of the dilution analysis only should be reported to the client (unless there are special project requirements to

report data at specific reporting limits). If an inaccurate dilution is suspected, a new dilution of the extract should be made and this second dilution analyzed. The results from all three runs should be compared to verify the corrective action and to determine which result should be reported to the client.

11.7.2 Reporting of the results for a sample where two different dilution analyses are conducted, two data sheets should be prepared for reporting the sample with all components reported from the initial run with the "E" flag for the over-range component, which should be reported from the dilution. The project narrative must accurately explain how the data are reported whether one or two data sheets are used to report one sample.

11.8 Calculations

11.8.1 To calculate the ***Relative Standard Deviation*** (RSD) of all target compounds for the initial calibration, use the formula below. See Section 10.2.4 for initial calibration acceptance criteria. Additionally, use the initial multi-point calibration to determine ***Relative Response Factors*** (RRF_Is) – *Internal Standard Calibration* or for or ***Calibration Factor (CF_I)*** – for *ExternalStandard Calibration Model* at each concentration level, for each analyte. Average the RRF_Is from the initial multi-point calibration, to generate mean RRF_Is, for quantification of each analyte. Follow the same calculations for each surrogate compound.

The RRF_Is are based on the internal standard compounds and are calculated using the formula below.

$$\mathbf{RSD = SD / mean\ RRF_I \times 100}$$

where:

SD = Standard deviation between the five points, for that target analyte.

$$\mathbf{RRF_I = (A_c \times C_{IS}) / (A_{IS} \times C_c)}$$
$$\mathbf{CF_I = A_a / C_c}$$

where:

A_c = Area of the standard compound to be measured.
A_{IS} = Area of the representative internal standard compound.
C_{IS} = Concentration of the representative internal standard compound (ug/L).
C_c = Concentration of the standard compound to be measured (ug/L).

11.8.2 Based on the mean RRF_Is, calculate the raw concentration for each target analyte and surrogate in the sample extracts using the following formula:

$$\mathbf{C_a = (A_a \times C_{IS}) / (A_{IS} \times RRF_I)}$$

where:

C_a = Calculated concentration of the target analyte, from quantitation report (ug/L).
A_a = Area of the target analyte.
A_{IS} = Area of the representative internal standard compound.
C_{IS} = Concentration of the internal standard compound added to each extract (ug/L).

11.8.3 Calculate the ***Sample Concentration*** (C) for each target compound by the following formula:

$$C = (C_a / V_s) \times FV \times DF$$

where:

C = Concentration in the sample (ug/L liquid, ug/Kg solid, ng/PUF).

V_s = Original volume or weight of sample extracted, corrected for % solids, if applicable. (To correct for % solids, multiply the sample weight by the % solid as expressed as a decimal. For example: 15.24g x 0.843 = 12.85, for a sample size of 15.24g at 84.3% solid).

DF = Dilution factor or fraction of the original extract to which internal standard is added.

FV = Sample Final Volume.

If the response of any analyte in a sample exceeds the linear response range, as defined by the initial calibration standards in Section 13.0, dilute the extract so that the concentrations of all target compounds fall within the range of the calibration curve.

11.8.4 Calculate the ***Surrogate Recoveries*** relative to the internal standards by the following formula:

$$\%R = \frac{C_S \times DF}{C_{Strue}} \times 100$$

where:

C_{Strue} = Actual concentration of the surrogate in the extract (ug/L).

C_S = Calculated concentration of the surrogate, from quantitation report (ug/L).

DF = Dilution factor or fraction of the original extract.

11.8.5 Compare results for each analyte in the ***Continuing Calibration Verification*** (CCV), to the true value by determining the percent difference.

$$\textbf{Percent Difference (\%D)} = ([C_{Ctrue} - C_C] / C_{true}) \times 100$$

where:

C_{Ctrue} = Actual concentration of the compound in the CCV (ug/L).
C_C = Calculated concentration of the compound in the CCV, from quantitation report.

11.9 All solids including soils, sediments, and sludges must be reported on a dry-weight basis. Tissue results may be reported in wet-weight, or "as received," depending upon client request. Air PUF samples are reported on an "as received" basis.

11.10 The primary analyst does data entry, or upload of the data, into the LIMS system. The LIMS is "linked" to the instrument, so the analyst must choose the sample(s) to be reported from that instrument's analytical sequence. All associated preparation and instrumental QC samples and dilutions are also chosen. Once the data/samples have been selected and "associated" with the proper QC samples, the batched data set is sent to the LIMS.

11.11 Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP, SOP/1729. All records shall be stored in such a manner as to be safe and accessible for at least 10 years.

11.12 Notebooks: Laboratory notebooks are designed to accommodate the specific analysis. Instrument printouts are used to document run sequences, and each daily sequence printout is filed in a three-ring notebook. If a sample requires re-analysis or re-extraction for any reason, a notation is made next to the sample entry on the sequence log. Requests for re-extraction are further documented in the "Request for Re-extraction" logbook. After one year of sample analysis, the sequence run log is permanently bound, assigned an internal ID number, and filed accordingly. Such files shall be archived so as to remain available for at least 10 years. All laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* Work Instructions,WI-108-01, and all record keeping and document control practices.

11.13 Electronic records: All data files from computers, attached to instruments, shall be backed up daily onto the proper directory on the server. The backups shall be stored so as to be accessible for 10 years. Movement of the data files to the server is the responsibility of the primary analyst. Server backup and storage is the responsibility of the IT department.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All PCB Aroclor and Congener results are reportable without qualification if analytical holding times are met, preservation requirements (including cooler temperatures) are met, and all QC criteria defined in the table below are met. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis.

QC Parameter	Acceptance Criteria

Initial Calibration Curve	20% RSD for all target analytes Correlation coefficient must be ≥ 0.99 – for linear model The COD or r^2 must be ≥ 0.99 – for non-linear model
Independent Check Verification	+/- 20% recovery of the true values, For Aroclors the Average of the 5 representative peaks are calculated to get the %D.
Continuing Calibration Verification	Analyzed every 12hr or at the minimum of every 20 samples, 20% D for all target analytes. For Aroclors the Average of the 5 representative peaks are calculated to get the %D.
Method Blank	No analyte at or above the reporting limit, "B" qualify analyte if detected < 10x the blank contamination
Laboratory Control Sample	Soil/Tissue/Aqueous/PUF: 40-140%; 50% RPD
Matrix Spike / Matrix Spike Duplicate	Same as for LCS; 50% RPD between the duplicates.
Sample / Sample Duplicate	50% RPD between the duplicates.
Surrogates	Soil: 30-150%; Aqueous: 30-150%; PUF: 60-120%
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
SRM	Same as for LCS, 40% - 140% recovery

Any deviations and observations made about the analyses must be documented in the instrument logbook and or project narrative. If a problem arises during analysis, document the problem and initiate corrective action. If there is a problem with a sample analysis which indicates that re-extraction should be performed, and if there is no additional sample available for re-extraction, the Project Manager needs to be informed immediately so that the client can be involved in the corrective action process. Re-extraction of a sample should be done within holding time if at all possible.

Evaluation of a sample result often requires knowledge about the results of all samples within a job. Therefore, use all of the data, if possible, from a job to judge whether or not corrective actions are needed. For example, if a sample is run and the surrogates are low, one might request a re-extraction immediately only to find that a MS/MSD were also done on this sample, with low surrogate and MS recoveries, confirming a matrix effect. In this case, a re-extraction is not required and the results would be reported with a narration explaining the evidence of a matrix effect on the low surrogate recoveries. The characteristics of the overall job may be invaluable in deciding how to report the data to the client.

If non-compliant PCB Aroclor or PCB Congener results are to be reported, the Department Manager and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Once satisfactory analytical results have been generated, the extracts are held for 30 days, or longer if specified by a client contract, then discarded into a 55-gallon drum labeled “Vial Waste”.

Refer to Alpha’s Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

1797 Waste Management and Disposal SOP

1816 Reagent, Solvent, and Standard Control SOP

SOP 1729 Document Control

SOP 1731 Manual Integration

16. Attachments

Table I: Reporting Limits (RLs) and Method Detection Limits (MDLs) for PCB Aroclors and Congeners by GC/ECD

Table II: LCS/LCSD, MS/MSD and Precision and Accuracy Evaluation Criteria

Table III: Surrogate Recovery Evaluation Criteria

Table I

Reporting Limits (RLs) and Method Detection Limits (MDLs) for PCB Aroclors and Congeners by GC/ECD

Compound	CAS Registry No	Aqueous in µg/L		Solid in µg/Kg		PUF in ng/PUF	
		MDLs	RLs	MDL	RLs	MDL	RLs
Aroclor 1016	12674-11-2	0.0050	0.03	8.01	30	8.01	30
Aroclor 1221	11104-28-2	0.0103	0.03	27.6	100	27.6	100
Aroclor 1232	11141-16-5	0.0092	0.03	9.98	30	9.98	30
Aroclor 1242	53469-21-9	0.0055	0.03	5.63	20	5.63	20
Aroclor 1248	12672-29-6	0.0051	0.03	5.62	20	5.62	20
Aroclor 1254	11097-69-1	0.0105	0.03	6.72	20	6.72	20
Aroclor 1260	11096-82-5	0.0045	0.03	6.22	20	6.22	20
Aroclor 1262	11100-14-4	0.0092	0.03	5.51	20	5.51	20
Aroclor 1268	37324-23-5	0.0032	0.03	4.89	20	4.89	20

IUPAC Name	BZ #	CAS Registry No.	Aqueous RL in µg/L	Solid RL in µg/Kg
2,4'-Dichlorobiphenyl	BZ 8	34883-43-7	0.003	2.0
2,2',5-Trichlorobiphenyl	BZ 18	37680-65-2	0.001	1.0
2,4,4'-Trichlorobiphenyl	BZ 28	7012-37-5	0.001	1.0
2,2',3,5'-Tetrachlorobiphenyl	BZ 44	41464-39-5	0.001	1.0
2,2',5,5'-Tetrachlorobiphenyl	BZ 52	35693-99-3	0.001	1.0
2,3',4,4'-Tetrachlorobiphenyl	BZ 66	32598-10-0	0.001	1.0
3,3'4,4'-Tetrachlorobiphenyl	BZ 77	32598-13-3	0.001	1.0
2,2',4,4',5-Pentachlorobiphenyl	BZ 99	38380-01-7	0.001	1.0
2,2',4,5,5'-pentachlorobiphenyl	BZ 101	37680-73-2	0.001	1.0
2,3,3',4,4'-pentachlorobiphenyl	BZ 105	32598-14-4	0.001	1.0
2,3',4,4'5-Pentachlorobiphenyl	BZ 118	31508-00-6	0.001	1.0
3,3',4,4',5-Pentachlorobiphenyl	BZ 126	57465-28-8	0.001	1.0
2,2',3,3',4,4'-Hexachlorobiphenyl	BZ 128	38380-07-3	0.001	1.0
2,2',3,4,4',5'-Hexachlorbiphenyl	BZ 138	35065-28-2	0.001	1.0
2,2',4,4',5,5'-Hexachlorobiphenyl	BZ 153	35065-27-1	0.001	1.0
3,3',4,4',5,5'-hexachlorobiphenyl	BZ 169	32774-16-6	0.001	1.0
2,2',3,3',4,4',5-Heptachlorobiphenyl	BZ 170	35065-30-6	0.001	1.0
2,2',3,4,4',5,5'-Heptachlorobiphenyl	BZ 180	35065-29-3	0.001	1.0
2,2',3,4',5,5'6-Heptachlorobiphenyl	BZ 187	52663-68-0	0.001	1.0
2,2',3,3',4,4',5,6-Octachlorobiphenyl	BZ 195	52663-78-2	0.001	1.0
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	BZ 206	40186-72-9	0.001	1.0
2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl	BZ 209	2051-24-3	0.001	1.0

Note: The reporting limits used for "Solid" refer to the matrices of soil, sediment, tissue, and any other possible non-aqueous matrix. The table above represents an example of reporting limits generally used in this analysis. The RLs for this analysis can vary based on sample matrix, amount extracted, final volume and cleanup schemes. Adjustments to these limits can be made to satisfy specific workplan or data quality objective requirements and specialty matrices.

Table II

LCS/LCSD, MS/MSD and Precision and Accuracy Evaluation Criteria

Spiked Component	Aqueous/Solid/Tissue/PUF (% recovery)	RPD
AR1016	40-140	50
AR1260	40-140	50
Congener	40-140	50

Table III

Surrogate Recovery Evaluation Criteria

Surrogate	Aqueous/Solid/Tissue (% recovery)	PUF (% recovery)
2,4,5,6-tetrachloro-m-ylene(TMX)	30-150	60-120
Decachlorobiphenyl (DCB)	30-150	60-120
DBOB	30-150	NA
BZ 198	30-150	NA

NOTE: Depending on project specific requirements, MS/LCS/Surrogate recoveries may be validated against a specified project QAPP, 8082 SW-846, TO-10A, CLP OLM04.2, DoD QSM 5.3, or in-house determined limits.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

THE ANALYSIS OF POLYCHLORINATED DIBENZO-p-DIOXINS AND POLYCHLORINATED DIBENZOFURANS (PCDDs/PCDFs)

BY

HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

CF-OA-E-002

APPLICABLE TO METHODS:
EPA SW-846 Method 8290A, EPA Method 1613B, EPA SW-846 Method 0023A, EPA Method TO-9a

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W. M. Larkins

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2.0 METHOD OBJECTIVE, PURPOSE, CODE, AND SUMMARY

This standard operating procedure (SOP) covers the analytical determination of PCDD/PCDFs according to the following methods:

2.1 SW-846 Method 8290A

2.2 EPA Method 1613B

2.3 SW-846 Method 0023A

2.4 EPA Method TO-9a (Jan 99)

3.0 APPLICABLE MATRICES

Applicable matrices for methods 8290A and 1613B include groundwater, wastewater, surface water, leachate, soil, sediment, sludge, oil, and tissue. The applicable matrix for method 0023A is an air sampling train, which may contain XAD resin (a hydrophobic crosslinked polystyrene copolymer resin, supplied as 20-60 mesh size white insoluble beads), filters, impinger water and solvent rinses. TO-9a is an ambient air sampling train which may contain polyurethane foam (PUF, a polyurethane foam, supplied as 1-3 inch cylinders approximately 3 inches long), XAD resin, filters, and solvent rinses.

4.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

4.1 Methods 8290A, 1613B and 0023A may be used to quantify PCDD/PCDFs that are soluble in methylene chloride and/or toluene. The compounds are separated using a gas chromatograph (GC) and detected using a high-resolution double focusing mass spectrometer (HRMS). Appendix 1 lists the analytes currently analyzed using these methods and their practical quantitation limits.

4.2 The practical quantitation limit (PQL) is the lowest level in the calibration curve. The PQL is the lowest level at which compounds may be accurately quantitated and is compound dependent. The calibration curve typically ranges from 1.0 ng/mL to 1000 ng/mL for methods 8290A, 0023A, and TO-9a, and from 0.5 ng/mL to 2000 ng/mL for method 1613B. These ranges reflect instrument readings, which are in ng/mL (ppb). It should be noted that the calibration range may vary between calibrations and instruments.

4.3 Method detection limit studies (MDLs) are performed and/or verified on an annual basis. MDLs are done for aqueous, solid, tissue and XAD matrices. For more information regarding MDLs, refer to The Determination of Method Detection Limits, CF-LB-E-001.

4.4 Qualified analysts must demonstrate proficiency initially and annually thereafter with an IDOC, CDOC, or PT study. Acceptability criteria may be found in the applicable analytical method.

4.4.1 To establish the ability to generate acceptable accuracy and precision, the analyst should perform an "analyst validation study" or Initial Demonstration of Capability. Four LCS standards are extracted and analyzed. Calculate the average recovery and the standard deviation of the recovery for each analyte of interest using the four results. Then compare the average and the standard

deviation with the corresponding criteria found in Table 6 of method 1613B, or with the determined limits for methods 8290A and 0023A. If the average and the standard deviation for all analytes of interest meet the acceptance criteria, then the analyst may begin work on actual samples. If the validation study fails for one or more of the compounds, then the study must be repeated for those compounds which failed.

5.0 METHOD VARIATIONS

5.1 Cape Fear Analytical analyzes a calibration point at 0.25 ng/mL, which is below the method required low point.

5.2 Standards and sample extracts are stored at room temperature to avoid analyte loss. Many of the target analytes in these methods form a strong cohesive bond with solids such as glass in cold temperatures; this type of analyte loss is not addressed in the method. (This is a variance from the following method recommendations: ≤6° per method 8290A; < -10°C per 1613B; -10° to -20° C per DoD QSM.)

5.3 Cape Fear Analytical utilizes the DB-5ms GC column (and may use the ultra inert version), which is capable of better resolution of the TCDF isomers. This column exhibits a different elution pattern than the DB-5 column referenced in the analytical methods. Relative retention time limits have been determined for this column for use with method 1613B, and are listed in Table 9.

5.4 Method 1613B does not address the reporting of EDL and EMPC. These values are reported for this method only when requested by the client.

6.0 DEFINITIONS

6.1 Accuracy: The degree of agreement between an observed value and an accepted reference value.

6.2 AlphaLIMS: The Laboratory Information Management System used at CFA, LLC.

6.3 Blank: An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and standard additions that are used with other samples. The LMB (Lab Method Blank) is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Contamination may be derived during sampling, transportation, storage or analysis. The blank may be used to establish a background value.

6.4 Calibration Standard (CAL): An aliquot of a primary standard solution or stock standard solution. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.5 Calibration Verification Standard (CVS, CCAL, CS3WT): A solution of target analytes with a concentration near the mid-point of the calibration range. It should be obtained from a second source vendor and is used to verify the initial calibration on a basis described in the determinative method. This solution may also contain the window defining analytes and the column performance mix.

6.6 Cleanup Standards: Isotopes added prior to cleanup that are used to measure the efficiency of the fractionation step alone. Method 1613B uses one compound (37Cl4-2378-TCDD) as the Cleanup Standard. Method 8290A does not address the use of cleanup standards.

6.7 Duplicate Analysis: The analysis or measurement of the variable of interest performed identically on two field subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision of sample, preservation, or storage internal to the laboratory.

6.8 Estimated Detection Limit (EDL): A calculation of the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. The EDL is calculated for each 2378-substituted congener that is not identified.

6.9 Estimated Maximum Possible Concentration (EMPC): A calculation for a peak characterized by a response with a signal-to-noise ratio of at least 2.5 for both the quantitation ions, and meeting all identification criteria except ion ratio. EMPC is a worst-case estimate of the concentration.

6.10 Extraction Standards: Isotopes added prior to extraction that serve as internal standards for many 2,3,7,8 substituted congeners. In addition, to measure the overall extraction and fractionation efficiencies. Method 8290A names them Internal Standards while Method 1613B uses the Labeled Compounds terminology.

6.11 Injection Standards: Isotopes added prior to injection to determine the recoveries of the Extraction and Cleanup Standards. Method 8290A names them Recovery Standards while Method 1613B calls them Internal Standards.

6.12 Internal Standard (ISTD): A known amount of standard added to a test portion of a sample as a reference for evaluating the retention time and concentration of dependent analytes and controlling the precision and bias of the applied analytical method.

6.13 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

6.14 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

6.15 Matrix Spike and Matrix Spike Duplicate (MS and MSD): Two separate aliquots of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS and MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results. The concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD adjusted. Percent recovery is calculated for both aliquots, and RPD is calculated between the two.

6.16 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

6.17 Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves, a data quality

indicator. Precision is usually expressed as standard deviation, variance or range in either absolute or relative terms.

6.18 Quantitation Limits (also PQL, RL): The value at which an instrument can accurately measure an analyte at a specific concentration (i.e., a specific numeric concentration can be quantified). These points are established by the upper and lower limits of the linear calibration range.

6.19 Sampling Standards: Isotopes added prior to field sampling for Method 0023A and Method TO-9a that are used to measure the efficiency of the sampling step alone.

7.0 INTERFERENCES/LIMITATIONS

7.1 Contaminants found in extraction glassware, solvents, and other sample processing hardware may jeopardize the integrity of this method.

7.2 Glassware must be scrupulously cleaned as soon as possible after extraction.

7.3 Contamination may also occur in the GC/MS system. High boiling materials tend to build up in the injection port and the front end of the column. The analyst should maintain a thorough working knowledge of keeping the injection port free of contamination, including changing out the septum, injection port liner, O-ring, ferrule, and gold seal.

7.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. If carryover is suspected, potentially impacted samples must be re-analyzed after any needed maintenance, solvent replacement, and/or cleaning has been done.

7.5 Upon review of a completed sequence, if one is required to perform a 200x or greater dilution because of a sample's target concentrations, that the rinse vials on the instrument that determined this dilution need must have it's solvent replaced. This action should be documented in the maintenance log.

8.0 SAFETY PRECAUTIONS AND WARNINGS

METHYLENE CHLORIDE IS A SUSPECTED CARCINOGEN AND A KNOWN SKIN IRRITANT.

NO OCCUPATIONAL EXPOSURE LIMIT FOR DIOXIN HAS BEEN ESTABLISHED. IT IS A KNOWN AND PROBABLE HUMAN CARCINOGEN.

CONTACT WITH OXIDIZERS MAY GENERATE EXPLOSIVE MIXTURES.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN USING METHYLENE CHLORIDE.

8.1 Eye protection should be worn when handling samples, reagents, or standards. NOTE: Contact lenses pose a special problem; soft lenses may absorb irritants and all lenses concentrate them. DO NOT wear contact lenses in the laboratory.

8.2 Treat all chemicals and samples as potential health hazards and reduce exposure to these chemicals to the lowest level possible. CFA maintains a current reference file of Material Safety Data Sheets (MSDS). These documents and individual sample MSDS provided by clients are maintained in the laboratory.

8.3 Personal Protective Equipment (PPE)

8.3.1 Gloves and eye protection should be worn when handling reagents, solvents, standards and samples.

8.3.2 Analysts should prepare samples and standards under the hood.

8.4 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.5 Never leave gas cylinders unchained or untied.

8.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, management should be notified of all accidents.

8.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers and fire blankets are located in the lab. Training is available on the proper operation of this equipment.

8.8 The analyst must use care when assembling and operating instrumentation. Check to see that the gas chromatograph equipment is properly assembled and hooked up to the proper gas cylinder and power, referencing the appropriate manual. Analytical equipment must only be operated by qualified personnel.

8.9 For further safety instructions, consult the Safety Manual, CF-LB-N-001.

9.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION

9.1 Equipment associated with this method includes:

9.1.1 Gas tight syringes

9.1.2 2 mL high recovery (conical) autosampler vials and storage racks

9.1.3 Teflon crimp tops

9.1.4 Crimper/De-crimper

9.1.5 GC Columns

9.1.5.1 Agilent DB5-MS or equivalent (i.e. ui); 60 m, 0.25 mm, 0.25 um

9.1.5.2 Agilent DB-225 or equivalent; 30 m, 0.25 mm, 0.25 um

9.1.6 Quartz/Glass injection port liners

9.1.7 Injection port liner O-ring seals

9.1.8 Gold seals

9.1.9 Ferrules

9.1.10 Column cleaving tool

9.1.11 Septa (thermogreen)

9.1.12 10-100 uL adjustable air displacement pipette with disposable tips

9.2 Instrumentation

9.2.1 Waters Autospec Premier high resolution mass spectrometer

9.2.2 Agilent 7890 Gas Chromatograph

9.2.2.1 A suggested temperature program for primary analysis follows:

Initial Temp.	140° C
Hold Time	1.0 min.
Rate 1	20° C/min.
Temperature 2	180° C
Time 2	2°/min
Temperature 3	235° C

Rate 3	30° C/min.
Final Temp.	290° C
Hold Time	13 min.
Run Time:	45 minutes (may vary due to column length or flow rate)
Solvent Delay:	18.0 min.
Splitless Valve Time:	1.5 min.
Flow:	1.8 mL/min.
Mass Range:	See descriptor definitions (Table 2)

NOTE: These instrument conditions and rates are guidelines which may change.

9.2.3 LEAP Technologies GC PAL Autosampler

9.2.3.1 Suggested parameters:

Sample volume – 1 µL

Air volume – 0.5 µL

Solvent push volume – 1 µL

Number of sample washes - 0

Solvent washes - 30

Sample viscosity wait – 1 second

Number of sample pumps - 0

Injection mode - Fast

10.0 REAGENTS AND STANDARDS

10.1 Reagents and standards

10.1.1 Nonane

10.1.2 Source Standards: Source Standards are purchased directly from vendors and may be diluted to make stock, intermediate, or working standards. These may include extraction standard, matrix spiking standard, cleanup standard, injection standard, as well as others. Source standards expire per the vendor expiration date or after five years from the date opened, whichever is shorter. Please reference CF-LB-E-007 and CF-OA-E-002 for further information regarding standards and their preparation.

10.1.3 Initial Calibration (ICAL) Standards: Certified calibration standards are purchased from commercial vendors at a minimum of five concentration levels. One of the calibration standards is at a concentration near, but above, the method detection limit; the others should correspond to the expected range of compounds found in samples. Calibration standards expire after a maximum of five years and should be monitored frequently for signs of degradation.

10.1.4 Calibration Verification Standards (CVS, CCAL, CS3WT): A certified CVS is purchased from a second source commercial vendor at a concentration that is near to the midpoint of the calibration curve.

10.1.5 Window Defining Mix and Column Performance Mix (WDM and CPM): A standard containing the first and last eluters for each homolog group, as well as the dioxin and furan isomers used to demonstrate isomer specificity on the GC column in use. These may be contained in the same standard as the calibration verification (known as CS3WT).

11.0 SAMPLE HANDLING AND PRESERVATION

11.1 Sample extracts have a 45-day holding time from the date of extraction by methods 8290A and 0023A, and a 365 day holding time from the date of extraction by 1613B. Note that per method 8290A, tissue extracts must be completely analyzed by 45 days from collection. TO-9a cartridges are considered clean for 30 days from preparation, samples must be extracted 7 days from collection and analyzed 40 days from extraction. See Table 13.

11.2 Sample extracts are delivered from the prep lab to the instrument lab and are stored in a darkened hood at room temperature. The extracts are usually grouped according to preparation batches and are accompanied by the batch pull sheet and other pertinent paperwork.

11.3 Custody of samples is monitored using the AlphaLIMS sample tracking system. Each analyst should scan the samples planned to run into their custody prior to analysis.

11.4 All sample extracts should be treated with caution as potential health hazards. Refer to Section 8.0 on safety.

12.0 SAMPLE PREPARATION

12.1 Before extracts can be analyzed on the instrument, they must first be evaporated to dryness under nitrogen and then spiked with injection standard to set the final volume nominally at 20 µL. A determination must also be made as to whether the extract should be diluted. The decision to dilute a sample extract is based on a number of factors: sample screening, historical data about the sample or sample site, the appearance of the extract (color, viscosity, incidental odor, turbidity, etc.), or regulatory considerations. The experience of the analyst is invaluable in making this determination.

NOTE: Sample extracts may contain multiple layers or sediment. Samples that contain sediment are returned to cleanup. Multiple layers are treated on a case-by-case basis. If the extract can be homogenized, then a uniform sample is achieved. If the extract remains bi-phasic, the PM and client are contacted for further guidance.

12.2 If a sample is to be analyzed without dilution ('neat'), 2 nanograms of injection standard solution is added to the extract using a pipette (20 µL of a 0.1 ng/µL = 100 pg/µL extract concentration). A cap is then placed on the vial and secured by crimping before vortexing the sample to ensure complete mixing and vial wall washing.

12.3 If samples require dilution, the dilution is made using nonane or appropriate solvent. If not previously added, 2 nanograms of JS is added to the autosampler vial. Dilution prep may involve the addition of supplemental extraction standard (ES) and is documented in the injection prep logbook.

12.4 Once samples are prepped, they are ready to be injected onto the instrument. An autosampler is used to inject standards and sample extracts on the instrument.

12.5 The need for dilution may also be determined after analysis is performed, and may still be performed as above. Under normal circumstances, a sample would be diluted if any chromatographic peaks saturate the detector.

13.0 QUALITY CONTROL REQUIREMENTS

Typically a blank (LMB), laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) are extracted and analyzed with each prep batch. Other client requirements may include a matrix spike (MS) and matrix spike duplicate (MSD) or sample duplicate (DUP).

13.1 Blanks

13.1.1 A blank is extracted with each batch of 20 or fewer samples to demonstrate that interferences from glassware, reagents and the analytical system are under control. Blanks are carried through all stages of sample preparation and analysis. For Method 1613B, an acceptable blank must be below the minimum levels listed in Table 2 of the method for all analytes. For Methods 8290A, 0023A, and TO-9a, all analytes must be below the Lower Method Calibration Limits.

13.1.2 The percent recovery of each labeled standard (extraction and cleanup) is calculated as shown in Sec. 17.4.5. Recoveries must be within the limits in Table 7 for method 1613B. For methods 8290A and 0023A, extraction standard recoveries must be within 40-135%. Sampling standards for Method 0023A must be within 70-130%. For method TO-9a, extraction standards must be within 50-120% for tetra- through hexa- and within 40-120% for hepta- and OCDD. Sampling standards for Method TO-9a must be within 50-120%.

13.2 Laboratory Control Samples and Matrix Spikes

13.2.1 The spiking standard for LCS/LCSDs and MS/MSDs contains all analytes listed in Table 5. For each LCS, LCSD, MS and MSD, the concentration of each analyte and its percent recovery are calculated as shown in Sec.17.4.1 and 17.4.5. For methods 8290A and 0023A, percent recoveries should be within 70-130%. For method 1613B, recovered concentrations should be within the limits in Table 6.

13.2.2 If recovery is not within these limits, the data may need to be re-checked for errors, or the samples and QC may need to be re-analyzed. In addition, the instrumentation may need to be checked for performance problems. If the LCS fails to meet acceptance criteria due to low recovery, the associated samples may have to be re-extracted and re-analyzed when possible. If one or more recoveries are high in the LCS and these analytes are not detected in the samples, the event should be documented and data may be reported. If the MS and MSD both fail due to matrix interference and/or dilution, data may be reported provided the associated LCS passes acceptance criteria.

NOTE: Many clients have contract specific criteria that must be considered when evaluating recovery of the Quality Control samples.

13.2.3 The percent recovery of each labeled standard (extraction and cleanup) is calculated as shown in Sec. 17.4.5. Recoveries must be within the limits in Table 6 for method 1613B. For methods 8290A and 0023A, extraction standard recoveries must be within 40-135%. Sampling standards for Method 0023A must be within 70-130%. For method TO-9a, extraction standards must be within 50-120% for tetra- through hexa- and within 40-120% for

hepta- and OCDD. Sampling standards for Method TO-9a must be within 50-120%.

13.3 Samples

13.3.1 The percent recovery of each labeled standard (as listed in SOP CF-OA-E-001) is calculated as shown in Sec. 17.4.5. Recoveries must be within the limits in Table 7 for method 1613B or 40-135% for method 8290A. For method TO-9a, extraction standards must be within 50-120% for tetra- through hexa- and within 40-120% for hepta- and OCDD. Sampling standards for Method TO-9a must be within 50-120%.

13.3.2 Calculated EDLs should be below the PQLs in Table 1. Any reported EDLs above the PQLs should be noted in the case narrative.

14.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE

14.1 Mass spectrometer performance

14.1.1 The mass spectrometer is operated in electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Static resolving power checks must be performed at the beginning and at the end of each 12-hr period of operation. Corrective action must be implemented whenever the resolving power does not meet the requirement.

14.1.1.1 Chromatography time for PCDDs and PCDFs exceeds the long term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass drift correction is mandatory. A lock-mass ion from the reference compound PFK is used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Lock mass ions may be found in the descriptor table, Table 2. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full scale deflection for a given set of detector parameters. Under these conditions, sensitivity changes that might occur during the analysis can be more effectively monitored. NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in an increase in downtime for source cleaning.

14.1.2 Documentation of the instrument resolving power must be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760) obtained during the above peak matching experiment by using the low mass PFK ion at m/z 304.9824 as a reference. The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 2) must allow manual determination of the resolution, i.e., the

horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10 percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

14.2 System Performance

System performance criteria are presented below. The laboratory may use the recommended GC column described in Sec. 9.1. The laboratory must document that all applicable system performance criteria are met before sample analysis begins. Sec. 9.2.2 provides recommended GC conditions that may be used to satisfy the required criteria. Mass spectrometer resolving power checks must be performed at the beginning and the end of each 12-hr period of operation. A GC column performance check is required at the beginning of each 12-hr period during which samples are analyzed. For Method 1613B, a continuing calibration must be performed at the beginning of the sequence, while for Methods 0023A and 8290A, continuing calibrations must be performed at both the beginning and the end of a sequence. An ending continuing calibration may also serve as the beginning check for the next sequence.

14.2.1 GC Column performance check

14.2.1.1 Inject 1 µL of an aliquot of the column performance check solution (Sec. 10.1.5) and acquire selected ion monitoring (SIM) data within a total cycle time of $\leq$ 1 second. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of $\leq$ 25 percent (Figure 1), where:

Valley percent = (x/y) × 100

x = measured as in Figure 1 from the 2,3,7,8-closest TCDD eluting isomer

y = the peak height of 2,3,7,8-TCDD

For 2378-TCDF confirmatory analysis, the chromatographic separation between 2378-TCDF and its closest eluters must be resolved with a valley of $\leq$ 25 percent.

14.2.1.2 It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions described in this SOP. Their retention times are used to determine the five homologue retention time windows that are used for qualitative (Sec. 15.3.1.1) and quantitative purposes. All peaks (including 13C12-2,3,7,8-TCDD) should be labeled and identified on the chromatograms. All first eluters of a homologous series should be labeled with the letter "F," and all last eluters of a homologous series should be labeled with the letter "L". Any individual selected ion current profile (SICP) or the reconstructed homologue ion current constitutes an acceptable form of data presentation. A SICP for the labeled compounds is also required.

14.2.1.3 Particular caution should be exercised for the switching time between the last tetra-chlorinated congener (1,2,8,9-TCDF) and the first penta-chlorinated congener (1,3,4,6,8-PeCDF), as these two compounds elute within 15 sec of each other on the 60m DB-5 column, and overlap on the 60m DB-5ms column. Both congeners must be acquired within one analysis.

14.2.1.4 The absolute retention time of $^{13}C_{12}$-1,2,3,4-TCDD must exceed 25.0 minutes on the primary GC column in use, and 15.0 minutes on the confirmatory GC column.

14.3 Initial Calibration

14.3.1 Prior to running a multi-level calibration, take precautions to ensure that the instrument meets system performance criteria. The analyst must document that all system performance criteria are met before analyzing an initial calibration.

14.3.2 Initial calibration is required before any samples are analyzed for PCDDs and PCDFs and must meet the acceptance criteria listed below. Initial calibration is also required if any routine calibration does not meet the required criteria listed in Sec. 15.2, and at a minimum, annually.

14.3.3 At a minimum, all five high-resolution concentration calibration solutions listed in Table 5 must be used for the initial calibration.

14.3.4 Tune the instrument with PFK to meet the above-specified system performance criteria.

14.3.5 Inject the GC column performance check solution and acquire SIM mass spectral data. The total cycle time for each descriptor must be < 1 second. The laboratory must not perform any further analysis until it is demonstrated and documented that the criteria listed in Sec. 15.1.1.1 are met.

14.3.6 By using the same conditions (GC and MS) that produced acceptable results with the column performance check solution, analyze each of the five concentration calibration solutions. Each injection must meet the following ion ratio and signal-to-noise (S/N) requirements:

14.3.6.1 The ratio of the areas of the integrated ion current for the ions appearing in Table 2 (homologous series quantitation ions) must be within the indicated control limits (set for each homologous series) in Table 3. These ion ratio requirements must be within the specified control limits simultaneously in one run. It is the analyst's responsibility to take corrective action if the ion abundance ratios are outside the limits.

14.3.6.2 For each selected ion current profile (SICP) and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the S/N ratio must be better than or equal to 10. Manual measurement of S/N is required for any GC peak that has an apparent S/N of less than 15:1. The result of the calculation must appear on the SICP above the GC peak in question.

14.3.7 Calculate the 17 relative response factors (RF) for unlabeled target analytes relative to their appropriate internal standards (see Table 10). Also calculate

the RFs for the ESs and CSs relative to the appropriate injection standards according to the following formula:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

Ax = Sum of the Areas of the two characteristic ions for the compound being measured.

Ais = Sum of the Areas of the two characteristic ions for the specific internal standard.

Cis = Concentration of the specific internal standard.

Cx = Concentration of the compound being measured.

The RF is a dimensionless quantity; the units used to express Cis and Cx must be the same.

14.3.8 The RF for other isomers within a homolog group shall be determined from the average RF of the 2,3,7,8-substituted isomers. For example, the RF for non-2,3,7,8-substituted HxCDD isomers (totals peaks) is the average of the three 2,3,7,8-substituted isomers. NOTE: If only one 2,3,7,8-substituted isomer is present in the calibration then use that isomer's RF for all isomers within its homolog group.

14.3.9 Because more than five calibration levels may be analyzed, the analyst may choose to deactivate one or more levels globally. If a level is not used, it will be deactivated in the method for all analytes in that calibration mixture. In some cases the upper level(s) of the calibration may be deactivated in order to meet method criteria for single compounds. This practice results in a narrower calibration range. The low standard representing the PQL cannot dropped. Please note that this practice does not represent "cherry picking," which is acknowledged as an unacceptable laboratory practice.

14.3.10 The average RF must be calculated for each compound as follows:

$$RF_{avg} = \frac{\sum_{i=1}^{n} X}{n}$$

Where:

N = number of calibration levels

X_i; i=1 to n, are the compounds RF values for each calibration point

14.3.11 Criteria for acceptable initial calibration

The criteria listed below for acceptable calibration must be met before sample analyses are performed.

14.3.11.1 Per method 8290A, the percent relative standard deviations for the mean response factors from the 17 unlabeled standards must not exceed ± 20 percent, and those for the nine labeled reference compounds must not exceed ± 20 percent. These limits also apply to Method 0023A. Per method 1613B, the percent relative standard deviations for the mean response factors from the 17

unlabeled standards must not exceed ± 20 percent, and those for the fifteen labeled reference compounds must not exceed ± 35 percent. See Table 12 for method TO-9a minimum requirements.

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

Where:

RSD = relative standard deviation

$\bar{x}$ = mean of 5 or more initial RFs for a compound

SD = standard deviation of average RFs for a compound

$$SD = \sqrt[2]{\frac{\sum_{i=1}^{n}(X - A)}{n-1}}$$

where:

n = number of calibration levels

X_i; i=1 to n, are the compounds RF values for each calibration point

A = average of the RFs from above

15.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION

15.1 Resolution check

15.1.1 At the beginning and end of each 12-hour window, mass resolution must be tuned and/or verified. A static resolving power of at least 10,000 must be demonstrated at appropriate masses before analysis is performed.

15.1.2 Using a PFK molecular leak, tune the instrument to the minimum required resolving power of 10,000 at m/z 330.9792 (for day to day operations, the instrument may be tuned to approximately 11,000). Verify that the exact mass of m/z 380.9760 is within 5 ppm of the required value.

15.2 Column Performance/Window Defining/Continuing Calibration Check (CS3WT)

15.2.1 Inject 1 uL of the CS3WT or CPM. This standard is obtained from a different manufacturer or is a different lot from the same manufacturer than the initial calibration standard. Note that for NC drinking waters a different manufacture must be used. Verify that all column performance and window defining criteria in Section 14.2.1 have been met.

15.2.2 The CS3WT also contains the analytes for continuing calibration. The initial calibration curve for each compound of interest must be verified once every 12 hours.

Calculate the percent difference using:

$$\%\ \text{Difference} = \frac{\left|\overline{RF}_i - RF_c\right|}{\overline{RF}_i} \times 100$$

Where:

$\overline{RF}_i$ = average response factor from initial calibration

RF_C = response factor from current CS3WT

Calculate analyte concentrations using:

$$\left[PCDD/PCDF\right] = \frac{\left(A_{unk}^{ion1} + A_{unk}^{ion2}\right)}{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)} \times \frac{Q_{ES}}{\overline{RF}}$$

Where:

A_{unk} and A_{ES} = the integrated area for each ion monitored.
Q_{ES} = the amount of extraction standard in pg/uL
RF = Average RF from the ICAL for the compound

15.2.2.1 For methods 0023A and 8290A, if the percent difference for each native analyte in the CS3WT is ≤ 20%, and for each labeled analyte is ≤ 30%, the initial calibration is assumed to be valid. For method 1613B, analyte concentrations must fall within the limits in Table 8. If the criteria are not met, corrective action should be taken. If no source of the problem can be determined after corrective action has been taken, a new calibration may need to be generated. For Method TO-9a See Table 12 for minimum requirements.

15.2.2.2 All ion ratios must be within the limits in Table 3.

15.2.2.3 For methods 0023A and 8290A, if no more than two unrelated compounds in the continuing calibration check performed at the end of a 12-hour period fail by no more than ±25% for the 17 unlabeled compounds and ±35% for the 9 labeled compounds, the average RF values from the beginning and ending continuing calibration checks should be used to compute the analyte concentrations, instead of the RF values obtained from the initial calibration. No further sample analyses should be performed until an acceptable calibration is achieved.

15.3 Sample Analysis

15.3.1 Data Interpretation

15.3.1.1 Qualitative Determination

For a peak to be identified as a PCDD or PCDF, it must meet all of the criteria listed below.

15.3.1.1.1 The signals for the two m/z's being monitored must be present and maximize within ±2 seconds of each other.

15.3.1.1.2 The signal-to-noise ratio between the two m/z's must be ≥ 2.5 for native compounds and ≥ 10 for labeled compounds.

15.3.1.1.3 Ion ratios must be within the limits in Table 3.

15.3.1.1.4 Relative Retention Times

15.3.1.1.4.1 For Methods 0023A and 8290A, congeners which have an isotopically labeled compound must fall within -1 to +3 seconds of the labeled compound. Congeners with no labeled compound must be within 0.005 retention time units of the RRT measured in the continuing

calibration. (See Table 11.) For method TO-9a, congeners which have an isotopically labeled compound must fall within -3 to +3 seconds of the labeled compound. Congeners with no labeled compound must be within 0.005 retention time units of the RRT measured in the continuing calibration.

15.3.1.1.4.2 For Method 1613B, relative retention times must be within the RRT limits found in Table 9.

15.3.1.1.4.3 For non-2378 peaks, retention times must be within the retention time windows established by the analysis of the window defining mixture (Sec. 14.2.1.2).

15.3.1.1.5 For PCDFs, no peak may be present in the associated PCDPE channel at the same retention time. If a PCDPE peak is present, the PCDF peak should be reported with a flag denoting the interference.

15.3.1.1.6 Any sample in which 2378-TCDF has been identified at or above the method reporting limit must be confirmed on a second column (DB-225 or equivalent).

15.3.1.2 Calibration Limit Exceedance

15.3.1.2.1 If a compound in a sample exceeds the upper calibration limit, all subsequent samples must be checked for carryover contamination.

15.3.1.2.2 When a subsequent sample is non-detect for the compound in question, the sequence is again considered acceptable for reporting.

15.3.1.2.3 All affected samples between the exceeding sample and the non-detect sample must be re-analyzed.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

16.1 Preventive maintenance on a HRGC/HRMS system involves the following basic areas:

16.1.1 Vacuum pumps for the inlets, source, and analyzer need a change of oil about every year or when system performance indicates it is needed.

16.1.2 The GC injection port is cleaned as needed, approximately once a week. It is recommended that the septum and injection port liner be replaced at the time of cleaning. Additionally, the gold plated seal should be cleaned or replaced.

16.1.3 Ion source maintenance is usage dependent. The type and quantity of samples that have been injected determine the frequency of ion source cleaning and filament replacement.

16.1.4 Autosampler maintenance is primarily that of cleanliness. Most autosamplers need their moving parts to be clean and lightly lubricated. The most frequent corrective maintenance is that of changing the syringe, usually about once per month.

16.1.5 Instrument maintenance logs are kept with each instrument and serve as a record of all the maintenance that has been done on the instrument.

16.2 Non-Routine Maintenance Procedures (Special, Operational or Failure Mode Maintenance)

16.2.1 Service is provided to the instrument via the analyst, the in-house instrument service engineer, or a technical support specialist from the manufacturer. When instrument failure occurs, different parts of the instrument are isolated to determine the root cause. For example, the injection port may be capped off if a leak is suspected to prove the leak is/is not coming from that source. Instrument maintenance logbooks are kept for each instrument detailing the type of maintenance performed on the instrument and when it was performed. Preventive maintenance visits are scheduled annually for the mass spectrometers.

16.2.2 Analytical GC columns are clipped or replaced when the existing column shows signs of excessive degradation or the inability to properly resolve chromatographic peaks. Excessive peak tailing, poor responses, and baseline disturbances may also indicate that the column needs to be replaced.

17.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS

17.1 Data are evaluated qualitatively and quantitatively using a software program such as Waters MassLynx, or equivalent data system.

17.2 Data are reviewed, and a hard copy is generated. If manual integrations are made, a hard copy of the manual integration is printed and initialed by the analyst and included with the raw data.

17.3 Additional supporting documentation, such as totals pages generated by the software may be included with the data.

17.4 Quantitative Analysis

17.4.1 The concentration (ng/L for aqueous, ng/g for solids) of each identified compound in the sample is calculated as follows:

$$\left[PCDD/PCDF\right] = \frac{\left(A_{unk}^{ion1} + A_{unk}^{ion2}\right)}{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)} \times \frac{Q_{ES}}{W_{unk} \times D \times \overline{RF}}$$

Where:

A_{unk} and A_{ES} = the integrated area for each ion monitored.
Q_{ES} = the amount of extraction standard added to the sample in nanograms
W_{unk} = the initial sample aliquot size, in liters for waters and in grams for solids.
D = (% moisture in sample)/100, or 1 for waters
$\overline{RF}$ = Average RF from the ICAL for the compound

17.4.2 The estimated detection limit (EDL) is calculated as follows:

$$\left[EDL_{ppt}\right] = 2.5 \times \frac{\left(H_{unk}^{ion1} + H_{unk}^{ion2}\right)}{\left(H_{ES}^{ion1} + H_{ES}^{ion2}\right)} \times \frac{Q_{ES}}{W_{unk} \times \overline{RF}}$$

Where:

H_{unk} = the height of the noise present in each ion monitored.
H_{ES} = the height of the extraction standard peak in each ion monitored.
2.5 = signal-to-noise factor for minimum height of peak.

17.4.3 The estimated maximum possible concentration (EMPC) is calculated in the same manner as a concentration (Section 17.4.1).

17.4.4 The concentration of each extraction and cleanup standard is calculated as follows:

$$\left[ES_{ng}\right] = \frac{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)}{\left(A_{JS}^{ion1} + A_{JS}^{ion2}\right)} \times \frac{Q_{JS}}{\overline{RF}}$$

Where:

A_{ES} and A_{JS} = the integrated area for each ion monitored.
Q_{JS} = the amount of injection standard added to the sample in nanograms
$\overline{RF}$ = Average RF from the ICAL for the compound

The cleanup standard concentration is calculated as above, substituting the area of the individual cleanup standard ions for the extraction standard ions.

17.4.5 Percent recovery is calculated as follows:

$$\%R = \frac{R_{ng}}{S_{ng}} \times 100$$

Where:

R_{ng} = the amount of standard recovered in nanograms.
S_{ng} = the amount of standard spiked in nanograms.

18.0 POLLUTION/CONTAMINATION

18.1 Work area should be maintained free of dust and dirt accumulations.

18.2 Fume hoods are utilized to remove fumes and reduce the risk of airborne contaminants to ensure personnel safety. Hoods are monitored in accordance with CF-FC-E-003 for Fume Hood Face Velocity Performance Checks.

18.3 The laboratory area is restricted to authorized personnel.

19.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

19.1 A review process is used to insure the quality of the data. Raw data are reviewed first by the analyst, then by a second (peer) analyst or a data validator. When the analyst is satisfied that the data have been correctly processed and uploaded to the LIMS, a data report is generated from AlphaLIMS. The AlphaLIMS report along with the raw data and supporting documentation, such as a run log and case narrative, are submitted for review to the data validator or another experienced analyst. The reviewer goes through the raw data as if he/she was working it up for the first time and verifies that they are correct. In addition, he/she must make sure that the data have been correctly entered into AlphaLIMS. AlphaLIMS reports may be self-reviewed. If errors are discovered in either the raw data or the AlphaLIMS report, then the two analysts should discuss the differences and how best to resolve them. In some cases, the peer review process may

uncover errors that lead to a sample being re-extracted or re-run. In cases such as these, a nonconformance report (NCR) should be completed and submitted to the Quality department. It is recommended that a copy of the NCR be given to the prep analyst if it involves a re-extraction and that a copy be kept with the original data.

19.2 Once the data review has been completed by the reviewer, the batch is returned to the analyst for corrections (if applicable) and the status is updated from REVW to DONE in AlphaLIMS.

19.3 Data may be transmitted automatically to AlphaLIMS. This automatic "upload" procedure may be activated prior to data review or after data review is complete. In either case, the data recorded in AlphaLIMS are checked by the analyst for accuracy and completeness.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data may require instrument maintenance, re-analysis, re-extraction, or a more complex set of actions. When troubleshooting measures fail to bring an analytical process or data into control, a nonconformance report and/or corrective action should be initiated in accordance with CF-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items, and CF-QS-E-002 for Conducting Corrective Action.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting is used to determine the appropriate action to take when an initial or continuing calibration, blank and/or laboratory control sample fails to meet the acceptance criteria defined for the method. Troubleshooting may involve one or more of the following actions:

21.1 If analytes in a multi-point calibration fail to meet specified criteria, additional standards for the failing compounds may need to be reanalyzed. If they still do not meet specifications, instrument maintenance or new standards may be required before work is continued.

21.2 If a continuing calibration fails to meet specified criteria, instrument tuning or inlet maintenance may be required. If routine maintenance procedures fail to produce a second consecutive calibration verification within acceptance criteria, then the laboratory must demonstrate acceptable performance after further corrective action with two consecutive calibration verifications, or a new initial calibration must be analyzed.

21.3 If a method blank fails to meet defined criteria, the source of contamination should be found and eliminated before proceeding with analysis.

21.4 If normal equipment and software operating procedures do not resolve trouble-shooting efforts, the manuals for software, hardware and other equipment discussed in this SOP are available for consultation and resolution. On-line support may be available from software and instrument manufacturers, as well. Any revisions, repairs or corrective actions required must be documented in accordance with the laboratory's Quality System as described in CF-QS-B-001.

22.0 RECORDS MANAGEMENT

22.1 Run logs are generated for each instrument each day that the instrument is run. These run logs serve as records of what is run on the instrument, including samples, QC, calibrations, tunes, etc. Additional information is provided in the run log, including the analyst's initials, run date and time, and file name.

22.2 Raw data are stored in the lab in filing cabinets and/or boxes as long as there is space available. When space runs out, the data are boxed and sent to storage.

22.3 All records generated as a result of this procedure are maintained as quality documents in accordance with CF-QS-E-008 for Quality Records Management and Disposition.

23.0 LABORATORY WASTE HANDLING AND DISPOSAL

Sample extracts that have been run are temporarily stored in case they have to be reanalyzed. Once space is no longer available to keep them in the lab, they are moved to Waste Disposal where they are handled and disposed in accordance with the Laboratory Waste Management Plan, CF-LB-G-001.

24.0 REFERENCES

24.1 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Feb. 2007. Method 8290A, "Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatorgraphy/ High Resolution Mass Spectrometry (HRGC/HRMS)," Rev. 1, Feb. 2007. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.

24.2 Method 1613, "Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS," Rev. B, Oct. 1994. USEPA, Office of Water, Engineering and Analysis Division, 401 M Street SW, Washington, D.C. 20460.

24.3 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Feb. 2007. Method 0023A, "Sampling Method for Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofuran Emissions From Stationary Sources," Rev. 1, Dec. 1996. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.

24.4 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition. "Compendium Method TO-9A, Determination of Polychlorinated, Polybrominated and Brominated/Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in Ambient Air." January 1999. Center for Environmental Research Information, Office of Research and Development, USEPA, Cincinatti, OH 45268.

24.5 The NELAC Institute, (TNI) 2009 Standard, EL-V1-2009.

25.0 HISTORY

Revision 1: Section 15.3.1.2 added.

Revision 2: Absolute RT information added in 14.2.1.4; Calibration limit exceedance information added in section 15.3.1.2; Table 8 footnote describing RRT window adjustment to column used.

Revision 3: Method 0023A requirements added.

Revision 4: 2378-TCDF confirmation procedure and requirements added.

Revision 5: Injection standard changed from Tridecane to nonane. Discussion of equipment use and operation instructions was added, per DoD ELAP gray box 22.

Revision 6: Added TO-9a support and additional Tables for Method 8290.

Revision 7: Removed references to 8290 cleanup standard. Added TO-9a reference.

Revision 8: RRT limits for 1613 adjusted to method limits, except for three which have methods widths but db-5ms centers.

Revision 9: Added air matrix descriptions.

Revision 10: Table 9 updated. Maintenance rule for highly contaminated samples. TNI reference updated.

Revision 11: Changed EDL signal to noise value to 2.5. Updated Table references.

Revision 12: Added Table 13, Method Holding Times.

Revision 13: Added a NC requirement that a 1613 DW CCAL standard must only be obtained from a different manufacturer.

Revision 14: Adjusted TO-9a SS limits, added DB-5ms ui column use.

TABLE 1: METHOD ANALYTES AND PQLs

Analyte	Solid/Tissues (pg/g)	Aqueous (pg/L)	Air (pg)	CAS Number*
2378-TCDD	1	10	10	1746-01-6
12378-PeCDD	5	50	50	40321-76-4
123478-HxCDD	5	50	50	39227-28-6
123678-HxCDD	5	50	50	57653-85-7
123789-HxCDD	5	50	50	19408-74-3
1234678-HpCDD	5	50	50	35822-39-4
OCDD	10	100	100	3268-87-9
2378-TCDF	1	10	10	51207-31-9
12378-PeCDF	5	50	50	57117-41-6
23478-PeCDF	5	50	50	57117-31-4
123478-HxCDF	5	50	50	70648-26-9
123678-HxCDF	5	50	50	57117-44-9
234678-HxCDF	5	50	50	60851-34-5
123789-HxCDF	5	50	50	72918-21-9
1234678-HpCDF	5	50	50	67562-39-4
1234789-HpCDF	5	50	50	55673-89-7
OCDF	10	100	100	39001-02-0

* Chemical Abstract Services number

TABLE 2: MASS DESCRIPTORS

Function (#)	Channel (#)	Mass (amu)	Dwell Time (ms)	I.C. Delay (ms)
1	1	303.9016	50	10
1	2	305.8987	50	10
1	3	315.9419	50	10
1	4	304.9824	50	10
1	5	304.9824	(Lock)	10
1	6	317.9389	50	10
1	7	319.8965	50	10
1	8	321.8936	50	10
1	9	327.8847	50	10
1	10	331.9368	50	10
1	11	333.9339	50	10
1	12	339.8597	50	10
1	13	341.8568	50	10
1	14	375.8364	50	10
2	1	339.8597	50	10
2	2	341.8568	50	10
2	3	351.9	50	10
2	4	353.897	50	10
2	5	355.8546	50	10
2	6	357.8517	50	10
2	7	366.9792	50	10
2	8	366.9792	(Lock)	10
2	9	367.8949	50	10
2	10	369.8919	50	10
2	11	409.7974	50	10
3	1	373.8207	50	10
3	2	375.8178	50	10
3	3	380.976	50	10
3	4	380.976	(Lock)	10
3	5	383.8639	50	10
3	6	385.861	50	10
3	7	389.8156	50	10
3	8	391.8127	50	10
3	9	401.8559	50	10
3	10	403.853	50	10
3	11	445.7555	50	10
4	1	407.7818	50	10
4	2	409.7788	50	10
4	3	417.8253	50	10
4	4	419.822	50	10
4	5	423.7767	50	10
4	6	425.7737	50	10
4	7	430.9728	50	10
4	8	430.9728	(Lock)	10
4	9	435.8169	50	10
4	10	437.814	50	10
4	11	479.7165	50	10
5	1	441.7427	50	10
5	2	443.7398	50	10
5	3	454.9728	50	10
5	4	454.9728	(Lock)	10
5	5	457.7377	50	10
5	6	459.7348	50	10
5	7	469.778	50	10
5	8	471.775	50	10
5	9	513.6775	50	10

TABLE 3: THEORETICAL ION RATIOS AND CONTROL LIMITS

Level of Chlorination	Theoretical Ratio	Control Limits	
		Lower	Upper
4	0.77	0.65	0.89
5	1.55	1.32	1.78
6	1.24	1.05	1.43
6[a]	0.51	0.43	0.59
7	1.05	0.88	1.20
7[b]	0.44	0.37	0.51
8	0.89	0.76	1.02

[a] Used only for ^{13}C-HxCDF
[b] Used only for ^{13}C-HpCDF

TABLE 4: 1613B LIMITS FOR TETRA ONLY TESTS

Compound Name	Test Conc. (pg/µL)	CCAL Limits (pg/µL)	OPR Limits (pg/µL)	Sample Limits (pg/µL)
2,3,7,8-TCDD	10	8.2 - 12.3	7.3 - 14.6	-
2,3,7,8-TCDF	10	8.6 - 11.6	8.0 - 14.7	-
$^{13}C_{12}$-2,3,7,8-TCDD	100	85 - 117	25 - 141	31 - 137
$^{13}C_{12}$-2,3,7,8-TCDF	100	76 - 131	26 - 126	29 - 140
$^{37}Cl_4$-2,3,7,8-TCDD	10	8.3 - 12.1	3.7 - 15.8	4.2 - 16.4

TABLE 5: INITIAL CALIBRATION CONCENTRATIONS

	Concentration (pg/uL)				
Analyte	CS-0.5	CS-2	CS-3	CS-4	CS-5
2378-TCDD	0.25	2	10	40	200
2378-TCDF	0.25	2	10	40	200
12378-PeCDD	1.25	10	50	200	1000
12378-PeCDF	1.25	10	50	200	1000
23478-PeCDF	1.25	10	50	200	1000
123478-HxCDD	1.25	10	50	200	1000
123678-HxCDD	1.25	10	50	200	1000
123789-HxCDD	1.25	10	50	200	1000
123478-HxCDF	1.25	10	50	200	1000
123678-HxCDF	1.25	10	50	200	1000
123789-HxCDF	1.25	10	50	200	1000
234678-HxCDF	1.25	10	50	200	1000
1234678-HpCDD	1.25	10	50	200	1000
1234678-HpCDF	1.25	10	50	200	1000
1234789-HpCDF	1.25	10	50	200	1000
OCDD	2.5	20	100	400	2000
OCDF	2.5	20	100	400	2000
Extraction Standards					
^{13}C-2378-TCDD	100	100	100	100	100
^{13}C-2378-TCDF	100	100	100	100	100
^{13}C-12378-PeCDD	100	100	100	100	100
^{13}C-12378-PeCDF	100	100	100	100	100
^{13}C-23478-PeCDF	100	100	100	100	100
^{13}C-123678-HxCDD	100	100	100	100	100
^{13}C-123478-HxCDD	100	100	100	100	100
^{13}C-123478-HxCDF	100	100	100	100	100
^{13}C-123678-HxCDF	100	100	100	100	100
^{13}C-123789-HxCDF	100	100	100	100	100
^{13}C-234678-HxCDF	100	100	100	100	100
^{13}C-1234678-HpCDD	100	100	100	100	100
^{13}C-1234678-HpCDF	100	100	100	100	100
^{13}C-1234789-HpCDF	100	100	100	100	100
^{13}C-OCDD	200	200	200	200	200
Cleanup Standards					
^{37}Cl-2378-TCDD	0.25	2	10	40	200
Injection Standards					
^{13}C-1234-TCDD	100	100	100	100	100
^{13}C-123789-HxCDD	100	100	100	100	100

TABLE 6: METHOD 1613B LCS LIMITS

LCS Recovery Limits		
Analyte	**Amount Spiked**	**Limit**
	(pg/uL)	**(pg/uL)**
2378-TCDD	10	6.7-15.8
12378-PeCDD	50	35-71
123478-HxCDD	50	35-82
123678-HxCDD	50	38-67
123789-HxCDD	50	32-81
1234678-HpCDD	50	35-70
OCDD	100	78-144
2378-TCDF	10	7.5-15.8
12378-PeCDF	50	40-67
23478-PeCDF	50	34-80
123478-HxCDF	50	36-67
123678-HxCDF	50	42-65
123789-HxCDF	50	39-65
234678-HxCDF	50	35-78
1234678-HpCDF	50	41-61
1234789-HpCDF	50	39-69
OCDF	100	63-170
^{13}C-2378-TCDD	100	20-175
^{13}C-12378-PeCDD	100	21-227
^{13}C-123478-HxCDD	100	21-193
^{13}C-123678-HxCDD	100	25-163
^{13}C-1234678-HpCDD	100	26-166
^{13}C-OCDD	200	26-397
^{13}C-2378-TCDF	100	22-152
^{13}C-12378-PeCDF	100	21-192
^{13}C-23478-PeCDF	100	13-328
^{13}C-123478-HxCDF	100	19-202
^{13}C-123678-HxCDF	100	21-159
^{13}C-123789-HxCDF	100	17-205
^{13}C-234678-HxCDF	100	22-176
^{13}C-1234678-HpCDF	100	21-158
^{13}C-1234789-HpCDF	100	20-186
^{37}Cl-2378-TCDD	10	3.1-19.1

TABLE 7: METHOD 1613B ES (SAMPLES & LMB) RECOVERY LIMITS

Compound Name	Amount Spiked (pg/µL)	Limits %
$^{13}C_{12}$-2,3,7,8-TCDD	100	25 - 164
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	25 - 181
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	32 - 141
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	28 - 130
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	23 - 140
$^{13}C_{12}$-OCDD	200	17 - 157
$^{13}C_{12}$-2,3,7,8-TCDF	100	24 - 169
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	24 - 185
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	21 - 178
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	26 - 152
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	26 - 123
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	28 - 136
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	29 - 147
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	28 - 143
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	26 - 138
$^{37}Cl_4$-2,3,7,8-TCDD	10	35 - 197

TABLE 8: METHOD 1613B CONTINUING CALIBRATION LIMITS

Compound Name	CCAL (pg/µL)	Limits (pg/µL)	Compound Name	CCAL (pg/µL)	Limits (pg/µL)
2,3,7,8-TCDD	10	7.8 - 12.9	$^{13}C_{12}$-2,3,7,8-TCDD	100	82 - 121
1,2,3,7,8-PeCDD	50	39 - 65	$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	62 - 160
1,2,3,4,7,8-HxCDD	50	39 - 64	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	85 - 117
1,2,3,6,7,8-HxCDD	50	39 - 64	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	85 - 118
1,2,3,7,8,9-HxCDD	50	41 - 61	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	72 - 138
1,2,3,4,6,7,8-HpCDD	50	43 - 58	$^{13}C_{12}$-OCDD	200	96 - 415
OCDD	100	79 - 126	$^{13}C_{12}$-2,3,7,8-TCDF	100	71 - 140
2,3,7,8-TCDF	10	8.4 - 12	$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	76 - 130
1,2,3,7,8-PeCDF	50	41 - 60	$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	77 - 130
2,3,4,7,8-PeCDF	50	41 - 61	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	76 - 131
1,2,3,4,7,8-HxCDF	50	45 - 56	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	70 - 143
1,2,3,6,7,8-HxCDF	50	44 - 57	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	73 - 137
2,3,4,6,7,8-HxCDF	50	44 - 57	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	74 - 135
1,2,3,7,8,9-HxCDF	50	45 - 56	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	78 - 129
1,2,3,4,6,7,8-HpCDF	50	45 - 55	$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	77 - 129
1,2,3,4,7,8,9-HpCDF	50	43 - 58	$^{37}Cl_4$-2,3,7,8-TCDD	10	7.9 - 12.7
OCDF	100	63 - 159			

TABLE 9: METHOD 1613B RELATIVE RETENTION TIME LIMITS

Compound	RRT Reference	RRT Limits
2,3,7,8-TCDD	13C -2,3,7,8-TCDD	0.999 - 1.002
1,2,3,7,8-PeCDD	13C -1,2,3,7,8-PeCDD	0.999 - 1.002
1,2,3,4,7,8-HxCDD	13C -1,2,3,4,7,8-HxCDD	0.999 - 1.001
1,2,3,6,7,8-HxCDD	13C -1,2,3,6,7,8,-HxCDD	0.997 - 1.003
1,2,3,7,8,9-HxCDD	13C -1,2,3,6,7,8,-HxCDD	0.997 - 1.016
1,2,3,4,6,7,8-HpCDD	13C -1,2,3,4,6,7,8-HpCDD	0.999 - 1.001
OCDD	13C –OCDD	0.999 - 1.001
2,3,7,8-TCDF	13C -2,3,7,8-TCDF	0.999 - 1.003
1,2,3,7,8-PeCDF	13C -1,2,3,7,8-PeCDF	0.999 - 1.002
2,3,4,7,8-PeCDF	13C -2,3,4,7,8-PeCDF	0.999 - 1.002
1,2,3,4,7,8-HxCDF	13C -1,2,3,4,7,8-HxCDF	0.999 - 1.001
1,2,3,6,7,8-HxCDF	13C -1,2,3,6,7,8-HxCDF	0.996 - 1.004
2,3,4,6,7,8-HxCDF	13C -2,3,4,6,7,8,-HxCDF	0.999 - 1.001
1,2,3,7,8,9-HxCDF	13C -1,2,3,7,8,9-HxCDF	0.999 - 1.001
1,2,3,4,6,7,8-HpCDF	13C -1,2,3,4,6,7,8-HpCDF	0.999 - 1.001
1,2,3,4,7,8,9-HpCDF	13C -1,2,3,4,7,8,9-HpCDF	0.999 - 1.001
OCDF	13C –OCDD	1.002 - 1.011
13C -2,3,7,8-TCDD	13C -1,2,3,4-TCDD	0.986 - 1.053
13C -1,2,3,7,8-PeCDD	13C -1,2,3,4-TCDD	0.849 - 1.416
13C -1,2,3,4,7,8-HxCDD	13C -1,2,3,7,8,9-HxCDD	0.980 - 1.003
13C -1,2,3,6,7,8-HxCDD	13C -1,2,3,7,8,9-HxCDD	0.983 - 1.005
13C -1,2,3,4,6,7,8-HpCDD	13C -1,2,3,7,8,9-HxCDD	1.068 - 1.092
13C –OCDD	13C -1,2,3,7,8,9-HxCDD	1.050 - 1.329
13C -2,3,7,8-TCDF	13C -1,2,3,4-TCDD	0.904 - 1.084
13C -1,2,3,7,8-PeCDF	13C -1,2,3,4-TCDD	0.893 - 1.318
13C -2,3,4,7,8-PeCDF	13C -1,2,3,4-TCDD	0.869 - 1.384
13C -1,2,3,4,7,8-HxCDF	13C -1,2,3,7,8,9-HxCDD	0.960 - 0.986
13C -1,2,3,6,7,8-HxCDF	13C -1,2,3,7,8,9-HxCDD	0.962 - 0.988
13C -2,3,4,6,7,8,-HxCDF	13C -1,2,3,7,8,9-HxCDD	0.957 - 1.019
13C -1,2,3,7,8,9-HxCDF	13C -1,2,3,7,8,9-HxCDD	0.973 - 1.043
13C -1,2,3,4,6,7,8-HpCDF	13C -1,2,3,7,8,9-HxCDD	1.026 - 1.068
13C -1,2,3,4,7,8,9-HpCDF	13C -1,2,3,7,8,9-HxCDD	1.050 - 1.144
37Cl -2,3,7,8-TCDD	13C -1,2,3,4-TCDD	0.988 - 1.051

Due to the use of the DB-5ms column, some compounds exhibit slightly different elution times, resulting in RRT limits which vary from the method. The widths of the limits are the same as the method, only the center of the window has been adjusted to the DB-5ms's elution times.

TABLE 10: Method 8290 IS assignments

Internal Standard References
Method 8290

Analytes	Internal Standards
2378-TCDD	^{13}C-2378-TCDD
12378-PeCDD	^{13}C-12378-PeCDD
123478-HxCDD	^{13}C-123678-HxCDD
123678-HxCDD	^{13}C-123678-HxCDD
123789-HxCDD	^{13}C-123678-HxCDD
1234678-HpCDD	^{13}C-1234678-HpCDD
OCDD	^{13}C-OCDD
2378-TCDF	^{13}C-2378-TCDF
12378-PeCDF	^{13}C-12378-PeCDF
23478-PeCDF	^{13}C-12378-PeCDF
123478-HxCDF	^{13}C-123678-HxCDF
123678-HxCDF	^{13}C-123678-HxCDF
123789-HxCDF	^{13}C-123678-HxCDF
234678-HxCDF	^{13}C-123678-HxCDF
1234678-HpCDF	^{13}C-1234678-HpCDF
1234789-HpCDF	^{13}C-1234678-HpCDF
OCDF	^{13}C-OCDD
Extraction Standards	**Injection Standards**
^{13}C-2378-TCDD	^{13}C-1234-TCDD
^{13}C-12378-PeCDD	^{13}C-1234-TCDD
^{13}C-123678-HxCDD	^{13}C-123789-HxCDD
^{13}C-1234678-HpCDD	^{13}C-123789-HxCDD
^{13}C-OCDD	^{13}C-123789-HxCDD
^{13}C-2378-TCDF	^{13}C-1234-TCDD
^{13}C-12378-PeCDF	^{13}C-1234-TCDD
^{13}C-123678-HxCDF	^{13}C-123789-HxCDD
^{13}C-1234678-HpCDF	^{13}C-123789-HxCDD
Injection Standards	
^{13}C-1234-TCDD	NA
^{13}C-123789-HxCDD	NA

TABLE 11: 8290 Retention time limits

Retention Time Limits
Method 8290

Analytes	Description	Limits
2378-TCDD	2,3,7,8-substituted congeners, which have an isotopically-labeled standard present in the sample extract	must be within -1 to +3 seconds of the isotopically-labeled standard
12378-PeCDD		
123678-HxCDD		
123789-HxCDD		
1234678-HpCDD		
OCDD		
2378-TCDF		
12378-PeCDF		
123678-HxCDF		
123789-HxCDF		
1234678-HpCDF		
123478-HxCDD	2,3,7,8-substituted compounds that do not have an isotopically-labeled standard present in the sample extract	must fall within 0.005 retention time units of the relative retention time as determined from the daily routine calibration
23478-PeCDF		
123478-HxCDF		
234678-HxCDF		
1234789-HpCDF		
OCDF		
Total TCDDs	Non-2,3,7,8-substituted target compounds	must be within the corresponding homologous retention time windows established by analyzing the column performance check solution, relative to an isotopically-labeled standard in the sample
Total PeCDDs		
Total HxCDDs		
Total HpCDDs		
Total TCDFs		
Total PeCDFs		
Total HxCDFs		
Total HpCDFs		
^{13}C-2378-TCDD	Isotopically-labeled standards	No method limits: allowed to shift as long as the predicted RT of the native window defining isomers established by analyzing the column performance check solution remain within the descriptor switching time
^{13}C-12378-PeCDD		
^{13}C-123678-HxCDD		
^{13}C-1234678-HpCDD		
^{13}C-OCDD		
^{13}C-2378-TCDF		
^{13}C-12378-PeCDF		
^{13}C-123678-HxCDF		
^{13}C-1234678-HpCDF		
^{13}C-1234-TCDD		
^{13}C-123789-HxCDD		

TABLE 12: METHOD TO-9A MINIMUM REQUIREMENTS FOR INITIAL AND DAILY CALIBRATION

	ICAL	CVS
Unlabeled Analytes	(RSD)	(%D)
2,3,7,8-TCDD	25	25
2,3,7,8-TCDF	25	25
1,2,3,7,8-PeCDD	25	25
1,2,3,7,8-PeCDF	25	25
2,3,4,7,8-PeCDF	25	25
1,2,4,5,7,8-HxCDD	25	25
1,2,3,6,7,8-HxCDD	25	25
1,2,3,7,8,9-HxCDD	25	25
1,2,3,4,7,8-HxCDF	25	25
1,2,3,6,7,8-HxCDF	25	25
1,2,3,7,8,9-HxCDF	25	25
2,3,4,6,7,8-HxCDF	25	25
1,2,3,4,6,7,8-HpCDD	25	25
1,2,3,4,6,7,8-HpCDF	25	25
OCDD	25	25
OCDF	30	30
Internal Standards		
13C-2,3,7,8-TCDD	25	25
13C-1,2,3,7,8-PeCDD	30	30
13C-1,2,3,6,7,8-HxCDD	25	25
13C-1,2,3,4,6,7,8-HpCDD	30	30
13C-OCDD	30	30
13C-2,3,7,8-TCDF	30	30
13C-1,2,3,7,8-PeCDF	30	30
13C-1,2,3,4,7,8-HxCDF	30	30
13C-1,2,3,4,6,7,8-HpCDF	30	30
Surrogate Sampling Standards		
37Cl-2,3,7,8-TCDD	25	25
13C-2,3,4,7,8-PeCDF	25	25
13C-1,2,3,4,7,8-HxCDD	25	25
13C-1,2,3,4,7,8-HxCDF	25	25
13C-1,2,3,4,7,8,9-HpCDF	25	25

TABLE 13: METHOD HOLDING TIMES

Method	Collection to Extraction	Extraction to Analysis
8290A *	30 days	45 days
1613B	365 days	365 days
DLM02.2	365 days	365 days
M23	30 days	45 days
TO-9a	7 days	40 days
CBC01.2	35 days collect to analysis	
1668A/C	365 days	365 days

* NOTE: The holding times listed in method 8290 are recommendations. PCDDs and PCDFs are very stable in a variety of matrices, and holding times under the conditions listed in this section may be as long as a year for certain matrices.

FIGURE 1: 2378-TCDD CHROMATOGRAPHIC SEPARATION

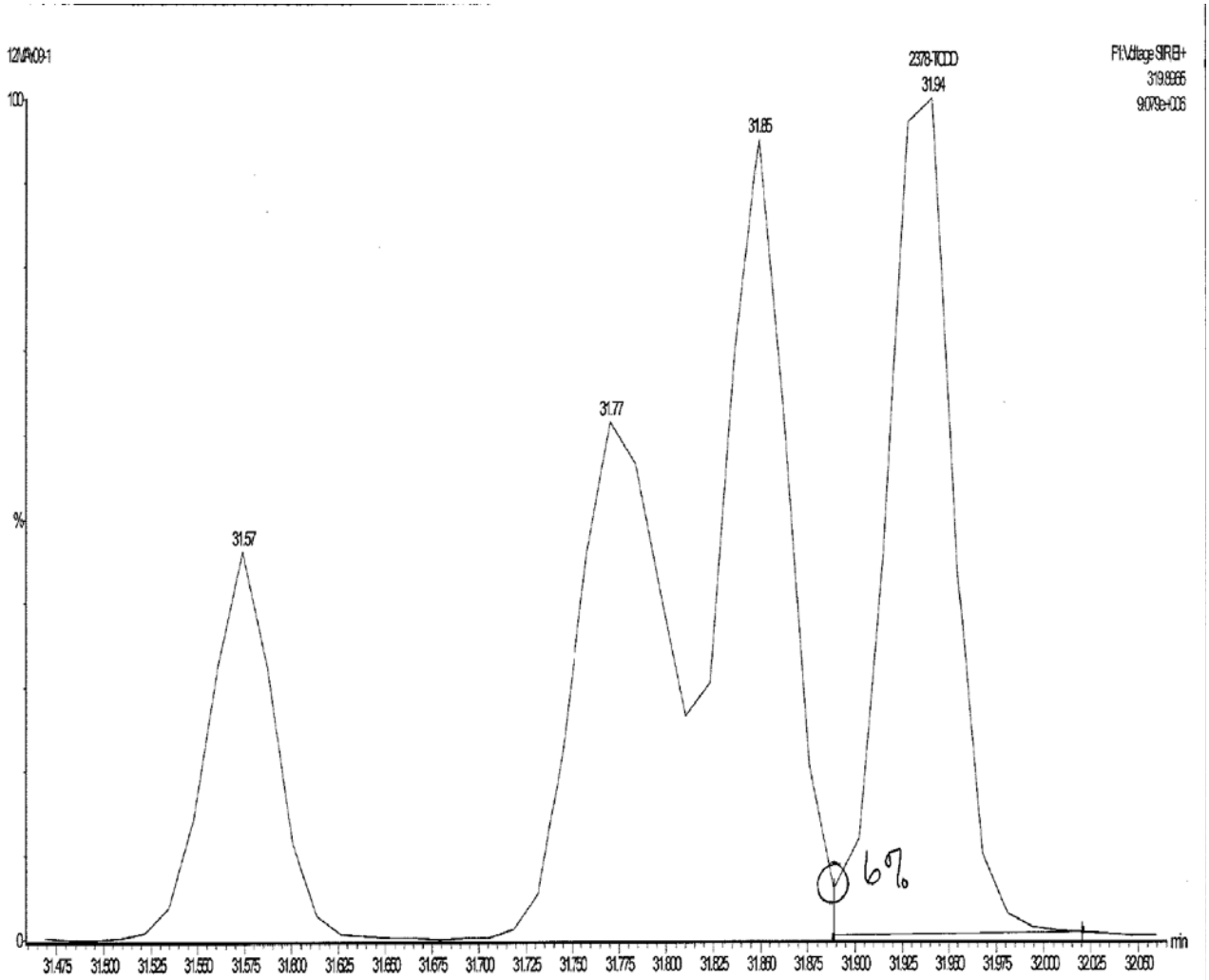


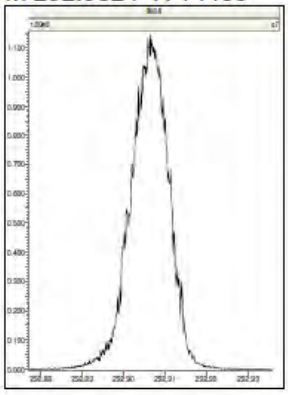
FIGURE 2: INSTRUMENT RESOLVING POWER (EXAMPLE)

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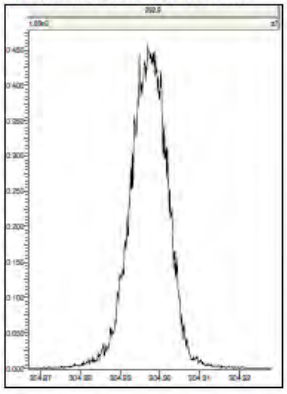
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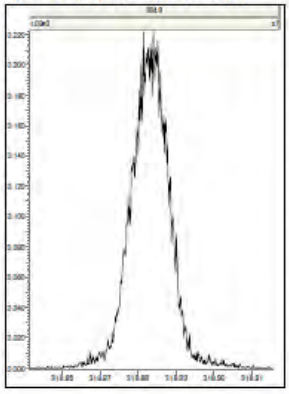
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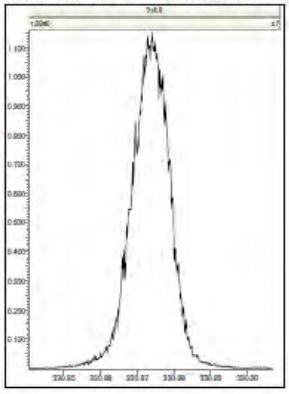
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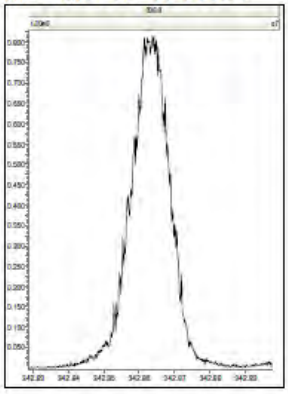
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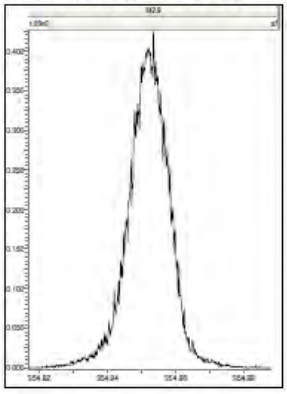
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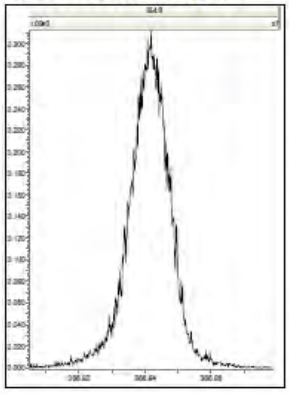
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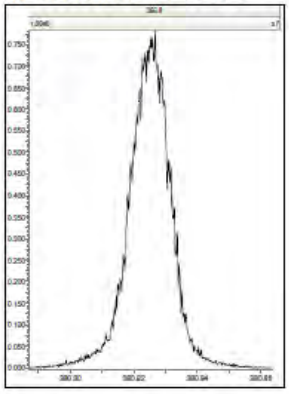
M 354.9792 R 12502



M 366.9792 R 12500



M 380.9760 R 13093



Determination of Organochlorine Pesticides By Gas Chromatography/Electron Capture Detection (GC/ECD)

References: Method 8081B Rev. 2, Update IV, Feb 2007, and Method 8000C Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final Update III, December 1996 (USEPA, Office of Solid Waste and Emergency Response, Washington, DC).

USEPA Contract Laboratory Program Statement of Work for Organics Analysis Multi-Media, Multi-Concentration" August 1991. Document Number OLM03.1 and OLM04.2.

Massachusetts Contingency Plan (MCP) WSC-CAM for SW846 Method 8081 Pesticides by GC/ECD. Section VB Rev4. August 2004 (Massachusetts Department of Environmental Protection Bureau of Waste site cleanup)

1. Scope and Application

Matrices: This method is applicable to the quantification of Organochlorine Pesticides in water, soil, sediment, sludge, product, and tissue (either animal or vegetative).

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the analysis and quantification of sample extracts for Organochlorine Pesticides by gas chromatography/electron capture detector. Target analytes include a wide range of pesticides, a basic list of which is included below. The sensitivity of the method usually depends on the concentration of interferences as well as instrumental limitations. If interferences prevent detection of the analytes, the method may also be performed on samples that have undergone cleanup. Typical reporting limits are based on the lowest level of the initial calibration curve and generally correspond to limits derived from quarterly LOQ studies to verify our MDLs.. Detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed.

This method is in substantial conformance with the guidelines established in SW-846 for performing chromatographic analysis as defined in the Method 8000B ("Gas Chromatography), Method 8081B ("Organochlorine Pesticides"), Method 3510C ("Separatory Funnel Liquid - Liquid Extraction"), and Method 3570 Microscale Extraction. The data quality objectives of these methods are met or exceeded by this SOP. The following extraction and cleanup methods may also apply, depending on project specifications:

- Extraction of Water Samples by Separatory Funnel (2156)
- Tissue Preparation and Homogenization (2166)
- Shaker Table Extraction (2261)
- Sulfur Cleanup (2168)
- Microscale Solvent Extraction - 3570 (2172)
- Silica cleanup (2170)
- GPC Cleanup by HPLC (2167)

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory

personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/ECD and in the interpretation of GC/ECD data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Table 2: Target Analytes for Pesticides by GC/ECD

Parameter	CAS
alpha-BHC	319-84-6
beta-BHC	319-85-7
gamma-BHC (Lindane)	58-89-9
delta-BHC	319-86-8
Heptachlor	76-44-8
Aldrin	309-00-2
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2
Heptachlor epoxide	1024-57-3
Oxychlordane	27304-13-8
Dieldrin	60-57-1
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
2,4'-DDD	53-19-0
2,4'-DDE	3424-82-6
2,4'-DDT	789-02-6
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Methoxychlor	72-43-5
Mirex	2385-85-5
Cis-Nonachlor	5103-73-1
Trans-Chlordane	5103-74-2
Hexachlorobenzene	118-74-1
Toxaphene	72-43-5
Chlordane	57-74-9

2. Summary of Method

An aliquot of a well mixed, homogeneous aqueous, solid, or tissue sample is accurately measured or weighed for sample preparation. Generally, 500mL or 1000mL of water sample, 1-5g of tissue sample, or 5g to 30g of sediment/soil sample is extracted. Water, soil/sediment, and tissue samples are spiked with surrogate compounds and then extracted using methylene chloride or a methylene chloride/acetone mixture. The extract is dried and exchanged into hexane during sample concentration. After extraction, cleanup techniques are applied. Generally extracts are cleaned by GPC (for hydrocarbon or lipid removal) and silica cartridge or automated silica column followed by copper (for sulfur removal). The extract is exchanged into hexane and concentrated to the appropriate volume, generally 5mL or 10mL, for analysis. Alternatively, this method can be employed for lower detection limits by decreasing the final volume to 1-5 mL.

After cleanup and re-concentration, the extracts are spiked with internal standards, and analyzed on a gas chromatograph, which is fitted with two capillary columns of differing polarities each employing a separate detector. The target analytes are resolved on each column and detected using an electron capture detector (ECD). Analytes are introduced into the GC/ECD by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC that is temperature and flow programmed to separate the compounds of interest. Identification of target analytes is accomplished by confirming a target hit on two dissimilar columns using Retention Time and Pattern Recognition. Concentrations are calculated from the ECD response using internal standard techniques, additionally external standard techniques can also be utilized circumstantially. Concentrations are determined using Mean Relative Response Factor from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards.

2.1 Method Modifications from Reference

This method exhibits some modification from the reference method. Method 8081B discusses the allowed shelf life for stock solutions as one year and working solutions as six months. The standard expiration protocol for the lab is the same however, if there is a significant amount of working spike or surrogate solution expired after the six-month period the lab will re-QC the solution. If the QC of the solution still passes the lab will extend the expiration date by another month. The method also indicates that a mid level multi-component standard is analyzed with the initial calibration for multi component compounds such as Toxaphene and Chlordane. The current lab procedure is to run the lowest possible level of the multi-component standard and include it with the Initial Calibration so the analyst can determine the lowest level at which the multi-component targets can be detected. If the multi-component analyte is then detected in a sample extract the analyst will then analyze a mid level standard for a single-point quantitation.

3. Reporting Limits

Table 1 lists the routine reporting limits.

4. Interferences

4.1 Contaminants in solvents, reagents, glassware, syringes, and other sample processing hardware may cause inferences that lead to discrete artifacts, biases, and/or elevated baselines. Demonstrate that all of these materials are free from interferences under the conditions of the preparation and analysis by extracting and analyzing a laboratory method blank with each batch of up to 20 samples. Also new solvent lots are to be prescreened and approved by GC/ECD analyst prior to use. All glassware and equipment should be

properly cleaned and solvent rinsed prior to use. Never use any glassware, or other implements such as syringes, on multiple samples within the batch unless there has been thorough cleaning and decontamination between samples.

4.2 Contaminants co-extracted from the sample may cause matrix interferences. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the environment and matrix being investigated. In some cases depending on the severity of the observed interferences, a target hit on one column may be biased high when interfered with. Skilled analyst judgment is imperative in determining if a chromatographic peak appears interfered with. In these cases the non-interfered, non-biased hit can be forced for reporting purposes providing a more suitable value. In extreme cases of interference additional cleanups can be employed, or it may be necessary to analytically re-run the sample at a dilution.

4.3 The ECD is theoretically a halogen specific detector. However, phthalate esters can be a major source of contamination in that they do respond to the ECD and can interfere with accurate determination of the compounds of interest. No forms of plastic or any material containing plasticizers (phthalates) should be used in conjunction with any part of this analysis.

4.4 Elemental sulfur is readily extracted from soil and sediment samples and may cause chromatographic interferences in the determination of pesticides. Sulfur can be removed through the use of Method 3660B.

4.5 Care must be taken to ensure a complete hexane exchange is performed on the extract, because residual methylene chloride would be an interferent in the analysis. Residual methylene chloride can also damage the electron capture detectors.

4.6 The injector system of the gas chromatograph can become contaminated with non-volatile components from samples. This can act to decrease the resolution and responsiveness of the system. The analysis of a DDT/Endrin breakdown standard is the best indicator of the condition of the injector system and the condition of the column. As the percent breakdown increases gradually the system should undergo general maintenance (liner changes, injection port cleaning, etc.) to avoid prolonged down time.

4.7 Aldol condensation products (4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) can arise during soil extraction when using methylene chloride: acetone. These products may interfere with detection of analytes and should be monitored.

4.8 Raw GC/ECD data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples should be collected in 500mL, 1L or 2L amber glass bottles and stored without preservative at 4°C. The minimum amount of sample needed to reach the reporting limits on Table 1 in Section 16 for this method for aqueous samples is 500mL. Additional sample is needed (approximately 3X the minimum amount) if MS/MSD analyses are to be performed.

Soil/sediment and Tissue samples should be collected in glass soil jars and stored at 4°C, or if desired, frozen. The minimum amount of sample needed to reach the reporting limits on Table 1 in Section 16 for solid and tissue matrices is 50g. Additional sample is needed (approximately 3X the minimum amount) if MS/MSD analyses are to be performed.

6.2 Sample Preservation

Aqueous samples should be stored without preservative at 4°C.

Soil/sediment and Tissue samples should be stored at 4°C, or if desired, frozen.

6.3 Sample Shipping

Sample Receipt & Login SOP 1559 describes how samples are normally shipped or obtained by the laboratory, precautions to be used in opening sample shipments, and sample storage conditions.

6.4 Sample Handling

The hold time for this method is 7 days for the extraction of aqueous samples and 14 days for the extraction of solid and tissue samples. If a sediment or tissue samples are frozen, this suspends the holding time until removal from the freezer. All extracts must be analyzed within 40 days of the extraction date. Extracts are stored in a refrigerator at 2-6ºC.

7. Equipment and Supplies

7.1 Gas Chromatograph: The instrumentation is a temperature-programmable gas chromatograph with heating range from 40ºC to 325ºC and splitless-type inlet system with electron capture detectors (Hewlett-Packard HP 5890 Series II GC, HP 6890 Plus, or similar).

7.2 Injection Port: Designed for splitless injection onto a capillary column. The injection port utilizes a uniliner for direct injection, to decrease the potential for breakdown of compounds in the injection port and to increase sensitivity. These uniliners are used in place of the standard splitless liners. The 6890 model requires a drilled uniliner due to Electronic Pressure Control (EPC). The injection port will require maintenance on an as needed basis if degradation or contamination is apparent. Please refer to the front of the *Instrument Maintenance Logbook, which* outlines the routine maintenance procedures

7.3 Columns and supplies

7.3.1 Restek's proprietary "RTX-5" Stationary Phase, 60m length x 0.25mm ID, 0.25□m film thickness - Restek Corporation, PN 10226, or equivalent.

7.3.2 Restek's proprietary "RTX-CLPesticide II" Stationary Phase, 60m length x 0.25mm ID, 0.2□m film thickness - Restek Corporation, PN 11326, or equivalent.

7.3.3 Guard column, deactivated fuse silica phenyl-methyl, 5 m x 0.32 mm ID. Restek PN 10027 or equivalent.

7.3.4 flow from the guard column can be split between the two analytical columns using a Siltek MXT-Y connecter Restek PN 20396

7.4 Guard Column: The GC/ECD utilizes one injector with guard column sealed to the uniliner inside the injection port. The guard column is split by connector into the two dissimilar columns listed above. Each column is then attached to a separate detector. The Rtx-5 is always the front detector column and the CLP II is the back detector column.

7.5 Auto sampler: Adapted onto the Gas Chromatograph. Model is HP 6890 series autosampler with a GC autosampler controller and HP 5890 series autosampler with controller, or equivalent.

7.6 Computer: with Windows XP operating software utilizing HP Enviroquant G1701DA Version D.03.00 software, or equivalent.

7.7 Gases: Hydrogen, ultra high purity grade (99.9999% pure) and Nitrogen of equivalent purity. Hydrogen is generally supplied by a Proton Hogen GC 600 Hydrogen generator.

7.8 Syringes: Hamilton Gas tight, various sizes.

7.9 Glass Pasteur Pipettes

7.10 Volumetric Flasks: Class A, various sizes

7.11 Vials: 2ml, 4ml, 10ml

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. See SOP *Reagent, Solvent, and Standard Control* (1816) for additional details regarding solvent purity. All solvent expirations determined as indicated by manufacturer guidelines

Analytical Standards are stored at 2-6ºC, according to manufacturer's recommended procedure. Primary standards are discarded as indicated by the vendor expiration. Stock standards are given 1 year expiration from the preparation date or the expiration of the primary vendor solution, whichever occurs first. Working standards are given a six month expiration from the preparation date or the expiration of the primary solution whichever occurs first. If breakdown of a solution is observed the solution will be discarded. All analytical standards are prepared in Hexane. All prep standards are prepared in Acetone or Hexane.

8.1 Methylene Chloride: ACS approved, Pesticide grade.

8.2 Acetone: ACS approved, Pesticide grade.

8.3 Hexane: ACS approved, Pesticide grade.

8.4 Methanol: Purge and Trap grade.

8.5 Surrogate Spiking Solution: Two surrogates are used for this method, 2,4,5,6-tetrachloro-m-xylene (TMX) and Decachlorobiphenyl (DCB). Alternate surrogates may be employed to avoid co-elutions or when DCB is a target analyte. Example alternate

surrogates would be DBOB (4,4'-Dibromooctafluorobiphenyl) BZ-198 (2,2',3,3',4,5,5',6 Octachlorobiphenyl). Surrogate Spiking solutions are stored in 40ml pre-cleaned amber VOA vials and placed in a standards freezer in the preparatory lab. The Pest/PCB Surrogate is made from the *Pesticides Surrogate Standard Spiking Solution* (catalog # ISM-320) commercially obtained from Ultra at a concentration of 200ug/mL. The spiking solution is the "Pest/PCB Surrogate" spike, which is made by diluting the primary standard appropriately to achieve a concentration of 1000ug/L. All solutions are made volumetrically in Acetone at desired final volume. Generally 200ml of standard is made but this can vary depending on use, need, and preference. The solution must be assayed by analysis before release to the preparation lab. Generally 1ml of surrogate spiking solution is spiked into the samples and QC. Alternative solution concentration and spiking amount can be used.

8.6 LCS and Matrix spiking solution: Two Pest LCS Spiking solutions are generally used in this method. Alternatively, solutions with project specific targets may be used. The LCS spiking solutions are stored in the same manner as surrogate solutions. The Pest LCS solutions are made from a combination of Restek Pest Mix 8081AB#1 cat#32291 at concentration 200ug/ml and Hexachlorobenzene and Additional Pesticide Mix (Quote#041310-632 from Ultra at a concentration of 100ug/ml. The spiking solution is labeled "Pest LCS" spike and is prepared by diluting the appropriate aliquot of each of the two primary solutions to obtain a spike solution concentration of 1000ug/L. All solutions are made volumetrically in Acetone at desired final volume. Generally 100ml of spiking solution standard is made but this amount can vary depending on use, need and preference. Generally 1 mL of LCS spiking solution is added into each laboratory control spike, matrix spike, and spike duplicate QC samples. Alternative solution concentration and spiking amount can be used.

8.7 Internal Standards (IS): PCB congener BZ 192 (2,2',3,4,4',5,6,6'-octachlorobiphenyl This solution is commercially obtained from Ultra at 100ug/mL. The IS solution is prepped by making a volumetric dilution of the primary standard in hexane to achieve a concentration of 2.5ug/ml. For method quantification by internal standard 20ul of internal standard is added into 1ml extracts, to produce a mid-calibration range response on column at 50ng/mL in final sample extract.

8.8 Breakdown Mix: This solution is commercially obtained from Ultra (PN ISM-450) containing Endrin at 1 μg/mL and 4,4'-DDT at 2ug/ml. The primary solution is volumetrically diluted to achieve a working standard concentration of 100ug/L for Endrin and 200 ug/L for 4,4'-DDT.

8.9 Toxaphene Calibration solution: Toxaphene solution is commercially obtained from Ultra (PP-270) at a concentration of 100ug/ml. The stock solution at the concentration of 10,000ug/L is prepared as an intermediate solution. The stock solution is diluted volumetrically to obtain a mixture at the concentration of 25ug/L. The 25ug/L solution is injected as a low standard and run with the calibration curve. If Toxaphene is detected an additional mid level Toxaphene standard is used for the one point quantitation of this compound. The stock solution is appropriately diluted volumetrically to obtain a concentration of 1000 ug/L for the Toxaphene and includes TMX/DCB at 100ug/L.

Toxaphene ICV solution is commercially obtained from NST (111-M) at a concentration of 2000ug/ml. The stock solution at the concentration of 20,000ug/L is prepared as an intermediate solution. The stock solution is appropriately diluted volumetrically to obtain a concentration of 1000 ug/L for the Toxaphene ICV working standard.

8.10 Chlordane Calibration solution: Chlordane solution is commercially obtained from Ultra (PP-151) at a concentration of 100ug/ml. The stock solution at the concentration of 10,000ug/L is prepared as an intermediate solution. The stock solution is diluted

volumetrically in hexane to obtain a mixture at the concentration of 25ug/L. The 25ug/L solution is injected as a low standard and run with the calibration curve. If Chlordane is detected and additional mid level Chlordane standard is utilized for the one point quantitation of this compound. The stock solution is appropriately diluted volumetrically to obtain a concentration of 500ug/L for Chlordane and includes TMX/DCB at 100ug/L

Chlordane ICV solution is commercially obtained from Restek (32021) at a concentration of 1000ug/ml. The stock solution at the concentration of 20,000ug/L is prepared as an intermediate solution. The solution is appropriately diluted volumetrically to obtain a concentration of 500 ug/L for the Toxaphene ICV working standard.

8.11 Independent Calibration Verification (ICV) standard: This is a secondary source standard separate from the calibration curve standards containing target pesticides compounds. The ICV can be made two ways as a combination of "Custom Standard – Pesticide Mix" (Quote#041310-631) and "Custom Standard – Additional Pesticide Mix" (Quote#041310-632) both commercially obtained from Ultra at a concentration of 100ug/ml. Alternatively, use Restek Pest Mix 8081AB#1 cat#32291 at concentration 200ug/ml and Hexachlorobenzene and Additional Pesticide Mix (Quote#041310-632) from Ultra at a concentration of 100ug/ml. Cis-Nonachlor obtained from Ultra at (PP-490) concentration 100ug/ml and Chlorpyriphos obtained from Restek (32212) at concentration 1000ug/ml are added to both mixtures to obtain all targets compounds. The ICV is prepared by diluting the appropriate aliquot of each of the two primary solutions to obtain a spike solution at the concentration of 50ug/L. This standard must be spiked with 20uL of the internal standard as referenced in section 8.7.

8.12 SRM: *1944 – New York/New Jersey Waterway Sediment, and SRM 2974a – Organics in Freeze Dried Mussel Tissue*, from National Institute of Standards & Technology (NIST). Please refer to the individual certifications for the assigned true values. These SRMs may be extracted and analyzed with sample batches as part of the overall QC evaluation, if requested by the client. Other certified SRMs may be used on a project specific basis.

8.13 Pesticide Calibration Standard: Commercially obtained primary solutions are used to volumetrically create stock solutions. All stock solutions are made by adding the appropriate amount of primary standard to a 25 mL volumetric flask to obtain the concentration of 5000 ug/L. The stock solutions are then used in various combinations and serial dilutions to make calibration curve standards (see Table 3).

Calibration standards are always prepared in hexane. The basic calibration curve is made from MCP Pest Stock, Additional Pest Stock, Chloropyrifos/Cis-nonachlor Stock and DBOB/BZ198 Surrogate Stock. MCP Pest Stock is derived from primary *Custom Pesticide Standard (PN # S-9553-SS)* commercially obtained from Accustandard, Additional Pest Stock is made from primary *Custom Pesticide Standard (PN#S-14378-R1)* commercially obtained from Accustandard, and DBOB/BZ198 Surrogate Stock is prepared from primary surrogate solutions commercially obtained from Ultra: *DBOB* – at a concentration of 5000 ug/mL *(catalog # PPS-172) and BZ#198* – at a concentration of 100 ug/mL *(catalog # RPC-075S).* Chloropyrifos/Cis-nonachlor Stock is prepared from primary solutions commercially obtained from Accustandard at a concentration of 100 ug/mL: Chloropyrifos (Dursban) *(catalog # P-094S) and Cis-nonachlor (catalog # P-297S).*

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

9.1.1 Method Blank

9.1.1.1 A method blank must be extracted (spiked with surrogates) and analyzed once per every 20 samples or per extraction batch, whichever is more frequent.

9.1.1.2 Because the analysis of a method blank is used to determine background contamination, an *acceptable* method blank should not contain any individual compound at the concentration of reporting limit, or above. For contaminated blanks all efforts must be made to identify and eliminate the source of contamination. The presence of analytes at concentrations at or above the reporting limit will warrant application of a “B” qualifier to that target compound(s) on all associated report forms, and perhaps re-extraction of all associated samples. The results are qualified with a “B” for any associated sample concentrations that are less then 10x the blank concentration for the analyte. Surrogate and internal standard recoveries must meet the QC limits for the method blank, see Sections 9.7.1 and 9.7.2. Re-extraction must be initiated immediately so that minimum time is wasted before re-extraction can occur - if at all possible, this re-extraction should take place within holding time. Re-extraction *corrective action* that would exceed the sample holding time criteria should be discussed with the client, Laboratory Director, QA Manager, and/or Section Supervisor prior to implementation. Exceptions may be made with approval of the Section Supervisor if the samples associated with an out of control method blank are non-detect for the affected compound(s) or if the concentration of the affected compound(s)in the sample is greater than 10x the blank level. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified, via the project narrative, of any method blank non-compliance associated with the sample results.

9.2 Laboratory Control Sample (LCS)

9.2.1 The laboratory control sample (LCS/LCSD) generally contains all Pesticides listed in Table 1, Section 16 except for Toxaphene & Chlordane. The number of Pesticide compounds in the LCS may vary with client specific requests. The Pesticide list reported to the client on the recovery form depends on the target list requested by the client and may also vary between different projects. The LCS/LCSD is extracted along with the samples and must be extracted and analyzed once per every 20 samples or per extraction batch, whichever is more frequent.

9.2.2 The acceptable recovery QC limits as well as RPD limits are found in Table 2 Section 16 for an aqueous, solid, and tissue LCS/LCSD. All recovery limits should

be monitored and documented in-house through control charts and updated as needed.

9.2.3 Corrective Action: Repeat analysis or check to see if an analytical or spiking error has occurred. If the LCS/LCSD recoveries are still out of control, re-extract and re-analyze the LCS/LCSD and all associated samples. Samples cannot be approved until an acceptable LCS is obtained. Exceptions may be made with approval of the Section Supervisor if the samples associated with the out of control LCS are also associated with a matrix spike and matrix spike duplicate that is in control which demonstrates an isolated problem pertaining to the LCS only. An explanation of this out of control LCS/LCSD recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS/MSD results as batch QC. For specific work plans the project requirements may supersede existing acceptance criteria. E.g., if the LCS/LCSD do not meet quality control criteria for recovery of some of the spike and surrogate compounds, the results for the other samples and quality control samples within the batch must be evaluated to determine if this is an isolated problem for the LCS/LCSD and whether the data should be reported with the affected LCS/LCSD.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.4

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4

9.5 Matrix Spike

9.5.1 Matrix spike and matrix spike duplicate analyses are performed on a project specific basis. If a project calls for a MS/MSD one is performed per 20 samples and per matrix.

9.5.2 The acceptable recovery and RPD QC limits are found in Table 2 for an aqueous, solid, tissue, and product MS/MSDs. All recovery limits should be monitored and documented in-house through control charts and updated as needed.

9.5.3 Corrective Action: Repeat analysis or check to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the control limits and the associated LCS is within control, include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the affected results as evidenced by the matrix spike and matrix spike duplicate exceedance.

9.6 Laboratory Duplicate

9.6.1 Laboratory matrix or sample duplicates are analyzed if requested by the client. The QC limit is 50% RPD for target compounds.

9.6.2 Corrective Action: If the %RPD exceeds the 50% control limit and the associated LCS/LCSD %RPD is within 50%, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results isolated to this sample, as evidenced by the matrix duplicate exceedance and the LCS/LCSD acceptance. If an MS/MSD was also extracted with the batch and the sample/duplicate as well as the MS/MSD exceed the control limits, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results as evidenced by the sample/duplicate and the MS/MSD exceedences.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogate

9.7.1.1 Surrogates are monitored for recovery for all matrices. Two surrogates are used, one that elutes at the beginning and one at the end of GC run. Every sample, blank, and quality control sample (LCS, LCSD, MS, MSD, Dup) must be spiked with the surrogates prior to extraction. The recovery limits are found in Table 2. The recovery limits should be monitored and documented in-house through control charts and updated as necessary.

9.7.1.2 Corrective Action: Check to see if an analytical, spiking, or dilution error occurred and re-calculate. If only one surrogate falls below the recovery limit and all QC samples have acceptable recovery indicating a matrix effect isolated to the sample, the exceedance is noted, with approval of the Section Supervisor, and the results are reported to the client with a notation in the case narrative. If all surrogates are recovered below the limit or there are outliers in any QC, re-extract the sample. If re-extraction occurred beyond the holding time and the re-extract surrogates are within the QC limits, report the re-extract results along with the original results. If the surrogates are recovered below the limit in the re-extract, this confirms suspected matrix interference on the surrogates, and only the original analysis needs to be reported unless a specific QAPP requires otherwise. If the chromatogram shows obvious matrix interference, no re-analysis or re-extraction is necessary. *This decision must be made with approval of the Section Supervisor and/or Data reviewer.* Surrogate outliers and sample re-extracts must be noted in the case narrative to the client.

9.7.2 Internal Standards

9.7.2.1 Internal standards are added to every field sample, QC sample, standard, and method blank. The Daily opening CCV IS areas are compared to the ICAL; for subsequent samples analyzed following CCVs – the IS areas are compared to the nearest opening CCV for each sample. The acceptance limits are -50% to +200% of the internal standard response (or area) of the daily continuing calibration verification standard.

9.7.2.2 Corrective Action: Check to see if an analytical, dilution or spiking error occurred. If the chromatogram shows obvious matrix interference, no re-analysis is necessary. *This decision must be made with approval of the Section Supervisor.* Note the exceedance in the case narrative to the client. If no obvious interference is present, re-analyze the extract. If internal standards are now within the acceptance limits, report only the re-analysis, as long as the re-analysis occurred within the 40-day analytical hold time. If the re-analysis occurred outside of the 40-day analytical hold time, both the original and re-analysis must be reported. If the internal standards again are outside the acceptance limits, after re-analysis, either within or outside of the 40-day hold time, report only the original analysis, and include a narrative to the client that the suspected matrix interference on the internal standards was confirmed by sample re-analysis.

9.7.3 SRMs

9.7.3.1 Standard reference materials (SRMs) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples on a project specific basis. These are not used as controls, but to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

9.7.3.2 Acceptance criteria for SRM analysis will vary from project to project depending upon client data quality objectives (DQOs). Generally, 40% - 140% recovery based on the true certified values of the target compounds of interest, serve as advisory acceptance criteria.

9.7.3.3 Corrective Action: Repeat analysis and/or check to see if an analytical error has occurred. If the % recovery still exceeds the control limits and the associated LCS and/or MS/MSD are within control, include a project narrative with the results to client noting that the observed recovered of the SRM are isolated to this sample as evidenced by the LCS and/or MS/MSD acceptance.

9.7.4 Evaluate the percent degradation of 4,4'-DDT (to 4,4'-DDE and 4,4'-DDD) and Endrin (to Endrin Aldehyde and Endrin Ketone) in the Performance evaluation mixture (PEM) to monitor the integrity of the injection system (see section 11.10 for calculation). The breakdown mixture must be analyzed daily to verify that degradation in the system is not excessive. Degradation is not considered to be a problem if the percent degradation of 4,4'-DDT and Endrin are ≤15%. If either compound does exceed 15% breakdown, the analysis must be stopped, the injection port serviced and other maintenance may need to be performed.

9.8 Method Sequence

PEM3060501	(PEM)
C3060501	(CCV at 50ug/L)
Prep Blank ID	Method Blank
LCD ID	LCS
LCSD ID	LCSD
Sample ID	about 10 samples
PEM3060502	(PEM)
C3060502	(CCV at 50ug/L)
Sample ID	about 10 samples
PEM3060503	(PEM)
C3060503	(CCV at 50ug/L)
Sample ID	about 10 samples
C3060504	(Closing CCV at 50ug/L)

10. Procedure

10.1 Equipment Set-up

10.1.1 Prior to the analysis of any standards or samples, the instrument acquisition and process methods must be set up. This includes the GC run parameters and retention time windows. An initial calibration must be analyzed to establish linearity of the instrument. The target analytes are resolved on each column and detected using an electron capture detector (ECD). Calibration standards, quality control samples, and sample extracts are injected into the GC which is temperature and flow programmed to separate the compounds of interest. Identification of target analytes is accomplished by confirming a target hit on two dissimilar columns using Retention time and pattern recognition. Concentrations are calculated from the ECD response using internal standard techniques. Concentrations are determined using Average Response Factor from a multi-level calibration curve. The calibration curves are generated during initial

calibration through the analysis of standards which define the working range of the method. The integrity of the chromatography system must be maintained throughout a run sequence. The chromatographer should ensure that all aspects of the system, from injection through data generation is done in a consistent manner across each batch.

10.1.2 PEM Evaluation

10.1.2.1 Prior to initial calibration and at the start of each run a PEM must be analyzed and evaluated as mentioned above in the quality control section 9.7.4.

10.1.3 GC/ECD Instrumental Conditions

10.1.3.1 Generally a 1 to 3uL aliquot is injected into a uniliner and onto the capillary column of the gas chromatograph at the conditions listed below. Once injected the aliquot moves through the Siltek guard column and into a MXT-Y connector allowing one injection to split onto the two dissimilar columns connected to two separate ECD detectors

GC Parameter	Setting	GC Parameter	Setting
Injector Temp:	250°C	Ramp Rate 3:	5 °C / minute
Initial Oven Temp	125°C	Final Temperature 3:	260 °C
Flow:	33.6 mL/min	Final Hold Time 3:	0 minutes
Initial Hold Time:	0 minutes	Ramp Rate 4:	25 °C / minute
Ramp Rate 1:	25 °C / minute	Final Temperature 4:	280 °C
Final Temperature 1:	197 °C	Final Hold Time 4:	9 minutes
Final Hold Time 1:	2 minutes	Total runtime:	37.68 minutes
Ramp Rate 2:	1 °C / minute	Mode:	Splitless / Ramped Flow
Final Temperature 2:	210 °C	Purge:	25mL / minute – on at 1 minute
Final Hold Time 2:	0 minutes	Detector temp:	300 °C

The settings listed may vary if situation arises requiring an adjustment to the chromatography. *Injection temperature, hold time, flow rate, purge time, and injection volume can affect chromatographic resolution and detection limits.* Only a trained and experienced GC analyst has the authority to change any setting. All standards and samples must be acquired using the same set of parameters. If any parameters are changed, a new initial calibration must be analyzed and accepted before any samples can be analyzed.

10.2 Initial Calibration

10.2.1 Before analysis of sample extracts, establish a multi-point response factor calibration curve showing the linear range of the analysis for all target analytes. At least 5 levels of standards must be analyzed generally the concentrations run are 0.5, 1, 10, 20, 50, 100 and 200. See Section 8.11 for the preparation of the standard solutions for the initial calibration curve.

10.2.2 Construct an analytical sequence using the HP Enviroquant software. ICAL standards are identified in a sequence with an “I” designation followed by the instrument number, the

date, and the sequential number assignment. In the comment section beside each level of the curve add the assigned calibration standard ID. In sequence editor construct a sequence similar to the example listed below:

An example initial calibration sequence, created on 06/05, follows:

Primer1	
PEM3060501	(PEM)
Hexane	
I3060501STD.5	(0.5ng/mL standard)
I3060502STD1	(1ng/mL standard)
I3060504STD10	(10ng/mL standard)
I3060505STD20	(20ng/mL standard)
I3060506STD50	(50ng/mL standard)
I3060507STD100	(100ng/mL standard)
I3060509STD200	(200ng/mL standard)
Hexane	
CQ3060510STD50	ICV@50ng/mL Initial Calibration Verification
Hexane	
I3060511STD25	(Toxaphene @ 25 ng/mL)
I3060512STD25	(Chlordane @ 25 ng/mL)

10.2.3 When the sequence has finished running, the Enviroquant software will generate "Not Reviewed" quantitation reports. All reports must be "Quant Reviewed" before they can become part of the initial calibration acquisition method for sample analysis.

- Enter into the "Environmental Data Analysis"
- Go to "File" and under method, select the appropriate quant method and then select the first data file.
- Go into "Quant" and select "QEdit Quant Results" to process the data files. See Section 11.0 for manual integration details and processing guidelines.
- When processing, peak identification, and retention update is complete for the first standard, "Save" the changes and "Exit." Re-print the re-processed data file by "Generating Quant Report," and save the hard copy for each level of the initial calibration.
- Repeat these steps for all initial calibration standards analyzed within the sequence.
- When all levels have been processed, go into "InitCal," and select "Update Levels," and enter all levels for the initial calibration at the proper concentrations.
- After all responses are entered, "Save" the completed method and print the resulting response factor summary by selecting "Response Factors to Printer" as well as "Response Factors to Forms File".

10.2.3.1 Acceptance Criteria: 20% RSD for all target compounds. All calibration standards must be analyzed within 12 hours.

10.2.3.2 The following *corrective actions* are recommended for failing initial calibrations:

- If a curve has outliers and greater than five calibration solutions were utilized in the initial calibration, standards can be excluded from either extreme to generate a passing calibration. That is, the low-level standard or the high-level standard may be dropped to generate a five-level initial calibration. However the analyst needs to be aware that dropping the low standard can affect the RLs for the project being analyzed. An intermediate-level calibration standard must not be dropped to convert a failing initial calibration curve into a passing five-level initial calibration curve. Reduction in the number of calibration standards must also reduce the linear dynamic range used to quantify analytes in the samples. The resulting average response factor for each target analyte in the initial calibration curve will be used by the computer software to calculate actual sample concentrations. See Section 11.0 for additional calculation details.
- Perform instrument maintenance and repeat the initial calibration

The choice of corrective action must be made in consultation with the Section Supervisor, QA Manager, Project Manager, and/or the client.

10.2.3.3 Alternately, a linear regression model may be employed, provided that the correlation factor (r) is $\geq$0.99. Otherwise, construct a non-linear calibration of no more than a third order equation. Statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approach. A quadratic (second order) model requires six standards, and a third order polynomial requires seven standards. In setting model parameters, do not force the line through the origin. The COD (coefficient of determination) or r^2 must be greater than or equal to 0.99. The experienced analyst must select the regression order, which introduces the least calibration error into the quantitation.

10.2.3.4 Complete the initial calibration by filling out the *Initial Calibration Checklist*. The initial calibration, along with any corresponding continuing calibration data and sample data, is then forwarded for secondary review.

10.2.4 Initial Calibration Verification - ICV (separate source)

10.2.4.1 The analysis of separate source standard must follow the initial calibration curve. All target analytes in this verification standard are at the mid-point concentration. Note: This standard must be re-"Calculated and Generated" in the "QEdit" field, against the completed initial calibration curve method and processed, before it can be reviewed for acceptability.

After final processing, calculate the percent recovery of each target by using the following calculation:

% Recovery = (Found Amount / True Value) x 100

Acceptance Criteria: All recoveries must be +/- 20% of the true values.

10.3 Equipment Operation and Sample Processing

10.3.1 Evaluate the PEM as described in Sections 9.7.4

10.3.2 After successful analysis of the PEM, "Edit" the "Sample Table Log" to include the 50 ug/L CCV standard (C3060501, where "C" is for CCV, "3" is for instrument number 3,

followed by the date, and "01" for the first CCV analyzed this day), and acquire the CCV against the correct initial calibration method. "Save," then "Load and Run" the sequence.

10.3.3 When the sequence has finished running, the Enviroquant software will generate a "Not Reviewed" quantitation report. All reports must be "Quant Reviewed" against the acquisition method for sample analysis.

10.3.4 Samples are processed from "Not Reviewed" data files, to "Quant Reviewed" data files in a similar way the standards were previously processed. See Section 11.0 for details on sample processing. If a CCV fails the criteria outlined in Section 10.4.2, all samples since the last acceptable CCV must be re-analyzed.

10.3.5 If the on-column concentration of any compound exceeds the calibration range of 200 ug/L, the sample must be diluted, re-spiked with the appropriate amount of internal standard and re-analyzed. Assuming all samples are at a 1mL final volume, the following example dilutions would apply. Adjust the volumes accordingly for other sample final volume amounts and other desired dilutions.

1:2 dilution = 500uL of sample: 500uL Hexane and 10uL of IS
1:4 dilution = 250uL of sample: 750uL Hexane and 15uL of IS
1:5 dilution = 200uL of sample: 800uL Hexane and 16uL of IS
1:10 dilution = 100uL of sample: 900uL Hexane and 18uL of IS, etc

10.4 Continuing Calibration

10.4.1 A continuing calibration verification (CCV) standard, at the concentration of the mid-level of the initial calibration curve, must be analyzed at the beginning and end of every analytical sequence, and every 12 hours within the sequence, to confirm instrument stability, via response factor, for each calibrated pesticide.

10.4.2 Acceptance Criteria: Compare the CCV resulting concentration against the concentration of the compound in the injected solution for each calibrated pesticide, and calculate the % difference (%D). See Section 11.0 for the calculations. The %D for each calibrated pesticide must be below 20%D. Due to the nature of the dual column method, this criterion is applied to each set of column results. If multiple CCVs are analyzed within an analytical sequence, each CCV must be analyzed within 12 hours of the previous CCV, and each CCV, including the ending CCV, must meet the acceptance criteria. Note: closing CCV is not required as long as all samples are analyzed within 12h and the IS meets the acceptance criteria in section 9.7.2.1. If the CCV meets the acceptance criteria, save the hard copy for each CCV standard and include it with the *Continuing Calibration Checklist.* The retention times from the ICAL should be updated to the retention times identified in the CCV. The samples associated to that CCV should be calculated against the retention times of that CCV.

10.4.3 If the CCV does not meet the criteria for each calibrated analyte, the following *corrective actions* are recommended:

- Perform instrument maintenance and repeat the CCV. If the CCV fails again the initial calibration may need to be repeated
- If CCV is biased high and samples are non-detect the data may be usable
- If there is a CCV outlier for a target compound on one column, the analyst can make a judgment to force hits for that target in associated samples from the opposing acceptable column.
- If within 12 hours and no maintenance or adjustments have been made to the instrument the closing CCV can be re-injected once in case of mis-inject etc.

The choice of corrective action must be made in consultation with the Section Supervisor, QA Manager, Project Manager, and/or the client. The reasoning for choosing the second **option must be documented in the project narrative to the client.**

10.5 Preventive Maintenance

10.5.1 Preventive maintenance may or may not include the following: replacing system uniliner, o-ring, merlin septum, MXTY connector or clipping a length of guard column.

10.5.2 Additionally, preventive or routine maintenance for GC/ECD systems may involve baking out the ECD detector's, replacing guard, RTX-5 or CLPII columns.

11. Data Evaluation, Calculations and Reporting

11.1 Analyze extracts using the same conditions used for the analysis of the calibration standards. After sample analysis, “Not Reviewed” quantitation reports are generated by the software system. It is expected that situations will arise when the automated quantitation procedures of the chromatographic software provide inappropriate quantitations or integrations. Those situations will warrant the analyst perform manual integrations. Due to the especially low levels of the calibration curve, limitations of the software, and nature of the ECD, low level standards may require considerable manual integration. Because of this, a detailed report of both the low level standard and mid level standard will be submitted to QC for integration review and comparison.

11.2 Identification of the Pesticide compounds are based on gas chromatographic relative retention times (RRTs) and pattern recognition from the analysis of initial calibration standards. For these compounds, manual quantitations are performed, if necessary, by integrating the area of the peak. Qualitative identification for pesticides is made when a discrete peak in a sample is observed within the retention time window for a calibrated analyte on both columns. A pesticide hit can only be acceptable if confirmed by identification on the second column. If a Pesticide hit is not accurate on one of the two columns, i.e. outside of the retention window or absent, then the hit should not be quantitated. If manual integration of a candidate peak is necessary a straight-line integration to the baseline, taking into account background noise should be made. Area measurements should all be to baseline unless an unresolved complex mixture (UCM) is observed, in which case, the areas measured should skim the top of the UCM. Manual integrations may also be necessary when there are interferences or co-eluting peaks. Please refer to the manual integration SOP for further information.

Note: Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system.

11.3 Due to the nature of this method requiring confirmation on two dissimilar columns, two quantitations are acquired. Calculate the relative percent difference (RPD) between compound concentrations between both columns. If the calculated RPD is >40%, check the chromatograms to see if an overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine baseline parameters established by the data system (or operator) during peak integration.

- If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result. This approach is conservative relative to the protection of the environment. The disparity between the two columns should be flagged with a “P” on the report.

- If the high RPD is clearly the result of an interference on one of the columns, the lower result is reported with an "IP" qualifier on the report.

11.4 Evaluate all samples according to the following criteria:

- Surrogates and internal standards should meet the acceptance criteria in Table 2.
- Examine the chromatograms for evidence of saturated targets. Re-analyze the sample(s) at the appropriate dilution(s), see Section 10.3.5, as needed for reference.
- Establish daily retention time windows as the retention time of the component in the opening verification standard ± 0.05. It has been found that this window works well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results. The window listed above is used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms.
- If one of the two results for a target appears to have a biased response due to interference the analyst can force the quantitation from the column providing the more accurate value. Any target hit forced off of the compliant column due to interference on the alternate column will be flagged with an "I" on the report only if the RPD between the two columns is greater than 40%.
- In the instance that a Pesticide co-elutes with another Pesticide or other target compound on one column, the compound will be "C" flagged and reported off the unbiased conformant column (i.e.: on the RTX-5 column Heptachlor Epoxide/oxychlordane co-elute). Studies of independent targets have been conducted for reference of co-eluting pesticides and congeners.

11.5 In instances where manual integrations have been performed, the analyst must identify/acknowledge such edits or manual procedures by initialing and dating the raw quantitation report. The initialed hard copy/printed report, will display a "m" qualifier next to any modified or manually integrated compound(s) and shall be included in the raw data for secondary review. These requirements apply to all QC samples, field samples and blanks. For all samples the signed hard copy will be a detailed report submitted for secondary review. As mentioned above there are guidelines for appropriate manual integrations and analyst initials indicate that they accept the "m" flagged changes as appropriate.

11.6 Qualitative identification of multicomponent pesticides requires pattern matching between the calibration standards and the response observed in the sample on both columns. Retention time windows should be used as a gauge, however, pattern recognition for the multicomponent analytes is most important for samples with Toxaphene or chlordane positively identified on both columns, inject an analytical standard at the mid-level or at a concentration estimated to be similar to the sample amount, and re-inject the sample. Quantitation is generally performed using 3 to 5 major representative peaks.

11.6.1 Multi-component analytes are quantitated on the basis of 3 to 5 major peaks using an internal standard approach to calculate individual response factors for each peak. Separate calibration standards are necessary for each multi-component target analyte (e.g., toxaphene and chlordane). Analysts should evaluate the specific toxaphene standard carefully. Some toxaphene components, particularly the more heavily chlorinated components, are subject to dechlorination reactions. As a result, standards from different vendors may

exhibit marked differences which could lead to possible false negative results or to large differences in quantitative results. The relative peaks and number of peaks in the sample should be similar to that observed in the standard; however, degradation, weathering and interferences may cause the sample pattern to differ from that observed from the standard.

11.7 To calculate the ***Relative Standard Deviation*** (RSD) of all Pesticide targets and surrogate compounds for the initial calibration, use the formula below. See Section 10.2.3 for initial calibration acceptance criteria. Additionally, use the initial multi-point calibration to determine ***Relative Response Factors*** (RRF_Is) at each concentration level, for each Pesticide. Average the RRF_Is from the initial multi-point calibration, to generate mean RRF_Is, for quantification of each Pesticide. Follow the same calculations for each surrogate compound. The RRF_Is are based on the internal standard compounds, and are calculated using the formula below. See Table I, for the listing of target compounds, RLs, and their associated internal standards for quantification.

$$\mathbf{RSD = SD / mean\ RRF_I \times 100}$$

where:

SD = Standard deviation between the five points, for that target analyte.

$$\mathbf{RRF_I = (A_c \times C_{IS}) / (A_{IS} \times C_c)}$$

where:

A_c = Area of the characteristic ion for the standard compound to be measured.
A_{IS} = Area of the characteristic ion for the representative internal standard compound.
C_{IS} = Concentration of the representative internal standard compound (ng/mL).
C_c = Concentration of the standard compound to be measured (ng/mL).

11.8 Based on the mean RRF_Is, calculate the ***Raw Concentration*** ($\mathbf{C_a}$) for each target analyte and surrogate in the sample extracts using the following formula:

$$\mathbf{C_a = (A_a \times C_{IS}) / (A_{IS} \times RRF_I)}$$

where:

C_a = Calculated concentration of the target analyte, from quantitation report (ug/L).
A_a = Area of the target analyte.
A_{IS} = Area of the representative internal standard compound.
C_{IS} =Concentration of the internal standard compound added to each extract (ug/L).

11.9 Calculate the ***Sample Concentration*** (C) for each Pesticide by the following formula:

$$\mathbf{C = (C_a / V_s) \times FV \times DF}$$

where:

C = Concentration in the sample (ug/L liquid or ug/Kg solid).
V_s = Original volume or weight of sample extracted, corrected for % solids, if applicable. (To correct for % solids, multiply the sample weight by the % solid as expressed as a decimal. For example: 15.24g x 0.843 = 12.85, for a sample size of

15.24g at 84.3% solid).
DF = Dilution factor or fraction of the original extract to which internal standard is added.
FV = Sample Final Volume

If the response of any analyte in a sample exceeds the linear response range, as defined by the initial calibration standards in Section 10.2, dilute the extract so that the concentrations of all target compounds fall within the range of the calibration curve.

11.10 The calculation for percentage breakdown for DDT and Endrin are:

$$\%\text{Breakdown DDT} = \frac{(\text{Area DDD} + \text{Area DDE})}{(\text{Area DDD} + \text{Area DDT} + \text{Area DDE})} \times 100$$

$$\%\text{Breakdown Endrin} = \frac{(\text{Area Endrin Ketone} + \text{Area Endrin Aldehyde})}{(\text{Area Endrin} + \text{Area Endrin Ketone} + \text{Area Endrin Aldehyde})} \times 100$$

11.11 Calculate the ***Surrogate Recoveries*** (%R) relative to the internal standards by the following formula:

$$\%R = \frac{C_S \times DF}{C_{Strue}} \times 100$$

where:

C_{Strue} = Actual concentration of the surrogate in the extract (ug/L).
C_S = Calculated concentration of the surrogate, from quantitation report (ug/L).
DF = Dilution factor or fraction of the original extract

11.12 Compare results for each analyte in the ***Continuing Calibration Verification*** (CCV), to the true value by determining the percent difference.

$$\textbf{Percent Difference (\%D)} = ([C_{Ctrue} - C_C] / C_{true}) \times 100$$

where:

C_{Ctrue} = Actual concentration of the compound in the CCV (ug/L).
C_C = Calculated concentration of the compound in the CCV, from quantitation report

11.13 All results must be reported to three significant figures. All solids including soils, sediments, and sludges must be reported on a dry-weight basis. Tissue results may be reported in wet-weight, or "as received," depending upon client request.

11.14 The primary analyst does data entry, or upload of the data, into the LIMS system. The LIMS is "linked" to the instrument, so the analyst must choose the sample(s) to be reported from that instrument's analytical sequence. All associated preparation and instrumental QC samples and dilutions are also chosen. Once the data/samples have been selected and "associated" with the proper QC samples, the batched data set is sent to print.

11.15 The laboratory generates two types of data packages from the LIMS: "Commercial" for routine projects, and "Full Deliverable" or "CLP-like" for fully data validated projects. A

Commercial package consists of sample results and the associated method blank and LCS results. A Full Deliverable package includes all sample results, all preparation and instrumental QC results and the associated supporting raw data. Check the "Report Type" on the project folder to ensure all required deliverables are included. A secondary review is performed on all data.

11.16 Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP, 08-01. All records shall be stored in such a manner as to be safe and accessible for at least 10 years.

11.17 Notebooks: Laboratory notebooks are designed to accommodate the specific analysis. Instrument printouts are used to document run sequences, and each sequence printouts are filed in a three-ring notebook. If a sample requires re-analysis or re-extraction for any reason, a notation is made next to the sample entry on the sequence log. Requests for re-extraction are further documented in the "Request for Re-extraction" logbook. Once a sequence binder if full, the sequence run log is permanently bound, given page numbers and filed accordingly. Such files shall be archived so as to remain available for at least 10 years. All laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* SOP, G-009, and all record keeping and document control practices.

11.18 Electronic records: All data files from computers, attached to instruments, shall be backed up onto the proper directory on the server. The backups shall be stored so as to be accessible for 10 years. Movement of the data files to the server is the responsibility of the primary analyst. Server backup and storage is the responsibility of the IT department.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Section 9, Quality Control defines the corrective actions that must be taken in instances where QC outliers exist. If the corrective actions have been followed and the data is still unacceptable.

12.2 Table 2 outlines sample batch QC acceptance criteria. If non-compliant Pesticide results are to be reported, the Section Supervisor and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may choose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Section Supervisor performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1559 Sample Receipt and Login

SOP 1729 Document Control

SOP 1732 MDL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1797 Hazardous Waste and Disposal

SOP 2165 Extraction of Water Samples by Separatory Funnel

SOP 2166Tissue Preparation and Homogenization

SOP 2167 GPC Cleanup by HPLC

SOP 2168 Sulfur Cleanup

SOP 2261 Shaker Table Extraction

SOP 2267 Silica cleanup

SOP 2172 Microscale Solvent Extraction

16. Attachments

Table 1: Reporting Limits

Table 2: QC Acceptance Criteria

Table 3: 5-8 Level Curve Preparation for Individual Components

Table 1: Reporting Limits (RLs) for Pesticides by GC/ECD

Parameter	Solid Samples (ug/Kg)	Aqueous Samples (ug/L)
alpha-BHC	0.0333	0.0005
beta-BHC	0.0333	0.0005
gamma-BHC (Lindane)	0.0333	0.0005
delta-BHC	0.0333	0.0005
Heptachlor	0.0333	0.0005
Aldrin	0.0333	0.001
Hexachlorobenzene	0.0666	0.001
alpha-Chlordane	0.0333	0.0005
gamma-Chlordane	0.0333	0.0005
Heptachlor epoxide	0.0333	0.0005
Oxychlordane	0.0666	0.001
Cis-Nonachlor	0.0333	0.0005
Trans-Nonachlor	0.0333	0.0005
Dieldrin	0.0333	0.0005
Endrin	0.0333	0.0005
Endrin aldehyde	0.1	0.0005
Endrin ketone	0.0333	0.0005
4,4'-DDD	0.0333	0.0005
4,4'-DDE	0.0333	0.0005
4,4'-DDT	0.0333	0.0005
2,4'-DDD	0.0333	0.0005
2,4'-DDE	0.0333	0.0005
2,4'-DDT	0.0333	0.0005
Endosulfan I	0.0333	0.0005
Endosulfan II	0.0333	0.0005
Endosulfan sulfate	0.0333	0.0005
Methoxychlor	0.333	0.005
Mirex	0.0333	0.0005
Toxaphene	1.67	0.025
Chlordane	1.67	0.025

Note: The reporting limits used for "Solid" refer to the matrices of soil, sediment, tissue, and any other possible non-aqueous matrix. The table above represents an example of the range of reporting limits generally used in this analysis. The RLs for this analysis can vary based on sample matrix, amount extracted, final volume and cleanup schemes. Adjustments to these limits can be made to satisfy specific workplan or data quality objective requirements and specialty matrices.

The quantitation Internal Standard for all the targets is BZ 192.

Table 2: QC Acceptance Criteria

QC Parameter	Acceptance Criteria
Initial Calibration Curve	20% RSD for all target analytes, r is ≥0.99, the COD (coefficient of determination) or r^2 ≥0.99
Independent Check Verification	+/- 20% recovery of the true values
Continuing Calibration Verification	Analyzed every 12 hours, 20% D
Method Blank	No analyte at or above the reporting limit, "B" qualify analyte if detected
Laboratory Control Sample	40-140% (Sporadic Marginal Failure Criteria allowed)
Matrix Spike / Matrix Spike Duplicate	Same as for LCS; 50% RPD between the duplicates.
Sample / Sample Duplicate	50% RPD between the duplicates.
Surrogates	30-150%
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
SRM	40-140%
%RPD between dual column	+/- 40%, "P" qualify analyste

Table 3: 5-8 Level Curve Preparation for Individual Components

Calibration Level	Volume of Pesticide/Surrogate Stocks added*	Aliquot of High Calibration level** added	Volume of Hexane added
Level 1 – 0.5 ug/L	NA	0.025 ml	10 mL final volume
Level 2 – 1.0 ug/L	NA	0.05 ml	10 mL final volume
Level 3 – 5.0 ug/L	NA	0.25 ml	10 mL final volume
Level 4 – 10 ug/L	NA	0.5 ml	10 mL final volume
Level 5 – 20 ug/L	NA	1.0 ml	10 mL final volume
Level 6 – 50 ug/L	0.25 mL	NA	25 mL final volume
Level 7 – 100ug/L	0.2 mL	NA	10 mL final volume
Level 8 – 150ug/L	0.3 mL	NA	10 mL final volume
Level 9 – 200ug/L	0.4 mL	NA	10 mL final volume

Up to 10 levels may be analyzed. Standards chosen should bracket the linear range of the instrument. A minimum of a 5-level curve must be analyzed, but 6-levels, or more, may be analyzed and evaluated depending upon client specific project detection limits. This is an example of possible curve levels, the curve used may vary based on project and instrument performance.
* Each calibration level is a combination of four stock solutions prepared at the concentration of 5000ug/L added at the indicated amount.
**High calibration level is 200ug/L.

Note: 20uL of the 2.5ug/mL chosen Internal Standard mix is added to each calibration level.

Microscale Solvent Extraction (MSE)

References: **EPA Method 3570,** Test Methods for Evaluating Solid Waste, SW-846, November 2002, Revision 0, (USEPA, Office of Solid Waste and Emergency Response, Washington, DC).

Method 3500C, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final Update III, February 2007, Revision 3, (USEPA, Office of Solid Waste and Emergency Response, Washington, DC)

Method 8081B, Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 2, February 2007.

Method 8082A, Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 1, February 2007.

Method 8270D, Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, February 2007

1. Scope and Application

Matrices: This method is applicable to solid samples (soils, sludges, tissues, sediments, and solids).

Definitions: Refer to Alpha Analytical Quality Manual.

This method is recommended for the extraction of the organic compound classes in solids. This method may be used on a project specific basis as determined by the Project Manager and the Laboratory Director. This method is appropriate when the sample size may be limited due to uncontrollable factors.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Samples are weighed on an appropriate balance (analytical or top loading), spiked with surrogate, and prepared by shake or spin extraction with an organic solvent in a sealed vessel. Sample extracts are dried and concentrated using the Kuderna-Danish (K-D) or Buchi and brought to the appropriate analytical final volume.

2.1 Method Modifications from Reference

2.1.1 Extraction time can be lengthened to one 4 hour tumble, then 1 hour tumble, then 30 minute tumble if necessary.

3. Reporting Limits

Refer to analytical SOPs for Reporting Limit information.

4. Interferences

4.1 Solvents, reagents and glassware may introduce interferences. These must be demonstrated to be free of interferences by the analysis of a method blank. See the *Reagent, Solvent and Standard Control* SOP (ID No: 1816) and *Laboratory Glassware Cleaning* SOP (ID No: 1753), for additional details.

4.2 Many interferences can be removed by sample cleanup. Other cleanup methods performed include the following: *Alumina Column Cleanup of Organic Extracts* (ID No: 2260), *Sulfur Cleanup* (ID No: 2168), *Sulfuric Acid* (ID No: 2169), and *GPC Cleanup* (ID No: 2167). Only appropriate cleanup techniques must be performed based on the suspected interference and the compounds of interest. For example, sulfuric acid cleanup is not applicable to samples requiring pesticide analysis because this rigorous cleanup will destroy the majority of pesticides.

4.3 Soapy residue may result in basic conditions on glassware and may cause degradation of the pesticides Aldrin and Heptachlor, and some organophosphorous pesticides. All glassware must be rinsed thoroughly with deionized water and solvent to remove soapy residue. See the *Laboratory Glassware Cleaning* SOP (ID No: 1753), for additional details.

4.4 Phthalate esters can be a major source of contamination if any material containing plasticizers (phthalates) comes in contact with the sample during the extraction process. Use of plastic or any material containing plasticizers (phthalates) should be avoided during extraction, cleanup, or analysis.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.2 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample collection and preservation requirements are described in the various analytical method SOPs.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

The samples must be refrigerated and maintained at 4°C ±2°C until extraction and analysis. All solid/tissue samples must be extracted within 14 days from the date of collection. Alternatively, samples may be stored frozen at -15°C±5°C for up to one year if the project specifies. This effectively arrests the hold time until samples are thawed for extraction. Sample extracts must be refrigerated and maintained at 4°C ±2°C until analysis. Sample extracts must be analyzed within 40 days from date of extraction.

7. Equipment and Supplies

7.1 Vials: 60mL, pre-cleaned, open-top screw cap with PTFE-lined silicone septum and 250ml Teflon wide mouth bottles.

7.2 Balances

7.2.1 Top-loading: Capable of weighing to 0.01g

7.2.2 Analytical: Capable of weighing to 0.0001g.

7.3 Spatulas: Stainless steel

7.4 Syringes: .100-10mL, varying volumes.

7.5 Custom Tumbler Set-up

7.6 Brady Labeling System

7.7 Kuderna-Danish (KD) Apparatus:

7.7.1 Evaporation Flask: 250 and 500mL KD flasks.

7.7.2 Concentrator Tube: 10mLor 25mL

7.7.3 3-Ball Macro Snyder Column

7.7.4 Plastic clips.

7.8 Buchi Concentration System:

7.8.1 Base unit, chiller, pump block, controller and 180mL glass vessels

7.9 Boiling Chips: Solvent rinsed, approximately 10/40 mesh (silicon carbide, or equivalent).

7.10 N-EVAP: Organomation; utilized for micro blow down.

7.11 Glass vials and Screw caps: 2, 4, 10mL volume

7.12 500 mL Erlenmeyer flask

7.13 Powder Funnels: Glass or stainless steel

7.14 Glass wool: Purified by heating to 400°C for 1 hour.

7.15 Disposable Glass Transfer Pipets.

7.16 SEVAP: Organomation; utilized for concentration

7.17 Whatman Paper Filters: Number 40, 150mm Ashless Circles

7.18 Recirculator: Set at 11°C ±3°C

7.19 Centrifuge

8. Reagents and Standards

Pesticide or reagent grade chemicals are used in all tests. All reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1 Reagent Water: All references to water in this method refer to reagent water from Alpha's DI water treatment system.

8.2 Methylene Chloride (DCM): Ultra Resi quality or equivalent.

8.3 Acetone: Ultra Resi quality or equivalent.

8.4 Hexane: Ultra Resi quality or equivalent.

8.5 Sodium Sulfate (Na_2SO_4): Granular anhydrous; purified by baking at 400ºC for 4 hours in a stainless steel cylinder. Store in closed glass containers. All references to sodium sulfate in this method refer to this prepared reagent.

8.6 Copper: Granular, 20-30 mesh

8.7 Spiking Solutions: There are various surrogate and LCS/MS spiking solutions used in the extraction steps. The preparation of these solutions is described in the analytical SOPs.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Each extraction batch contains various QC samples used to ensure the validity of the sample results. The particular QC elements performed for a given extraction batch are determined by the requirements of the determinative method. The purpose and definition of the QC samples performed are listed below.

9.1 Blank

A method blank must be prepared in sodium sulfate once per every 20 samples or per extraction batch, whichever is more frequent. If samples will be extracted for a variety of determinative analyses (*i.e.,* PAH, Pesticide and PCBs within the same extraction batch) a method blank for *each* analysis must be prepared and carried through the same extraction procedures as the samples.

9.2 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

Laboratory control samples (LCS/LCSD) must be prepared once per every 20 samples or per extraction batch, whichever is more frequent, in sodium sulfate and spiked with a solution prepared from a second source or lot number, other than the source used to verify the accuracy of the standard curve for the determinative analytical method. The LCS/LCSD contains all target compounds of interest, and is extracted along with the samples as verification of the accuracy of the entire extraction procedure. If samples will be extracted for a variety of determinative analyses (*i.e.,* PAH, Pesticide and PCBs within the same extraction batch) a LCS/LCSD for *each* analysis must be prepared and carried through the same procedures as the samples.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike

Matrix spike / matrix spike duplicate (MS/MSD) samples are performed once per 20 samples (5% frequency) per client request. If samples will be extracted for a variety of determinative analyses (*i.e.,* PAH, Pesticide and PCBs within the same extraction batch) a MS/MSD pair for *each* analysis must be prepared and carried through the same procedures as the samples. If less than 20 samples are prepared in a one-week time frame, a MS/MSD pair will be extracted at the beginning of each week.

9.6 Laboratory Duplicate

Duplicate analyses (matrix or sample duplicate) are performed once per 20 samples (5% frequency) per client request. For Organic analyses, the matrix duplicate is usually in the form of the matrix spike duplicate, see Section 9.5.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Surrogates are compounds specified by the analytical method that are added to all samples and QC samples prior to beginning the extraction process. Surrogate recoveries are calculated and serve as a sample specific quantitative check of the extraction. The various spiking solutions are prepared according to the directions found in the analytical SOPs.

9.8 Method Sequence

See Section 10.

10. Procedure

Samples are prioritized by the Organic Department Manager or Preparation Group Leader for extraction based on hold time and client due date. If tissue samples will be extracted, refer to the *Tissue Preparation and Homogenization* SOP (2166) for initial sample processing details that must be employed prior to MSE extraction.

10.1 Sample Preparation and Extraction

10.1.1 Using a solvent cleaned spatula, weigh approximately 5 or 10 grams, determined by Total Solid Percentage, of homogenized sample into a pre-labeled 60mL VOA vial. For Blank, LCS, LCSD samples use only sodium sulfate. Smaller sample weights may be used, depending upon the sample volume received. Sample weights of less than 1.0g must be weighed on the Analytical balance. See MSE Extraction Chart (Form No.: 13022) for additional details as needed.

10.1.1.1 **30g MSE Extraction**: Using a solvent cleaned spatula, weigh approximately 30 grams of homogenized sample into a pre-rinsed Teflon bottle. For Blank, LCS, LCSD samples use only sodium sulfate.

10.1.2 Spike all samples with the appropriate volume of surrogate solution, and spike all QC samples with the appropriate MS and/or LCS solution. See MSE Extraction Chart (Form No.: 13022) for specific spiking details. Add 30mL of DCM to each sample depending on analysis. See MSE Extraction Chart (Form No.: 13022) for more information on different analyses. Add sodium sulfate to dry the sample. For sediment/soil samples, add approximately 2-5g of activated copper with the extraction solvent. If the sample turns the copper black add more copper until is the sample no longer turns black. This copper addition will aid in the reduction of sulfur in the sediment samples. For the preparation, storage, and expiration of activated copper, see the Sulfur Cleanup SOP (2168)

10.1.2.1 **30g MSE Extraction**: Spike all samples with the appropriate volume of surrogate solution and spike all QC samples with the appropriate MS and/or LCS solution. Add 100mls of methylene chloride (DCM) to each sample followed by sodium sulfate to dry the sample. For sediment samples add approximately 2-5 g of activated copper with DCM. Vigorously shake sample until the slurry is free flowing. Breakup any chucks with a spatula if necessary.

10.1.3 Spin the extraction vessels for at least 4 hours end over end on the automated tumbler, and then allow them to settle.

10.1.4 Decant the extract through a powder funnel into a pre-labeled KD, Buchi tube or a 500mL Erlenmeyer flask, and cap. Centrifuge the VOA vials if there is a suspension and the extraction solvent cannot be decanted. If sample material is fine use a Whatman # 40 paper filter in the funnel instead of glass wool and sodium sulfate.

10.1.5 Add 30mLs of Methylene Chloride to the samples depending on analysis. The volume may vary, however the samples must be completely covered with Methylene Chloride.

10.1.5.1 **30g MSE Extraction**: Add another 100mLs of Methylene Chloride to the samples. Make sure all of the sample is covered with DCM.

10.1.6 Tumble the samples on the automated tumbler for 1 hour, and then let the samples and Methylene Chloride settle. Alternatively, samples can be shaken by hand for 2 minutes depending on analysis.

10.1.7 Decant the sample extract into the pre-labeled collection vessel, adding this extract directly into the extract from 10.1.4 and cap. Centrifuge the VOA vials if there is a suspension and the Methylene Chloride cannot be decanted.

10.1.8 Repeat steps 10.1.5 thru 10.1.7, this time tumbling the samples for 30 minutes or hand shaking again for 2 minutes depending on analysis.

10.1.9 Samples are now ready for concentration by KD or Buchi. Samples that are not concentrated immediately are stored in a refrigerator.

10.2 Initial Concentration: KD Technique

10.2.1 If the sample was collected in a 500mL Erlenmeyer flask then you will need to assemble the Kuderna-Danish (KD) apparatus (Section 7.7) by attaching a 10mL or 25mL KD tube to a 250 or 500mL KD flask. Rinse the apparatus completely.

10.2.2 Transfer the sample extract from the Erlenmeyer flask into the 250 or 500-mL K-D flask with the 10mL or 25mL concentrator tube clipped to the bottom. Once the transfer is complete, rinse the Erlenmeyer flask with aprox 5ml of DCM and transfer that rinse into the KD.

10.2.3 Place the K-D flask on the SEVAP with 1-2 boiling chips in the bottom of the tube and the macro Snyder column on top. The bath temperature should be 80 $\pm$ 5°C. Macro-concentrate the sample to approximately 4mL. This will take approximately 35-40 minutes. If the sample requires solvent exchange, add 25mL of the exchange solvent to the KD when it reaches approximately 5mLs. Move samples to an SEVAP bath set at 95 °C and add another 25ml of the exchange solvent to the KD when it reaches approximately 5mLs. Blow samples down to approximately 4mls or to the final volume required by method. At the proper rate of distillation, the balls of the column will chatter, but the column should not flood with condensed solvent. See Table I, Section 16 for solvent exchange information.

10.2.4 If the extract appears extremely viscous and reduces in volume very slowly, a final volume of greater than 10mL may be used to ensure that there is no loss of surrogates or the compounds of interest. See the Section Supervisor or Laboratory Director for additional guidance on troublesome matrices. See Organic Prep Lab Final Volumes (Form No.:102-20) for guidance on sample final volumes.

10.2.5 Remove the sample from the bath and allow it to settle and cool for approximately 10 minutes. Remove the clip form the KD and then dry the outside of the KD using a paper tissue. Remove the KD flask from the concentrator tube..

10.2.6 Move the cooled sample, still in the concentrator tube, to the N-EVAP unit for micro-concentration, and bring the extract to the final volume required before cleanup. The extract must be concentrated under a *gentle* steady stream of nitrogen. The solvent level of the sample must be positioned to prevent water from condensing into the sample (*i.e.,* the solvent level should be below the level of the water in the NEVAP). If sample has been solvent exchanged place cooled samples on the 67°C N-EVAP to finish the solvent exchange. Re-concentrate to the final volume according to the Organic Prep Lab Final Volumes (Form No.:102-20).

10.2.7 If the extract appears extremely viscous and reduces in volume very slowly then a final volume of 5-10 mL should be used to ensure that there is no loss of surrogates or the compounds of interest. Note: Micro-concentration may not be needed depending upon the determinative analytical method. Client specifications may be different than this SOP. Always see the Section Supervisor or Laboratory Director for additional guidance when needed. See Organic Prep Lab Final Volumes (Form No.: 102-20) for guidance on sample final volumes.

10.2.8 If the sample in the concentrator tube is dark and viscous, an “auto-vial”, pre-fitted with a filtration disk, can be employed to remove particulate material. This is particularly evident in heavily contaminated petroleum samples. Reduce the extract to just less than 10mL. Remove it from the concentrator tube with an appropriate size syringe. Pass it through the auto-vial, and back into the concentrator tube. Rinse the syringe, tube and auto-vial as needed to ensure a thorough transfer. The extract may now concentrate more easily with the majority of the particulate matter removed. However, do not force the concentration as this may jeopardize the surrogate and the compounds of interest recoveries.

10.2.9 In some heavily contaminated petroleum extracts, it is possible to perform an extra step of hexane exchange to remove the asphaltene material that precipitates out in hexane. This decision must be made with the Department Supervisor approval. This may preserve the integrity of surrogates and the compounds of interest. This extract can also be auto-vialed to further remove any unwanted particulate materials. Micro-concentration may then continue.

10.2.10 Transfer the sample extract from the concentrator tube to a vial of the appropriate final volume size. The sample may now undergo any necessary cleanup that may be required prior to analysis. If the sample does not require cleanup, it may be directly transferred at the correct final volume, with copies of the sample preparation extraction log.

10.3 Alternative Concentration Technique: Buchi

10.3.1 Refer to Buchi Work Instruction ID No: 16257 for concentration instructions.

10.4 Preventive Maintenance

10.4.1 SEVAP

10.4.1.1 The SEVAP should be kept full at all times. Add reagent water as necessary.

10.4.1.2 Keep unit clean. Avoid solvent spills on or around unit. Clean periodically with a damp cloth.

11. Data Evaluation, Calculations and Reporting

Not Applicable.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence, improper preservation and observed sample headspace are noted on the nonconformance report form.

When analysis of samples indicates possible extraction problems, such as poor surrogate recoveries, poor LCS/MS/MSD recoveries, or suspected contamination in blanks or samples, re-extractions are required. Depending on the particular failure, the re-extraction may be of a specific sample or the entire extraction batch.

The analyst that determines the need for re-extraction must fill out a sample re-extract request form. This form notes the reason for the re-extraction request along with any special requirements, and the date and time that the re-extract is needed. Re-extraction request forms are maintained on file to help track the cause for re-extractions, and to be used as a tool in improving systems to minimize the need for re-extractions.

Depending on the results of the re-extraction, the first, second, or both sets of results may be reported to the client, along with a narrative report detailing the problems encountered and the resolution.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

1559 Sample Receipt & Log-In

1732 MDL/LOD/LOQ Generation

1739 IDC/DOC Generation

1797 Hazardous Waste and Sample Disposal

1816 Reagent, Solvent and Standard Control

1753 Laboratory Glassware Cleaning

2166 Tissue Preparation and Homogenization
2167 GPC Cleanup
2168 Sulfur Cleanup
2260 Alumina Column Cleanup of Organic Extracts
2169 Sulfuric Acid
Form 13022 – MSE Extraction Chart

16. Attachments

Table 1: Solvent Exchange per Method

Table 1

Solvent Exchange per Method

Method	Exchange Solvent
8081B	HEXANE
8081B-low	HEXANE
8082	HEXANE
8082-low	HEXANE
Congener	HEXANE
Homolog	HEXANE
209 PCB	HEXANE
8270D	NONE
PAH-SIM	NONE

Semivolatile Organic Compounds By Gas Chromatography / Mass Spectrometry (GC/MS)

References: **EPA 8270D**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision IV, February 2007.

EPA 8000C, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision III, March 2003.

Massachusetts DEP Bureau of Waste Site Cleanup - Quality Control Requirements and Performance Standards for the Analysis of Semivolatile Organic Compounds by GC/MS in Support of Response Actions under the Massachusetts Contingency Plan (MCP), Revision 1, July 2010.

NJDEP Site Remediation Program, Data of Known Quality Protocol, Version 1, April 2014.

1. Scope and Application

Matrices: Solid, waste and waste water, soil, sediment, tissue and ground water.

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the quantification of acidic, neutral and basic organic compounds soluble in methylene chloride. The extraction and cleanup methods that are typically used for this method are listed in Section 10.3.1. Please see the appropriate extraction SOP(s) for further information. The individual target compounds are found in Section 16, Table D, and include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, pesticides, phthalate esters, nitrosamines, aldehydes, ethers, anilines, pyridines, aromatic nitro compounds and phenols, including nitro phenols.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Aqueous samples are extracted with methylene chloride in a 2 Liter Separatory Funnel (Method 3510) at the required pH. Soil samples are generally extracted by Microscale Solvent Extraction (Method 3570), however other extraction methods can be applied. The extract is concentrated to its final volume in preparation for analysis, or may now undergo cleanups.

Analytes are introduced into the GC/MS by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC equipped with a narrow-bore capillary

column. The GC column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer (MS). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of the calibration standards. Concentrations are determined using mean relative response factors from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Analytes determined are measured in the range of 0.5 to 25ug/L for water samples, and 33.3 to 1667ug/Kg for soils, sediments, and solids. Tissue samples are measured in the range of 200 to 10000ug/Kg. Detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed. Analytes detected over these concentration ranges will be diluted and re-analyzed for accurate quantification.

4. Interferences

4.1 Phthalate esters can be a major source of contamination if any material containing plasticizers (phthalates) comes in contact with the sample during the extraction process. Use of plastic or any material containing plasticizers (phthalates) should be avoided during extraction or analysis.

4.2 The injection port of the gas chromatograph can become contaminated with high boiling compounds resulting in the loss of sensitivity of Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, Benzoic Acid, Benzaldehyde, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 4-chloroaniline, benzyl alcohol, 2,4-Dinitrotoluene and 2, 3, and 4-Nitroaniline. It may be necessary to replace the injection port liner routinely to prevent this loss of sensitivity. Clipping off approximately four inches of the column at the injection end may also increase sensitivity of the phenolic compounds. Low instrument response can be detected during the daily tuning procedure by including pentachlorophenol and benzidine in the daily tuning mix.

4.3 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

4.4 Solvents, reagents and glassware may introduce interferences. These must be demonstrated to be free of interferences by the analysis of a method blank. See the *Reagent, Solvent and Standard Control* SOP (1816) and *Laboratory Glassware Cleaning SOP* (1753), for additional details.

4.5 Many interferences can be removed by sample cleanup. The cleanup methods performed include those listed in Section 10.3.1. Only appropriate cleanup techniques must be performed based on the suspected interference and the compounds of interest.

4.6 Benzidine and pyridine may be subject to oxidative losses during solvent concentration and demonstrate poor chromatographic behavior.

4.7 Hexachlorocyclopentadiene and pyridine are subject to thermal decomposition in the injection inlet, and hexachlorocyclopentadiene is subject to chemical reaction with acetone, and photochemical decomposition.

4.8 N-nitrosodimethylamine is difficult to separate from the solvent front under the

chromatographic conditions described within.

N-nitrosodiphenylamine decomposes in the GC inlet and cannot be separated from diphenylamine.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Solid samples: A minimum of 100 grams of sample must be collected in a glass jar with a Teflon lined screw cap.

Water samples: A minimum of 2 liters of sample must be collected in amber glass bottles.

Tissue samples: A minimum of 20 grams of sample must be collected in a glass jar with a Teflon lined screw cap.

6.2 Sample Preservation

Solid samples: The samples must be refrigerated and maintained at 4±2°C until extraction and analysis. Sediment samples can be frozen at -20±5°C until extraction to extend hold time. The extracts must be refrigerated and maintained at 4±2°C until analysis.

Water samples: The samples must not be preserved except by refrigeration at 4±2°C until extraction and analysis. The extracts must be refrigerated and maintained at 4±2°C until analysis.

Tissue samples: The samples must be stored frozen at -20±5°C until extraction. The extracts must be refrigerated and maintained at 4±2°C until analysis.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Solid samples: All solid samples must be extracted within 14 days from the date of collection. Frozen sample hold times are monitored up to 14 days from the date removed from the freezer. The extracts must be refrigerated and maintained at 4±2°C until analysis. Sample extracts must be analyzed within 40 days from date of extraction.

Water samples: All water samples must be extracted within 7 days from the date of collection. Sample extracts must be analyzed within 40 days from date of extraction.

Tissue samples: Frozen sample hold times are monitored up to 14 days from the date removed from the freezer. The extracts must be refrigerated and maintained at 4±2°C until analysis. Sample extracts must be analyzed within 40 days from date of extraction.

7. Equipment and Supplies

7.1 Gas chromatograph: Programmable, heating range from 40C to 350C; split-splitless-type inlet system, (Agilent 7890 or similar); mass selective detector (Agilent 5975 or similar); automatic injector (Agilent 7683 or similar).

7.2 Chromatography Column: Fused silica capillary column, 0.25mm ID x 30m length, .25um film thickness (Zebron ZB-SemiVolatiles, Phenomenex Corporation, 5% Polysilarylene - 95% Polydimethylsiloxane , or equivalent).

7.3 Data Acquisition System: Computerized system for collecting, storing, and processing detector output (Hewlett Packard Enviroquant target software) or equivalent.

7.4 Gases: Ultra high purity BIP helium (99.9995%); Compressed Nitrogen for N-Evap.

7.5 Syringes: 10uL to 1.0mL.

7.6 Glass: Class A volumetric glass with varying volumes

7.7 GC Injection Port Liner: Phenomenex Direct Connect Top Hole

8. Reagents and Standards

Use reagent grade chemicals for all reagents. Deionized (DI) water is ASTM Type II laboratory reagent grade water.

8.1 Methylene Chloride: ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (1816) for additional details regarding solvent purity.

8.2 Acetone: ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (1816) for additional details regarding solvent purity.

8.3 Hexane: ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (1816) for additional details regarding solvent purity.

8.4 Methanol: ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (1816) for additional details regarding solvent purity.

8.5 Ether: ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (1816) for additional details regarding solvent purity.

8.6 Analytical Standards: Standards should be stored at –10°C or less, away from light when not in use. They should be discarded after 1 year unless the vendor expiration date states otherwise or if breakdown is observed. Stock standards are given a 1 year expiration from the preparation date, or the expiration of the primary vendor solution, whichever occurs first. Working standards are given a 6 months expiration from the preparation date or the expiration of the primary solution whichever occurs first.

8.6.1 Surrogates: Base/Neutral stock surrogate (5000ug/mL) – includes Nitrobenzene-d5, 2-Fluorobiphenyl, and Terphenyl-d14. Acid stock surrogate

(10000 ug/mL) - includes Phenol-d5, 2-Fluorophenol, and 2,4,6-Tribromophenol. Restek, Ultra Scientific, or equivalent.

8.6.1.1 To prepare the surrogate spiking solution mix for 8270: Add 4.0mL of the base neutral mix (5000 ug/mL) and 2.0mL of the acid surrogate mix (10000 ug/mL) to a 250mL volumetric flask. Dilute to volume with Acetone to provide an 80 ug/mL surrogate solution.

8.6.2 Stock Calibration Mix Standards: Pre-made, commercially available solutions.

- PAH Mix - ECS-A-032 (2000ug/mL)
- EPA 8270 Add-ons - ECS-A-031 (2000ug/mL)
- EPA 625 B/N Mix#1 - ECS-A-030 (2000ug/mL)
- Benzidines Mix - ECS-A-007 (2000ug/mL)
- Phenols Mix - ECS-A-006 (2000ug/mL)
- Custom Semivolatiles Mix (containing acetophenone, atrazine, benzaldehyde, biphenyl, and caprolactam) – Restek – cat # 57685 (2000ug/mL)
- PAH Two Component Mixture – C.I.L. (2000uf/mL)
- Custom Four Component Mixture - N.S.I Q-7610-O (2000ug/ml)

The surrogate stock mixes noted in sections 8.6.1 are also added to the stock standard mixture. Due to the highly reactive nature of Benzidine and Benzaldehyde and their tendency to degrade when mixed with all the other target analytes found in this method, two separate ICAL solutions are prepared.

8.6.2.1 Preparation of the first stock standard (100ug/mL) to ***10mL*** stock solution in methylene chloride is as follows:

8.6.2.1.1 EPA 8270 Add-ons (2000ug/mL): 0.5mL

8.6.2.1.2 EPA 625 B/N Mix #1 (2000ug/mL): 0.5mL

8.6.2.1.3 Phenols Mix (2000ug/mL): 0.5mL

8.6.2.1.4 PAH Mix (2000ug/mL): 0.5mL

8.6.2.1.5 Acid Surrogate (10000ug/mL): 100uL

8.6.2.1.6 B/N Surrogate (5000ug/mL): 200uL

8.6.2.1.7 Cust. Field Surrogate Mix (2000ug/mL): 0.5mL

8.6.2.1.8 Custom Four Component Mixture (2000ug/ml): 0.5ml

8.6.2.2 Preparation of the second stock standard (100ug/mL) to 10mL in methylene chloride is as follows:

8.6.2.2.1 Benzidines Mix (2000ug/mL): 0.5mL

8.6.2.2.2 Custom Semivolatiles Mix (2000ug/mL): 0.5mL

8.6.2.3 Preparation of calibration levels using the above stock solutions. Eight levels are prepared using the first stock solution. Only 7 additional levels are prepared using the second stock solution.

8.6.2.3.1 **Level 8 (50ug/mL):** 0.5mL of Calibration Stock diluted to a final volume of 1.0mL in methylene chloride.

8.6.2.3.2 **Level 7 (25ug/mL):** 2.5mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride

8.6.2.3.3 **Level 6 (15ug/mL):** 1.5mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

8.6.2.3.4 **Level 5 (10ug/mL):** 1.0mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

8.6.2.3.5 **Level 4 (5.0ug/mL):** 0.5mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

8.6.2.3.6 **Level 3 (2.0ug/mL):** 0.20mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

8.6.2.3.7 **Level 2 (1.**0ug/mL): 0.1mL of Calibration Stock diluted to a final volume of 10mL in Methylene chloride.

8.6.2.3.8 **Level 1 (0.5ug/mL):** 0.05mL of Calibration stock diluted to a final volume of 10mL in methylene chloride.

8.6.3 Initial Calibration Verification (ICV):

Initial Calibration Verification primary solutions (200ug/mL):
Benzoic Acid Solution (2000ug/mL): Restek
Benzaldehyde Solution (2000ug/mL): Restek
Benzidine solution (1000ug/mL): Restek
Custom 8270 Mix (2000ug/ml): Restek
8270 Megamix (1000ug/ml): Restek

8.6.3.1 **Initial Calibration Verification solution (10ug/mL):** 0.05mL of Custom 8270 Mix and 0.1ml of 8270 Megamix diluted to a final volume of 10mL in methylene chloride.

8.6.3.2 **Initial Calibration Verification solution** for Benzoic Acid (10ug/mL): 50 uL of Benzoic Acid Solution diluted to a final volume of 10mL in Methylene chloride.

8.6.3.3 **Initial Calibration Verification solution** for Benzidine (10ug/mL): 100uL of Benzidine Solution diluted to a final volume of 10ml in Methylene Chloride.

8.6.3.4 **Initial Calibration Verification solution** for Benzaldehyde (10ug/ml): 50uL of Benzaldehdye solution diluted to a final volume of 10ml in Methylene Chloride.

8.6.4 Continuing Calibration Verification (CCV) solution (10ug/mL):
1.0mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride. This solution is also used as the Level 5 of the ICAL.

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8.6.5 **LCS1/Matrix Spiking Solution (100/500ug/mL):** Prepared with the same standards and volumes used to formulate the first ICAL stock as seen in section

8.6.2.1., minus the 3 surrogate solutions, with additional Benzoic Acid. Brought to a final volume of 10ml in Methanol. Formulation Below.

8.6.5.1 EPA 8270 Add-ons (2000ug/mL): 0.5mL

8.6.5.2 EPA 625 B/N Mix #1 (2000ug/mL): 0.5mL

8.6.5.2.1 Phenols Mix (2000ug/mL): 0.5mL

8.6.5.2.2 PAH Mix (2000ug/mL): 0.5mL

8.6.5.2.3 Custom Four Component Mixture (2000ug/ml): 0.5ml

8.6.5.2.4 Benzoic Acid Solution (Restek 2000ug/ml): 2.0ml

8.6.6 LCS2/Matrix Spiking Solution (100ug/mL):: Prepared by diluting 0.5ml of the Restek Custom Semivolatiles Mix Cat# 57685(2000ug/ml) to 10mL in Methanol.

8.6.7 LCS3/Matrix Spiking Solution (100/250ug/mL): Prepared by diluting 0.5ml of Benzidines Mix (ECS-A-007 - 2000ug/mL), and 1.5ml of Benzidine Solution (Restek 31441 1000ug/ml) to 10mL in Methanol.

8.6.8 Internal Standard Primary Standard: Six component standard commercially available from Restek – PN 31206-510. 2000ug/mL solution containing 1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, and Perylene-d12.

8.6.8.1 8270 IS working solution (250ug/mL): 3.125mL of Internal Standard Primary Standard diluted to a final volume of 25mL in methylene chloride. 20uL of the solution is spiked into 1mL sample and standard vials. The final concentration on column is 5.0ug/mL.

8.6.9 DFTPP Primary Standard: Four component standard commercially available from UltraScientific – PN GCM-150. 1000ug/mL solution containing Decafluorotriphenylphosphine, Benzidine, Pentachlorophenol, and 4,4'-DDT.

8.6.9.1 8270 GC/MS Tuning Solution (50ug/mL): 1.25mL of DFTPP Primary Standard diluted to a final volume of 25mL in methylene chloride.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 Method Blank: A method blank must be prepared once per every 20 samples or per extraction batch, whichever is more frequent.

Compounds of interest must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: Extraction of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. The results are qualified with a "B" flag for any associated sample concentrations that are less then 10x the blank concentration for the analyte. For New Jersey regulatory work, all target analytes must be < RL except phthalates which must be < 5X the RL. Sample results are qualified with a "B" flag for analytes observed in the blank > RL and phthalates observed in the blank > 5X RL. Exceptions may be made with if the samples associated with the out of control method blank are non-detect for the compound of interest, or if sample concentrations are greater than 10x the blank levels. In such cases when the sample results are accepted, the client is notified in a project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

Laboratory control sample (LCS) and laboratory control duplicate (LCSD) must be prepared once per every 20 samples or per extraction batch, whichever is more frequent. The LCS/D may contain all target compounds of interest, or a smaller subset may be analyzed, and is extracted along with the samples as verification of the accuracy of the entire extraction procedure.

Project specific requirements, QAPPs, and/or other DQOs may supersede general limits and QC requirements.

Corrective Action: Base/Neutral compounds should be recovered at 40%-140% and Acid compounds at 30%-130%. If ≤10% of the total compounds are outside of these limits, and the recovery is at least 10% for these compounds, re-extraction is not required. If the LCS recovery is still out of control, re-extract and re-analyze the LCS and all associated samples. Samples cannot be reported until an acceptable LCS is obtained. Exceptions may be made in some cases for documented difficult/poor performing compounds such as Benzidine, Benzoic Acid, 4-Nitrophenol, Aniline, Pentachlorophenol, 2,4-Dinitrophenol, 2,4-Dimethylphenol, 4,6-Dinitro-2-methylphenol, Benzaldehyde, Caprolactam and Pyridine. An explanation of any out of control LCS recovery must be included in the project narrative to the client.

9.3 Initial Calibration Verification (ICV)

The initial calibration for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing a second source calibration standard (Section 8.6.3).

Calculate the percent recovery for each analyte. If the percent recovery for each analyte is within 70%-130% of their true value, then the calibration is assumed to be valid. If any compounds do not meet this criterion, attempts should be made to locate and correct the source of the problem. 10% of all analytes are allowed to be outside of 70%-130%, but must be within 60%-140%. The ICV may be reanalyzed once. If the standard failure continues, corrective action must be taken prior to sample analysis.

A new initial calibration must be performed and acceptable ICV results obtained prior to any sample analysis.

9.4 Continuing Calibration Verification (CCV)

On a daily basis after the DFTPP has passed, a mid-level (10ug/mL) continuing calibration standard which contains all of the analytes of interest is analyzed.

Refer to Section 10.4 for further information.

9.5 Matrix Spike/ Matrix Spike Duplicate (MS/MSD)

Matrix spike / matrix spike duplicate (MS/MSD) samples will be performed per batch of 20 samples of the same matrix processed together in those cases where the client does not specify omitting the MS/MSD for the analyses of their samples.

See Section 12 for MS/MSD recovery limits and %RPD limits. Calculate the %RPD as described in Section 9.6.

Corrective Action: Repeat analysis or check to see that an analytical error has occurred. If the % recovery and/or RPD still exceeds the control limits, and the LCS/D is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by MS/MSD recoveries and/or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses are performed upon client and/or QAPP request. *For Organic analyses, the matrix duplicate is usually in the form of the matrix spike duplicate, see Section 9.5.*

Acceptable relative percent differences (RPD) of duplicates is listed in Section12. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:
R1 = sample Replicate #1
R2 = sample Replicate #2

Corrective Action: Repeat analysis if an analytical error is suspected. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported organic results as evidenced by the matrix duplicate % RPD outlier.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogate Spikes: Surrogate spikes must be added to QC and field samples to evaluate the extraction method performance.

See Section 12 for surrogate recovery acceptance criteria.

Corrective Action: Up to one surrogate can be out in each fraction (Acid and Base/Neutral) but not less than 10% recovery, before any corrective action is necessary. Analysis must be repeated once if an analytical error is suspected. If the percent recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm the sample matrix. If *obvious* matrix interferences are noted, consultation with the Semivolatiles Department Manager, Laboratory Director or QA Officer may be necessary to confirm the

need for sample re-extraction. If no re-extraction occurs, the surrogate results and reasons for the no re-extract decision must be discussed in the project narrative to the client.

9.7.2 Internal Standards: Internal standards must be added to all sample extracts, QC samples and standards for quantitation purposes.

The internal standards in the samples should remain at constant area counts with respect to the continuing calibration analyzed at the beginning of the run. Sample IS areas must be 50% to 200% of the Internal Standards in the Continuing Calibration. Additionally, the RRT of the detected sample compounds must be within 0.06RRT units of the retention times of the associated continuing calibration standard.

Corrective Action: Analysis must be repeated once unless there are obvious sample matrix interferences, e.g. If the sample extract was very colored and viscous, or there are obvious chromatographic interferences. If *obvious* matrix interferences are noted, consultation with the Semivolatiles Department Manager, Laboratory Director or QA Officer may be necessary to confirm the need for sample re-analysis.

9.8 Method Sequence

- Tune
- CCV
- Method Blank
- LCS
- LCSD
- Samples

10. Procedure

10.1 Equipment Set-up

10.1.1 The **basic GC parameters** are as follows:

Injection Port Temp: 260°C
Oven Equib Time: 0.50 min
Oven Max: 350°C
Oven: On
Cryo: Off
Ambient: 45°C
Cryo Blast: Off

Initial Temp.: 40°C
Initial Time: 1.00 min

Level	Rate (°C /min)	Final Temp. (°C)	Final Time (min)
1	25.00	200	0
2	9.00	270	0
3	12.00	320	4

Final Time: 23.344

Please note that this may differ slightly from one instrument to another.

The **basic injection port parameters** are as follows:
Pulsed Splitless “Split” mode
Equilibrium Time: 0.50 minute
Temp: 260°C
Pressure: 9.2752 psi
Total Flow: 34.2mL/min
Split: NA
Split Flow: NA
Septum Purge Flow: 3mL/min
Injection Pulse Pressure: 30psi Until 0.2min
Purge Flow to Split Vent: 30mL/min at 0.6 min
Injection volume: 1uL

This may differ slightly from one instrument to another.

10.1.2 **Column conditions** parameters are as follows:

Constant Pressure: 9.1473 psi
Column flow: 1.2mL/min
Linear Velocity: 39.723 cm/sec

This may differ slightly from one instrument to another.

10.1.3 **Mass Spectrometer** Scan Parameters
Scan from mass 35.0 amu to 450 amu

10.1.4 Tuning

Tune acceptance must be verified at the beginning of every 12 hour analytical sequence. The DFTPP may be combined with the calibration verification standard as long as both tuning and calibration acceptance criteria are met.

Before the analytical standards are analyzed the mass spectrometer must be adjusted to meet the proper ion criteria for DFTPP. This is demonstrated by injecting into the GC/MS system 1 uL of a 50 ug/mL DFTPP solution.

Note: Within the Enviroquant software, Autofind is used first to evaluate Tune. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or any other discrete peak that does not coelute with DFTPP.

Average or single scan may be used, if Autofind does not pass, but caution should be used in tuning on a single scan too close to the front or back of the DFTPP peak. If the system will not tune under ideal conditions, injection port maintenance, retuning, and/or a source clean should be performed.

The response of Benzidine and Pentachlorophenol, when monitored on a daily basis, will indicate the efficiency of the chromatography system. Benzidine and Pentachlorophenol should be present at their normal responses and no peak tailing should be visible. The tailing factor of Benzidine and Pentachlorophenol must not exceed 2. DDT breakdown must be <20%. Although moderate tailing may indicate maintenance on the instrument will be required soon, these additional compounds in the DFTPP standard are included as an aid in instrument performance evaluation.

The following DFTPP mass intensity criteria should be used:

DFTPP KEY MASSES AND ABUNDANCE CRITERIA

Mass	m/z Abundance criteria
51	10-80% of Base Peak.
68	Less than 2% of mass 69.
70	Less than 2% of mass 69.
127	10-80% of Base Peak.
197	Less than 2%of mass 198.
198	Base peak, or > 50% of Mass 442.
199	5-9% of mass 198.
275	10-60% of Base Peak.
365	Greater than 1% of mass 198.
441	Present but less than 24% of mass 442.
442	Base Peak, or > 50% of mass 198.
443	15-24% of mass 442.

10.2 Initial Calibration

10.2.1 After the DFTPP passes criteria, a set of multi-level calibration standards listed in Section 8.6.2.3 are analyzed, from low concentration to high. A minimum of five calibration levels are analyzed. The calibration standards are stored in the standards freezer. When the Initial Calibration is typed into a sequence, the standard ID for each of the levels must be noted in the "comments" of the sequence. (Refer to the Reagent Solvent and Standard Control SOP (1816) for further information regarding standard labeling convention.)

10.2.2 Once the standards have been analyzed, they are reduced by the search software of the Enviroquant data system. Once all the components are identified, a linear curve is calculated for the components. The criteria for evaluation are as follows:

The average response factor and the response factor of the lowest calibration standard of each target analyte should be checked against the recommended minimum response factor criteria found in Table C. This information may help to quickly identify poor responding compounds and the source of failure for initial calibrations. The %RSD for each target analyte must not exceed 20%. If the %RSD for any target exceeds 20%, then the linear or quadratic calibration models must be employed. A linear fit is acceptable if the correlation coefficient is greater than 0.99. A minimum of 5 levels are required for a linear calibration model and a minimum of 6 levels are required for a quadratic calibration model. When calculating calibration curves using the linear regression model, a quantitiation check must be performed on the lowest calibration point, or the point that corresponds to that compound's established reporting limit. This recalculated concentration should be within +/-30% of the true value. If this criterion is not met then corrective action must take place and a new initial calibration must be performed prior to sample analysis.

10.2.3 Once the calibration curve is accepted, the Initial Calibration Verification (ICV) must be analyzed.

Reference standards from a separate source or different lot are analyzed after every initial calibration for evaluation of the calibration standard solutions. The response factors for all compounds should be evaluated against the minimum RF listed in Table C. The percent difference should not be greater than +/-30%D of the true value for all target compounds. If any compounds do not meet this criterion, attempts should be made to locate and correct the source of the problem. 10% of all analytes are allowed to be outside of 70%-130%, but must be within 60%-140%. The ICV may be reanalyzed once. If the standard failure continues, corrective action must be taken prior to sample analysis.

A new initial calibration must be performed and acceptable ICV results obtained prior to any sample analysis.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Extraction and Cleanup

Samples for 8270 are generally extracted using one of the following SOPs:

- *Shaker Table Extraction* (2261)
- *Separatory Funnel Extraction* 3510C (2165)
- *Microscale Solvent Extraction* 3570 (2172)

Samples for 8270 may be cleaned up before transfer to the instrument laboratory using one of the following SOPs:

- *Gel Permeation Column Cleanup* SOP (2167)
- *Silica Gel Clean-up* SOP (2170) – limited target compounds only.

10.3.2 The preparation lab staff will transfer the samples to the instrument laboratory. The samples are generally brought to a 1.0 to 5.0mL final volume; 1.0mL is transferred and any remaining sample is put into archive. One aliquot of each sample is then logged into the Internal Chain of Custody book and placed in the sample extract holding refrigerator located in the instrument laboratory.

10.3.3 All samples and standards are spiked with the six Internal Standards (IS) compounds before analysis, Section 8.6.8.1. The internal standard is intended to be used for both quantitation and the establishment of relative retention times. Samples at a volume of 1.0mL (including the QC samples of a method blank and LCS) are spiked with 20uL of internal standard for a concentration of 5ug/mL. The samples are shaken briefly after the internal standard is added to ensure mixing. If a different volume of sample is transferred the volume of internal standard added is adjusted accordingly.

10.3.4 The analyst may decide to screen any samples to determine is an upfront dilution is needed. An analyst might decide a dilution is needed prior to analysis for high target analyte concentration or to protect the instrument from of matrix related interferences.

10.3.5 After the samples have been analyzed, the data files from the Instrument are transferred to the server. The samples are quantitated versus the proper method. The QCPRN1.MAC macro creates a form with which to easily check internal standard and surrogate criteria. The istdrpt macro may, also, be utilized to check the internal standard and surrogate criteria. The following should be reviewed initially:

10.3.5.1 Are all the surrogates within QC criteria? Please see Section 12 for surrogate information.

10.3.5.2 Are all the internal standards of the samples within 50-200% of the daily CCV standard? If not, the samples should be checked for matrix interferences that may be causing these issues. The IS peaks should also be evaluated for peak splitting or incorrect integration by the software. A sample may not need to be reanalyzed if it can be determined (with guidance from a supervisor) that the QC is exceeded due to matrix interference.

10.3.5.3 Are all target analytes within calibration range? If not, the sample should be diluted and re-analyzed. If a dilution is performed after the internal standard has already been added, it will be necessary to add additional IS in order to provide a concentration of 5ug/mL. Conversely, if a sample has been over-diluted, it may need to be analyzed at a lesser dilution to detect target analytes that may have been diluted out.

10.3.5.4 Are all analyses within 12 hour tune time? If a sample is analyzed outside tune time, it will need to be re-analyzed in another tune clock.

10.3.6 The sequence should be printed out from Environmental Data Analysis, initialed and dated, placed in the standard logbook.

10.3.7 If anything in the initial review of the data indicates that there should be a re-analysis or a re-extract, the reason for re-analysis or re-extract should be noted on the sequence.

10.3.8 If a re-extract is required, the “Organics Re-Prep Request Form” google doc should be filled out and an email should be sent to the extractions lab.

10.3.9 A standard sequence (saved as S8073001;”S” for semivolatile;”8” for BNA8; 0730 for July 30, and 01 is the first sequence that day) would appear as follows:

Tune: WG#-1, -4, etc. This is the Analytical working group number generated by the Alpha LIMS that denotes the Tune.

CCV: WG#-2, -3, etc. This is the Analytical working group number generated by the Alpha LIMS that denotes the calibration verification.

10.4 Continuing Calibration Verification (CCV)

10.4.1 On a daily basis after the DFTPP has passed, a mid-level (10ug/mL) continuing calibration standard which contains all of the analytes of interest is analyzed. The criteria for acceptance are:

10.4.1.1 The %D for all targets must be ≤20%. Up to 20% of all targets are allowed to have %D > 20% with high bias as long as all associated samples are non-detect for those analytes. If any of the targets have %D > 20% with low bias corrective action must take place and all associated samples must be reanalyzed. The retention times of the internal standards must be within 0.5min of the mid-level standard of the most recent ICAL.

10.4.1.2 The area counts of the internal standards within the continuing calibration must be within 50 to 200% of the mid-level in previous ICAL.

10.5 Preventative Maintenance

All repair and non-routine maintenance records including outside service visits are maintained in the instrument maintenance logbooks.

Injection Port Maintenance: Maintenance should be done when the daily CCAL starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed.

Septum Maintenance: The septum needs to be changed approximately every two-hundred injections. Unscrew the top septum nut, remove the pierced septum, and replace with a new 11mm Thermolite green septum (Restek) or equivalent. Screw the top septum nut back on.

Column Maintenance: Maintenance should be done when the daily CCAL starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed. Generally maintenance is performed by trimming 6 cm off the front of the column. The column is then installed into the injection port liner, and the inlet nut is tightened.

11. Data Evaluation, Calculations and Reporting

11.1 Qualitative Analysis

11.1.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

11.1.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. A peak selected by the data system, based on the presence of target specific ions at a target specific retention time will be accepted as meeting this criteria.

11.1.3 The relative retention time of the sample component is within $\pm$ 0.06 RRT units of the RRT of the standard component, taking into consideration matrix effects which may cause retention time shifts.

11.1.4 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%). Outlying abundances maybe included due to the presence of non-target interference.

11.1.5 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.1.6 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by

more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e. a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

11.1.7 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes coelute, (i.e. only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum may contain extraneous ions contributed by the coeluting compound.

11.1.8 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification (TICs) will be determined by the purpose of the analyses being conducted. Data system library searches must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

11.1.8.1 For example, the RCRA permit or waste delisting requirements may require the reporting of non target analytes. Only after a visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

11.1.8.1.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.1.8.1.2 The spectral library match must be $\geq$ 85% for a tentatively identification to be made.

11.1.8.1.3 The relative intensities of the major ions should agree within $\pm$ 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%).

11.1.8.1.4 Molecular ions in the reference spectrum should be present in the sample spectrum.

11.1.8.1.5 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for background contamination or presence of co-eluting compounds.

11.1.8.1.6 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.2 Quantitative Analysis

11.2.1 Response factors and % RSD to evaluate Initial Calibration acceptability.

Calculate RF by: $$RF = \frac{area_{cmp}}{area_{is}} \times \frac{conc_{is}}{conc_{cmp}}$$

where:

area cmp = Area of the characteristic ion for the compound being measured.
area is = Area of the characteristic ion for the specific internal standard.
conc. is = Concentration of the specific internal standard.
conc. cmp = Concentration of the compound being measured.

Calculate %RSD by: $$\%RSD = \frac{SD}{\bar{x}} \times 100 \qquad SD = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \bar{x})^2}{N-1}}$$

where:

%RSD = Percent Relative Standard Deviation
x = Average of RF's
SD = Standard Deviation
x_i = Analytical results of each level in the final reporting units
N=Number of results (levels)

11.2.2 For compounds in the initial calibration in which the Average of Response Factors calibration model is used:

Calculate %Difference (%D): $$\%D = \frac{\bar{RF_i} - RF_c}{\bar{RF_i}} \times 100$$

where:

RFi - Initial Calibration average RF
RFc = Continuing Calibration RF

11.2.3 For compounds in the Initial Calibration for which the Linear Regression calibration model is used:

$$\% \text{ Drift} = \frac{\text{Calculated [ ] – Theoretical [ ]}}{\text{Theoretical [ ]}} \times 100$$

11.2.4 Results of Water Analysis- calculation as performed in report form:

$$\text{Concentration (ug/L)} = \frac{(Conc)\,(Vf)\,(DF)}{(Vi)} \times 1000$$

where:

Conc =Raw on-column concentration obtained from the quantitation report using Initial Calibration results.
Vf = Final volume of extract (mL)
Vi = Volume of sample extracted (mL)
DF = Dilution factor, for manually prepared dilutions

11.2.5 Results of Sediment/Soil and Sludge Analysis- calculation as performed in report form:

$$\text{Concentration (ug/Kg)} = \frac{(\text{Conc})\ (\text{Vf})\ (\text{DF})}{(\text{W})\ (\%\text{S})} \times 1000$$

where:

Conc =Raw on-column concentration obtained from the quantitation report using Initial Calibration results.
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
Vf = Extract final volume (mL)
W = Aliquot of sample (wet), g
%S = Sample % solid (in decimal form)

11.2.6 The calculation for percentage breakdown for DDT is:

$$\%\ \text{Breakdown DDT} = \frac{(\text{Area DDD} + \text{Area DDE})}{(\text{Area DDD} + \text{Area DDT} + \text{Area DDE})} \times 100$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All results for the organic compounds of interests are reportable without qualification if extraction and analytical holding times are met, preservation (including cooler temperatures) are met, all QC criteria are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for the necessity of re-extraction and/or re-analysis.

If non-compliant organic compound results are to be reported, the Semi volatile Organics Department Manager, the Laboratory Director, and/or the QA Officer must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

QC Parameter	Acceptance Criteria
Initial Calibration Curve	≤20%RSD or ≥0.99 linear/quadratic for all targets. If the linear or quadratic calibration model is used, recalculation of the lowest calibration standard is required to be within +/-30% of the true value. Average RFs and the lowest calibration standard should be checked against the recommended minimum RFs listed in Table C as a troubleshooting tool.
Independent Check Verification	+/- 30%D for all compounds; ICV is still accepted if up to 10% of all compounds that fail to meet +/-30%D, are within 60%-140%D
Continuing Calibration Verification	Analyzed every 12hr or at the minimum of every 20 samples; ≤ 20%D for all compounds.
Method Blank	No analyte detected at or above the reporting limit, “B” qualify analyte if detected concentration is less than 10X the concentration found in the associated sample(s)
Laboratory Control Sample / Lab Control Sample Duplicate	40%-140% (Base/Neutral compounds) and 30%-130% (Acid compounds) for all matrices; 30% RPD for soils; 20% RPD for waters; 10% allowed out provided %Recovery is at least 10%, exception made for “difficult” compounds (Section 9.2)
Matrix Spike / Matrix Spike Duplicate	Same as for LCS/D
Sample / Sample Duplicate	30% RPD for soils; 20% RPD for waters
Surrogates	Soil: 30-130% Waters: 30-130% for BN and 15-115% for Acid
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
Project specific QAPP and DQOs supersede the above limits.	

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOPs for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1732 MDL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1797 Waste Management and Disposal

SOP 1731 Manual Integration

SOP 1816 Solvent, Reagent, and Standard Control

SOP 1753 Laboratory Glassware Cleaning

16. Attachments

Table A: Surrogate Recovery Acceptance Criteria

Water: 15-115% for Acid compounds and 30-130% for Base/Neutral Compounds
Soils: 30-130%

Note: The above limits will be replaced by laboratory limits, when available. Limits are determined by sample DQO and matrix. The limits above may also vary with project specific requirements, QAPP, or DQOs.

Table B: Target Compounds and Quantitation Ions

Compound	Quantitation Ions Primary	Secondary
Aniline*	93	39, 93
Acenaphthene (CCC)	153	154, 152
Acenaphthene-d10 (IS)	164	162, 160
Acenaphthylene	152	151, 153
Acetophenone	105	77, 51
Anthracene	178	176, 179
Azobenzene *	182	77
Atrazine	200	173, 215
Benzaldehyde	77	105, 106
Benzo(a)Anthracene	228	229, 226
Benzo(a)pyrene D12 (SURR)	264	132
Benzidine*	184	183,92
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(a)pyrene	252	253, 125
Benzoic Acid*	105	122, 77
Biphenyl	154	153, 76
Benzyl Alcohol	108	79, 77
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-chloroethyl)ether	93	63, 95
Bis(2-chloroisopropyl)ether	45	77, 79
Bis(2-ethylhexyl)phthalate	149	167, 279
4-Bromophenylphenylether	248	250, 141
Butylbenzylphthalate	149	91, 206
Carbazole*	167	139,166
Caprolactam	113	55, 56
2-Chloroaniline	127	129
4-Chloroaniline	127	129
4-Chloro-3-methylphenol (CCC)	107	144, 142
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130
4-Chlorophenylphenylether	204	206, 141
Hexachlorobenzene	284	142, 249

Chrysene	228	226, 229
Chrysene-d12 (IS)	240	120, 236

Table B, continued: Target Compounds and Quantitation Ions

Compound	Quantitation Ions Primary	Secondary
Dibenz(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
Di-n-butylphthalate	149	150, 104
1,2-Dichlorobenzene	146	148, 113
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene (CCC)	146	148, 113
1,4-Dichlorobenzene-d4 (IS)	152	115
3,3'-Dichlorobenzidine*	252	254, 126
2,4-Dichlorophenol (CCC)	162	164, 98
Diethylphthalate	149	177, 150
2,4-Dimethylphenol	107	122, 121
Dimethylphthalate	163	194, 164
4,6-Dinitro-2-methylphenol	198	182,77
2,4-Dinitrophenol (SPCC)	184	63, 154
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	63, 121
Di-n-octylphthalate (CCC)	149	167, 150
Fluoranthene (CCC)	202	101, 100
Fluoranthene D10 (SURR)	212	106
Fluorene	166	165, 167
2-Fluorobiphenyl (SURR)	172	171,173
2-Fluorophenol (SURR)	112	64,91
Hexachlorobutadiene (CCC)	225	223, 227
Hexachlorocyclopentadiene (SPCC)	237	235, 272
Hexachloroethane	117	201, 199
Isophorone	82	95, 138
Indeno(1,2,3-cd)pyrene	276	138, 227
2-Methylnaphthalene	142	141
2-Methylphenol	108	107
4-Methylphenol	108	107
Naphthalene	128	129, 127
Naphthalene-d8 (IS)	136	68
2-Nitroaniline	65	92, 138
3-Nitroaniline	138	108, 92
4-Nitroaniline	138	108, 92
Nitrobenzene	77	123, 65
Nitrobenzene-d5 (SURR)	82	128, 54
2-Nitrophenol (CCC)	139	109, 65
4-Nitrophenol (SPCC)	109	139, 65
N-Nitrosodiphenylamine (CCC)	69	168, 167
N-Nitroso-di-n-propylamine (SPCC)	169	42, 101, 130
N-Nitrosodimethylamine*	74	42, 44
Pentachlorophenol (CCC)	266	264, 268
Pentachloronitrobenzene	237	214, 249
Perylene-d12 (IS)	264	260, 265

Phenanthrene	178	179, 176
Phenanthrene-d10 (IS)	188	94, 80
Phenol (CCC)	94	65, 66

Table B, continued: Target Compounds and Quantitation Ions

Compound	Quantitation Ions Primary	Secondary
Phenol-d5 (SURR)	99	42,71
Pyrene	202	200, 203
Pyridine	79	52
Terphenyl-d14 (SURR)	244	122. 212
1,2,4,5-Tetrachlorobenzene	216	214, 179
2,3,4,6-Tetrachlorophenol	232	131, 230
2,4,6-Tribromophenol (SURR)	330	332, 141
1,2,4-Trichlorobenzene	180	182, 145
2,4,5-Trichlorophenol	196	198, 97, 132, 99
2,4,6-Trichlorophenol (CCC)	196	198, 200

* Indicates compound is not part of standard reporting list.
Note: Target list will vary based on project specific requirements.

Table C: Recommended Minimum Response Factor Initial and Continuing Calibration

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.01
Phenol	0.8
Bis(2-chloroethyl)ether	0.7
2-Chlorophenol	0.8
2-Methylphenol	0.7
2,2'-Oxybis-(1-chloropropane)	0.01
Acetophenone	0.01
4-Methylphenol	0.6
N-Nitroso-di-n-propylamine	0.5
Hexachloroethane	0.3
Nitrobenzene	0.2
Isophorone	0.4
2-Nitrophenol	0.1
2,4-Dimethylphenol	0.2
Bis(2-chloroethoxy)methane	0.3
2,4-Dichlorophenol	0.2
Naphthalene	0.7
4-Chloroaniline	0.01

Hexachlorobutadiene	0.01
	0.01
Caprolactam	

Table C Continued: Recommended Minimum Response Factor Initial and Continuing Calibration

Semivolatile Compounds	Minimum Response Factor (RF)
Hexachlorocyclopentadiene	0.05
1,1'-Biphenyl	0.01
2-Chloronaphthalene	0.8
2-Nitroaniline	0.01
Dimethyl phthalate	0.01
2,6-Dinitrotoluene	0.2
Acenaphthylene	0.9
3-Nitroaniline	0.01
Acenaphthene	0.9
2,4-Dinitrophenol	0.01
4-Nitrophenol	0.01
Dibenzofuran	0.8
2,4-Dinitrotoluene	0.2
Diethyl phthalate	0.01
1,2,4,5-Tetrachlorobenzene	0.01
4-Chlorophenyl-phenyl ether	0.4
Fluorene	0.9
4-Nitroaniline	0.01
4,6-Dinitro-2-methylphenol	0.01
4-Bromophenyl-phenyl ether	0.1
N-Nitrosodiphenylamine	0.01
Hexachlorobenzene	0.1
Atrazine	0.01
Pentachlorophenol	0.05
Phenanthrene	0.7
Anthracene	0.7
Carbazole	0.01
Di-n-butyl phthalate	0.01
Fluoranthene	0.6
Pyrene	0.6
Butyl benzyl phthalate	0.01
3,3'-Dichlorobenzidine	0.01
Benzo(a)anthracene	0.8

Chrysene	0.7
Bis-(2-ethylhexyl)phthalate	0.01
Di-n-octyl phthalate	0.01

Table C Continued: Recommended Minimum Response Factor Initial and Continuing Calibration

Semivolatile Compounds	Minimum Response Factor (RF)
Benzo(b)fluoranthene	0.7
Indeno(1,2,3-cd)pyrene	0.5
Dibenz(a,h)anthracene	0.4
Benzo(g,h,i)perylene	0.5
2,3,4,6-Tetrachlorophenol	0.01

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

References: **Method 8260C**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, 2006.

Method 5035A, Closed System Purge &Trap and Extraction for Volatile Organics in Soil and Waste Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, Draft, July 2002.

Method 5030B, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December, 1996.

Method 5030C, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, May, 2003.

1. Scope and Application

Matrices: Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Definitions: Refer to Alpha Analytical Quality Manual.

The following compounds may be determined by this method:

8260C LIST OF ANALYTES		
Dichlorodifluoromethane	Carbon tetrachloride	Isopropylbenzene
Chloromethane	1,2-Dichloroethane	1,4-Dichloro-2-butane
Vinyl chloride	Benzene	1,1,2,2-Tetrachloroethane
Chloroethane	Trichloroethene	Trans-1,4-dichloro-2-butene
Bromomethane	1,2-Dichloropropane	1,2,3-Trichloropropane
Trichlorofluoromethane	Bromodichloromethane	n-Propylbenzene
Ethyl ether	Dibromomethane	Bromobenzene
Acetone	4-Methyl-2-pentanone	2-Chlorotoluene
1,1-Dichloroethene	cis-1,3-Dichloropropene	1,3,5-Trimethylbenzene
Carbon disulfide	Toluene	4-Chlorotoluene
Methylene chloride	Trans-1,3-dichloropropene	Tert-butylbenzene
Acrylonitrile	Ethyl-methacrylate	1,2,4-Trimethylbenzene
Methyl-tert-butyl ether	1,1,2-Trichloroethane	Sec-butylbenzene
Trans-1,2-dichloroethene	2-Hexanone	p-Isopropyltoluene
1,1-Dichloroethane	1,3-Dichloropropane	1,3-Dichlorobenzene
Vinyl acetate	Tetrachloroethene	1,4-Dichlorobenzene
2-Butanone	Chlorodibromomethane	n-Butylbenzene
2,2-Dichloropropane	1,2-Dibromoethane	1,2-Dichlorobenzene
Cis-1,2-dichloroethene	Chlorobenzene	1,2-Dibromo-3-chloropropane
Chloroform	1,1,1,2-Tetrachloroethane	1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl benzene	Hexachlorobutadiene
Tetrahydrofuran	p/m Xylene	Naphthalene
1,1,1-Trichloroethane	o Xylene	1,2,3-Trichlorobenzene

8260C LIST OF ANALYTES (continued)		
1,1-Dichloropropene	Styrene	Bromoform
Acrolein	2-Chloroethylvinyl ether	Ethanol
Cyclohexanone	Ethyl acetate	1,3,5-Trichlorobenzene
Iodomethane	Methyl methacrylate	Tert-amyl methyl ether
Di-isopropyl ether	n-Butanol	1,4-Dioxane
Ethyl Tert-Butyl Ether	Pentachloroethane	Isopropyl Alcohol (IPA)

There are various techniques by which these components may be introduced into the GC/MS system. Purge-and-trap, by Methods 5030C (aqueous samples) and 5035A (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. One technique is direct injection of an aqueous sample (concentration permitting).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph/mass spectrometers and in the interpretation of mass spectra and their use as a quantitative tool. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection. The analytes are introduced to a narrow-bore capillary column for analysis. The Gas Chromatograph (GC) is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the GC.

Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard, comparing sample response to the calibration standards.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our typical reporting limits.

4. Interferences

4.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be free from contamination under the conditions of the analysis. Running laboratory reagent blanks as described in Section 10.3 and 9.1

demonstrates the system is free of contamination. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system must be avoided.

4.2 Sample contamination occurs by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A trip blank or a field reagent blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.

4.2.1 Storage blanks shall be analyzed if contamination is suspect. If contamination is confirmed by positive detections in the sample storage blanks, all data from samples contained in the relative refrigerator or freezer shall be evaluated for possible contamination. If the samples contain suspected contamination, the Client Services department shall be notified in order to contact the necessary clients regarding the contamination. Samples shall be reanalyzed if so desired by the client. If suspected contamination is not confirmed by storage blanks, no further action shall be pursued concerning said blanks. It is recommended that further action be taken to determine the possible cause of suspected contamination.

4.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. Whenever a highly concentrated sample is being encountered, it should be followed by an analysis of reagent water (instrument blank) to check for potential contamination. If carry-over is suspected, then numerous instrument blanks may be required; additionally all affected samples are rerun for confirmation.. In case of severe contamination, preventive maintenance of the entire system may be required.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, standards, or solvents.

5.2 All stock solution standard preparation must be performed in the volatiles hood. Initial calibration, continuing calibration, laboratory control sample and client sample dilutions do not need to be performed in the hood.

5.3 All expired standards must be placed into the waste bucket in the lab, for future disposal. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing Methanol must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Storage, Shipping and Handling

6.1 Sample Collection and Preservation

6.1.1 Aqueous Samples

Grab samples are collected in standard 40mL amber glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two or more VOA vials should be filled per sample location. EPA Method 8260 requires that samples be acidified to eliminate the possibility of biological degradation. Unless otherwise directed for project-specific reasons, all VOA vials are delivered to the client with approximately 2 – 4 drops of 1:1 HCl added to the vial, which is sufficient to adjust the pH of the sample to < 2. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Fill the sample vial to the point of overflowing so that no headspace is contained within. Samples must be introduced into the vials gently to reduce agitation, which might drive off volatile compounds or cause loss of the HCl preservative.

Seal the bottle so that no air bubbles are in the VOA vial. If preservative has been added, shake vigorously for one minute. Invert the bottle and tap to check for air bubbles. Recollect the samples if any air bubbles are present.

Maintain the hermetic seal on the VOA vial until time of analysis.

6.1.2 Soil Samples

The recommended sampling method for soil samples is EPA 5035A. Method 5035A provides for two distinct sampling procedures, depending on the required reporting limits and suspected or known concentration levels of target analytes. These methods are referred to as the High Level and Low Level methods. Both are listed below, but depending on the samples only one of the methods may be required. If concentration levels are unknown, it is recommended that samples be collected using both procedures. The Lab will analyze the high level sample first, followed by the low level sample if the results from the high level analysis show that the sample is clean or contains analytes at low levels. The typical reporting levels of the two methods are listed in Table 1.

6.1.2.1 High Level Soil Samples

Collect sample in a standard 40mL amber glass screw-cap vial with Teflon lined silicon septa (VOA vial). The vial is provided containing 15mL of Purge and Trap Grade methanol, and is labeled and weighed prior to addition of sample. Record the weight of the vial with methanol on the vial label. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Approximately 15g of soil is added to the vial in the field, making sure that the sample is completely covered by the methanol.

Maintain the hermetic seal on the VOA vial until the time of analysis.

An additional sample of the soil must also be obtained (without methanol) to be used for the determination of soil moisture content to allow for the calculation of the dry weight results, and to calculate the methanol dilution effect. (See Sections 11.1.2.2.2 and 11.1.2.2.3)

6.1.2.2 Low Level Soil Samples

` Collect sample in a standard 40mL amber glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two samples should be taken per sample location. Vials are provided containing a magnetic stirring bar and 5 mL of either 200g/L sodium bisulfate solution or water, prepared by a certified vendor. These vials are labeled and weighed prior to addition of sample. Record the weight of the vial with the stirring bar and preservative on the vial label.

Approximately 5g of soil is added to the vial in the field, making sure that the sample is completely covered by the sodium bisulfate solution or water.

Maintain the hermetic seal on the VOA until the time of analysis.

6.2 Sample Handling and Storage

Document client specific sample handling, preservation and collection criteria in the project file. The laboratory Log-in staff documents sample temperature at the time of receipt.

Record deviations from this SOP or client specific criteria on the chain of custody form.

Record holding time exceedence, improper preservation and observed sample headspace on the nonconformance report form.

6.2.1 Aqueous Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 1 and 4 °C. Sample receiving personnel note on the sample delivery group form when samples received at the laboratory are not within the temperature criteria. If more than one vial is received for a sample the vials are stored in separate refrigerators. Storing the vials apart provides a useful check if laboratory contamination of a sample is suspected. Samples must be analyzed within 14 days of collection. Unpreserved samples requiring aromatic analysis must be analyzed within 7 days of collection.

6.2.2 High Level Soil Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria.

6.2.3 Low Level Soil Samples

Ice or refrigerate samples preserved with water or sodium bisulfate from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Samples preserved with water are to be immediately frozen after sampling. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria.

6.3 Sample Shipping

Samples requiring shipment to the laboratory are shipped in ice-packed coolers via an overnight delivery service in accordance with applicable Department of Transportation regulations.

7. Equipment and Supplies

7.1 Purge and Trap System (For Aqueous samples and High Level Soils): The purge-and-trap system consists of two separate pieces of equipment: a purging device (autosampler) (Varian Archon/8100, Tekmar Solatek, EST Centurion) coupled to the desorber (concentrator) (Tekmar Velocity or EST Encon).

7.1.1 Purge gas = Helium, analytical grade (99.999%).

7.1.2 The purging device is configured with 25 mL sample purge tubes, and the helium purge gas is introduced at the bottom of the water column as finely divided bubbles

7.1.3 The trap used in the desorber is typically a Supelco “K” trap. Different traps may be used if equivalent performance is demonstrated.

7.1.4 The desorber is capable of rapidly heating the trap to 260^{o}C. The trap is not heated above manufacturer’s specifications

7.2. Purge and Trap System (For Low Level Soil Samples): The purge and trap system consists of two separate pieces of equipment: a purging device (autosampler) coupled to the desorber (concentrator) (Varian Archon/8100, Tekmar Solatek, EST Centurion with EST Encon, Tekmar Velocity, or equivalents).

7.2.1. Purge gas = Helium, analytical grade (99.999%).

7.2.2. The autosampler purging device is a closed system, designed to accept the 40mL VOA vials. The VOA vial, containing the soil sample, water (or sodium bisulfate), and stirring bar is placed into the autosampler tray. The instrument automatically adds reagent water, internal standards, and surrogates to the unopened VOA vial. The vial is heated to 40 °C, and the helium purge gas is introduced into the aqueous portion to purge the volatile components onto the trap.

7.2.3. The trap used in the desorber is typically a Supelco “K” trap. Different traps may be used if equivalent performance is demonstrated.

7.2.4. The desorber is capable of rapidly heating the trap to 260 °C. The trap is not heated above manufacturer specifications.

7.3 Gas Chromatography/Mass Spectrometer/Data System:

7.3.1 Gas Chromatograph, Agilent 6890/7890 or equivalent: An analytical system complete with a temperature-programmable gas chromatograph with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source of the GC/MS system.

7.3.2 Typical Gas Chromatographic Columns:

7.3.2.1 Column 1: Restek 502.2, 40 meter, 0.18mm ID, or equivalent.
7.3.2.2 Column 2: Restek RTX-VMS, 30 meter, 0.25mm ID, or equivalent

7.3.3 Mass Spectrometer, Agilent 5973/5975/5978 or equivalent: Scanning from 35 to 300 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 3, when 50ng of the GC/MS tuning standard (BFB) are injected through the GC. For all SIM analysis, the mass spectrometer must also be able to acquire data in a dual acquisition mode (SIM and full scan).

7.3.4 Data System: Hewlett-Packard EnviroQuant software is used for data acquisition, and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

Thruput Target 4.12 software or Enviroquant E.02.02 (or equivalent) is used for data processing, and allows searching of any GC/MS data file for ions of a specified mass, and plotting such ion abundances versus time or scan-number.

The most recent version of the EPA/NIST Mass Spectral Library is loaded onto the Target / Enviroquant data system.

7.4 Wiretrol or Microsyringes: 10µL - 1,000µL.

7.5 Syringes: 5mL, 10mL, or 25mL, glass with Luerlock tip.

7.6 Balances: Top-loading, capable of weighing 0.1g.

7.7 Vials: 2mL, 4mL.

7.8 Disposable Pipets.

7.9 Volumetric Flasks: Class A, appropriate sizes, with ground-glass stoppers.

7.10 Eppendorf Pipets

8. Reagents and Standards

Reagent grade organic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Great care must be taken to maintain the integrity of all standard solutions. Standards in methanol are stored at –10ºC or less, in amber vials with PTFE-lined screw-caps.

8.1 Organic-free Reagent Water:

All references to water in this method refer to organic-free reagent water, which is tap water passed through activated carbon and air bubbled through.

8.2 Methanol:

Purge and Trap Grade or equivalent. Store in flammables cabinet.

8.3 Stock Solutions:

All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial with minimal headspace. The expiration date of the stock solution is either the vendor specified expiration date or 6 months from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 4.

8.4 Intermediate Standards: Intermediate standards are prepared volumetrically by diluting the appropriate stock standard(s) with methanol. Initial Calibration solutions expire 2 months from the date of preparation, or sooner if daily continuing calibration checks do not achieve the method acceptance criteria. If the Intermediate Standards are used as a second source to verify a valid Initial Calibration solution, there is no expiration date.

8.4.1 Internal Standard Solutions:

The internal standards are Fluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The intermediate IS solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of IS solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample or standard. Internal standard is added at the same concentration to all standards, samples, and QC samples.

8.4.2 Surrogate Standard Solutions:

The surrogate standards are Dibromofluoromethane, 1,2-Dichloroethane-d_4, Toluene-d_8, and 4-Bromofluorobenzene. The intermediate surrogate solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of surrogate solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample.

8.4.3 Target Compound Solutions:

The target analytes routinely reported by this method are listed in the beginning of this SOP. The intermediate target compound solutions are prepared by diluting the stock solution(s) with methanol. This set of solutions, at concentrations of 200 µg/mL, is used for preparation of the calibration standards.

8.4.4 4-Bromofluorobenzene (BFB) Tune solution:

A solution containing BFB at a concentration of 50 µg/mL is prepared by volumetrically diluting the BFB stock solution. 1 µL of this solution is direct-injected or purged into the GC/MS system to verify system performance prior to any standard or sample analysis.

8.5 Calibration Standards:

There are two types of calibration standards used for this method – initial calibration standards and calibration verification standards.

8.5.1 Initial Calibration Standards:

Initial calibration standards can be prepared at the levels listed in Table 4 (other/different levels are allowed). The Initial Calibration needs to have a minimum of 5 standards, 6 if a quadratic curve fit is used. Prepare these solutions in organic-free reagent water. The standards correspond to the range of concentrations found in typical samples and do not exceed the working range of the GC/MS system. Initial calibration should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

8.5.2 Initial Calibration Verification Standard (ICV):

The initial calibration verification standard is at the same concentration as the level 3 initial calibration standard. This standard is made from a second source than the Initial Calibration Standards.

8.5.3 Continuing Calibration Verification Standard:

The continuing calibration verification standard, or calibration check standard, should be analyzed near the action level of the project. Since most projects are focused on achieving low reporting limits, the continuing calibration verification standard is at the same concentrations as the level 3 initial calibration standard. This standard is run at the beginning of each analytical sequence, following the BFB tune standard, to verify system performance.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Blank samples must be matrix specific, i.e. methanol samples need to have methanol in the blank; sodium bisulfate samples need to have a sodium bisulfate blank analyzed; TCLP samples need a TCLP blank.

Analyze a matrix-specific blank each day prior to sample analysis to demonstrate that interferences from the analytical system are under control. The blank must contain the internal standards and surrogates.

Analyze the reagent water blank from the same source of water used for preparing the standards, QC samples and making sample dilutions. The method blank must not contain any target analytes at or above the compound reporting limits.

9.2 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

A LCS/LCSD pair is analyzed at the beginning of each analytical sequence. Since the LCS contains the same compounds at the same concentrations as the continuing calibration check standard, the same analysis is used to satisfy both QC elements. The LCS/LCSD acceptance criteria are based on in-house control limits, unless specified by project/regulation.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.5.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.4.

9.5 Matrix Spike/ Matrix Spike Duplicate

Upon Client Request, a matrix spike/matrix spike duplicate pair may be analyzed with each batch of 20 or less samples. The MS/MSD are sample aliquots spiked with the target compounds at the same concentration as the continuing calibration standard. The MS/MSD acceptance criteria are based on in-house control limits. If the MS/MSD does not meet the criteria, but the LCSD does, the failure may be attributed to sample matrix. Report the MS/MSD, including a narrative sheet for inclusion with the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Internal Standards

Area counts of the internal standard peaks in all samples and QC samples must be between 50-200% of the areas of the internal standards in the QC check standard.

If any individual percent recovery falls outside the range, that parameter has failed the acceptance criteria. For calibration standards, CCVs, LCS/LCSD or blanks the internal standard must be within the range for data to be reported to the clients. For samples, matrix spikes and duplicates: if the data is not within the range, the sample is rerun to confirm that the failure is due to sample matrix. A nonconformance report form is completed to ensure client notification and reporting if matrix effect is confirmed.

9.7.2 Surrogates

Surrogates are added to each field sample and QC sample. The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The surrogate acceptance criteria are listed in Table 2. Since the SIM analysis is acquired in dual mode, the surrogates from the full scan are used to evaluate the entire sample (SIM and full scan).

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is as follows:

- BFB
- QC Check Standard/Laboratory Control Sample/LCSD
- Method Blank
- Samples
- MS/MSD (upon Client request, may be run anytime after the Method Blank)

10. Procedure

10.1 Equipment Set-up

Typical instrument operating conditions are listed below. Alternate conditions are allowed, as long as method performance criteria can be met.

10.1.1 GC Conditions:

Temperature 1:	35°C	Carrier gas:	Helium, 99.999%
Hold Time 1:	4 minutes	Carrier mode:	Constant flow
Ramp 1:	6°C/minute	Carrier flow:	1 mL/minute
Temperature 2:	150°C		
Hold Time 2:	0 minutes		
Ramp 2:	8°C/minute		
Temperature 3:	220°C		
Final Time:	1 minute		

10.1.2 MS Conditions:

Mass scan range:	35 – 260 amu
Scan time:	0.5 minutes/scan
Source temperature:	230°C

10.1.3 Velocity Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	2 minutes
Desorb preheat:	250°C
Desorb temp:	255°C
Desorb time:	2 minutes
Bake temp:	290°C
Bake time:	10 minutes

10.1.4 Encon Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	1 minute
Desorb preheat:	245°C
Desorb temp:	255°C
Desorb time:	1 minute
Bake temp:	270°C
Bake time:	10 minutes

10.2 Initial Calibration

10.2.1 The initial calibration is performed at a minimum of five (5) concentration levels listed in Table 4, the low level of the either at or below the reporting limit. The calibration is performed using instrument conditions listed in Section 10.1.

BFB must be analyzed prior to analysis of the initial calibration standards, and must pass the criteria listed in Table 3. The mass spectrum of BFB should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of BFB.

This is done automatically with the ThruPut Target / Enviroquant software.

10.2.1.1 **Low Level/High Level Soil Curve on Archon or Centurion:** To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 50mL volumetric flask using a microsyringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 5mL syringe with standard and transfer to a 40mL VOA vial containing a magnetic stir bar. Load the vial onto Archon Autosampler.

10.2.1.2 **Aqueous/High Level Soil Curve on Solatek or Centurion:** To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 100mL volumetric flask using a microsyringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 40mL VOA vial to the top. Load the vial onto the Autosampler.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in Section 10.1. The same operating conditions are used for calibration and sample analyses. Create the analytical sequence using the HP Enviroquant data acquisition software.

Relative Response Factors: The internal standard calibration technique is used. In each calibration standard, calculate the relative response factor for each analyte and the relative standard deviation (RSD) of the response factors using the Target / Enviroquant data processing software. The response factors are calculated using the areas of the characteristic (quantitation) ion for each target analyte and internal standard. The calculations are performed automatically using the Target / Enviroquant software, using the formulae listed in Alpha's Quality Manual.

10.2.3 **Initial Calibration Criteria:** The following sections outline the method acceptance criteria for an initial calibration curve. All criteria must be met for the calibration to be deemed acceptable, and for sample analysis to proceed.

10.2.3.1 **Relative Standard Deviation Criteria:** If the RSD for each target analyte is less than or equal to 20%, then the response for this compound is considered linear over the calibration range and the mean calibration factor can be used to

quantitate sample results. If the 20% RSD criterion is not met for an analyte linear regression may be used if **r** ≥ 0.990, weighted linear with a weighting factor of 1/SD2 and r > 0.990, or quadratic fit if $\mathbf{r^2}$ ≥ 0.995. A minimum of six points is required and the low point of the calibration must be re-quantitated and recover within 70-130% to be deemed acceptable. The calibration must be repeated for any compounds that fail. If more than 10% of the compounds exceed the 20% RSD limit and do not achieve the minimum correlation coefficient for alternative curve fits, sample analysis cannot proceed.

10.2.3.2 Minimum Response Factors: Table 1 lists the suggested minimum response factors for the most common analytes. Each calibration level must be evaluated against the specified criteria. Analytes that fall below the criteria, but are greater than or equal to 0.05, are narrated for inclusion on the final report. There are certain very poor purgers (1,4-Dioxane, Acrolein, ketones, alcohols and other water soluble compounds) that should meet a 0.001 response factor. If an analyte falls below 0.05 (or 0.001 for 1,4-Dioxane, Acrolein, ketones, alcohols and other water soluble compounds), then corrective action must be taken to resolve the problem before analysis can proceed.

10.2.4 Evaluation of Retention Times: The relative retention times used for identification of target analytes are +/- 0.06 RRT (Relative Retention Time) units, based on the most recent standard run. It has been determined that these limits work well, being wide enough to eliminate false-negative results while being tight enough to eliminate false positive results. Due to the selectivity of the mass spectrometer, compound identification is more definitive than when using a less selective detector.

10.2.5 Initial Calibration Verification: After each calibration and before the analysis of samples, an ICV must be analyzed at or near the midpoint of the curve. The ICV must be prepared using a different source than the Initial Calibration and must contain all target analytes. The percent recoveries must be between 70% and 130% for target analytes except for “difficult” analytes (Table 7), which must exhibit percent recoveries between 40% and 160%. Corrective action is required if greater than 10% of all analytes are outside the prescribed criteria.

10.3 Equipment Operation and Sample Processing

The same GC, MS, and Purge and Trap conditions used for the initial calibration must be employed for sample analysis. After verification of system performance by analysis of BFB, the continuing calibration standard and method blank, samples are analyzed and processed as described below.

10.3.1 Analysis of Samples

Retrieve sample VOA vials from the sample bank refrigerator just prior to loading onto the purge and trap system. High level soil samples must be shaken for 1 – 2 minutes to extract the volatile components into the methanol. Let sample settle prior to taking methanol aliquot. Low level soil sample should be shaken briefly to ensure that the stir bar is loose, and will spin on the Archon or Centurion unit.

10.3.1.1 Low level soil samples: (Archon or Centurion)
Take the low level VOA vial and place directly into the rack of the Archon sampling unit. Surrogate and internal standards are added automatically by the Archon prior to sample purging.

10.3.1.2 Aqueous samples: (Solatek or Centurion)

Load the VOA vial directly on the sampling rack. Dilutions may be prepared volumetrically and poured into VOA vials ensuring there is no headspace left in the vial. The auto-sampler will then sample 10mL from the VOA vial.

10.3.1.3 High level soil samples: (Archon/Solatek/Centurion)

Shake for 2 minutes, ensuring the methanol has completely penetrated the soil in the vial.

10.3.1.3.1 Through liquid path

Load a maximum of 430µL or appropriate dilution of the methanol into a half-full VOA vial. Fill the VOA vial up to the top with water and cap with no headspace. Allow the auto-sampler to sample 10mL out of the VOA vial which would be equivalent to injecting 100µL of the methanol extract. Prepare dilutions accordingly.

10.3.1.3.2 Through soil path

Into a VOA vial with a stir bar added, load 4.9mL of water plus a maximum of 100 µL of methanol or appropriate dilution of methanol extract from a 5mL luerlock syringe. Cap the vial and load onto the auto-sampler.

10.3.2 Qualitative Analysis:

10.3.2.1 The qualitative identification of each compound is based on retention time and on comparison of the sample mass spectrum with the reference mass spectrum. The reference mass spectrum must be generated by the laboratory on the same GC/MS system. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.3.2.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. The Target / Enviroquant data system is configured to make this check.

10.3.2.1.2 The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the RRT of the standard component.

10.3.2.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

10.3.2.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs (i.e., m and p-xylene).

10.3.2.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

10.3.2.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

10.3.2.2 For samples containing non-target analytes, a library search will be performed at client request. Compound identification will be classified as "tentative", and the concentration will be reported as an estimate as no quantitative standards are run for these compounds.

1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
2) The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks.

10.3.3 Quantitative Analysis:

10.3.3.1 Quantitation of a target compound detected in a sample is performed automatically by the Target / Enviroquant data processing software, using the formulae found in Alpha's Quality Manual. Either the average response factor or calibration curve will be used for sample quantitation, depending on how the particular analyte was processed in the initial calibration curve.

If non-target compounds are to be reported, the quantitation is performed automatically by the Target / Enviroquant software using the total area of the compound and the nearest internal standard, and assuming a relative response factor of 1.0.

10.4 Continuing Calibration

Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

10.4.1 Prior to the analysis of samples or calibration standards, inject or purge 1 µL (50 ng) of the 4-Bromofluorobenzene standard (Section 8.4.4) into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 3 before sample analysis begins.

10.4.2 The initial calibration curve for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing the continuing calibration check standard (Section 8.5.3).

10.4.3 A method blank must be analyzed prior to any samples, typically immediately following the continuing calibration check standard, to ensure that the analytical system is free of contaminants. The method blank must not contain any target analytes at or above the required compound reporting limits.

10.4.4 The percent difference or drift for each target analyte must be less than or equal to 20% (30% for all SIM compounds). If greater than 20% of target analytes exceed the %D criteria corrective action must be taken prior to the analysis of samples. If less than or equal to 20% of compounds exceed the criteria, corrective action is not required.

10.4.5 The continuing calibration standard must also be evaluated for the suggested minimum response factor criteria, as specified in section 10.2.3.2

10.4.6 Internal Standard Retention Time:

The retention times of the internal standards in the calibration verification standard are evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.7 Internal Standard Response:

If the area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed on the analytical system. This includes replacement of GC septa and periodic rinsing or replacement of purge and trap tubes and sparge needles. The trap should be replaced every six months, or sooner if performance criteria cannot be met. Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

If system performance deteriorates, additional maintenance may be required. This includes replacement of injector ports and seals, clipping several inches off of the front end of the GC column, or in extreme cases the replacement of the GC column. Flushing or replacement of purge and trap lines may be necessary if they become contaminated or develop active sites.

Perform routine preventative maintenance as described throughout this SOP. Record all maintenance in the instrument logbook.

11. Data Evaluation, Calculations and Reporting

11.1.1 LIMS Data Corrections

Please note that the Laboratory Information Management System (LIMS) automatically adjusts soil sample results to account for the % Total Solids of the sample (as determined per Alpha SOP/07-38) and the methanol preservation dilution effect.

11.1.2 Data Calculations

11.1.2.1 Results of Aqueous Sample Analysis:

$$concentration\ (ug/L) = \frac{(Conc.)\ (Vp)\ (DF)}{(Vs)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
Vp = Volume purged, 10 mL is standard
Vs = Volume of sample purged
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".

11.1.2.2 Results of Sediment/Soil, Sludge, and Waste Analysis:

All solids including soils, sediments, and sludges must be reported on a dry-weight basis.

11.1.2.2.1 Low-Level Samples:

$$concentration\ (ug/Kg) = \frac{(Conc.)\ (Vp)\ (DF)}{(W)\ (\%S)}$$

11.1.2.2.2 High-Level Samples:

$$concentration\ (ug/Kg) = \frac{(Conc.)\ (Vp)\ (5000)\ (DF)}{(W)\ (Ve)\ (\%S)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
Ve = Extract volume, mL
Vp = Volume purged, 5 mL is standard
W = Aliquot of sample (wet), g
%S = Sample % solid
5000 = Constant representing the final volume of the methanol extraction.

11.1.2.2.3 High-Level Samples Corrected for Total Water/Solvent Mixture (V_t):

Samples that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the water/solvent mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture calculation.

$$\%\ moisture = \frac{g\ of\ sample - g\ of\ dry\ sample}{g\ of\ sample} \times 100$$

$$V_t = \frac{[mL\ of\ solvent + (\%moisture \times g\ of\ sample)]}{100} \times 1000mL/mL$$

The calculated V_t value is now added to the volume of methanol in the sample (typically 5000µL), and the corrected concentration is calculated using the equation below:

$$Corrected\ concentration\ (mg/Kg) = \frac{(Conc.)\ (V_t + methanol\ vol.)\ (Vp)\ (DF)}{(W)\ (Ve)\ (\%S)}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All batch and sample specific QC criteria outlined in section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP/14-01 Waste Management and Disposal SOP

16. Attachments

TABLE 1: 8260 REPORTING LIMITS

TABLE 2: 8260 QC ACCEPTANCE CRITERIA

TABLE 3: BFB TUNING CRITERIA

TABLE 4: STANDARD SOLUTIONS

TABLE 5: 8260C Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

TABLE 6: 8260C Quantitation Ions

Table 1
Standard Reported Detection Limits
US EPA METHOD 8260C and 5035A/8260C

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)[1]	RDL (µg/KG)[2]
Acetone [3,4,5]	0.100	5.0	10	250
Acrolein [5]		5.0	25	1250
Acrylonitrile [3,4]		5.0	5	200
Benzene [3,4,5]	0.500	0.5	1	50
Bromobenzene [3,4]		2.5	5	250
Bromochloromethane [3,4,5]		2.5	5	250
Bromodichloromethane [3,4,5]	0.200	0.5	1	50
Bromoform [3,4,5]	0.100	2.0	4	200
Bromomethane [3,4,5]	0.100	1.0	2	100
2-Butanone [3,4,5]	0.100	5.0	10	500
n-Butyl benzene [3,4]		0.5	1	50
sec-Butyl benzene [3,4]		0.5	1	50
tert-Butyl benzene [3,4]		2.5	5	250
Carbon disulfide [3,4,5]	0.100	5.0	10	500
Carbon tetrachloride [3,4,5]	0.100	0.5	1	50
Chlorobenzene [3,4,5]		0.5	1	50
Chloroethane [3,4,5]	0.100	1.0	2	100
2-Chloroethylvinyl ether [3]		10.0	20	1000
Chloroform [3,4,5]	0.200	0.75	1.5	75
Chloromethane [3,4,5]	0.100	2.5	5	250
o-Chlorotoluene [3,4]		2.5	5	250
Cyclohexane [5]	0.100	10	20	1000
Cyclohexanone		10	20	1000
p-Chlorotoluene [3,4]		2.5	5	250
Dibromochloromethane [3,4,5]	0.100	0.5	1	50
1,2-Dibromo-3-chloropropane [3,4,5]	0.050	2.5	5	250
1,2-Dibromoethane [3,4,5]	0.100	2.0	5	250
Dibromomethane [3,4]		5.0	10	500
1,2-Dichlorobenzene [3,4,5]	0.400	2.5	5	250
1,3-Dichlorobenzene [3,4,5]	0.600	2.5	5	250
1,4-Dichlorobenzene [3,4,5]	0.500	2.5	5	250
1,4-Dichlorobutane [3,4]		5.0	10	500
trans-1,4-Dichloro-2-butene [3,4]		2.5	5	250
Dichlorodifluoromethane [3,4,5]		5.0	10	500
1,1-Dichloroethane [3,4,5]	0.200	0.75	1.5	75
1,2-Dichloroethane [3,4,5]	0.100	0.5	1	50
1,1-Dichloroethene [3,4,5]	0.100	0. 5	1	50
cis-1,2-Dichloroethene [3,4,5]	0.100	0.5	1	50
trans-1,2-Dichloroethene [3,4,5]	0.100	0.75	1.5	75

Table 1 (continued)

Standard Reported Detection Limits

US EPA METHOD 8260C and 5035A/8260C

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)[1]	RDL (µg/KG) [2]
1,2-Dichloropropane [3,4,5]	0.100	1.75	3.5	175
1,3-Dichloropropane [3,4]		2.5	5	250
2,2-Dichloropropane [3,4]		2.5	5	250
1,1-Dichloropropene [3,4]		2.5	2.5	250
cis-1,3-Dichloropropene [3,4,5]	0.200	0.5	1	50
p-Diethylbenzene [4]		2.0	4	200
Diisopropyl Ether [6]		2.0	4	200
1,4-Dioxane [5] (non-SIM)		250	100	5000
trans-1,3-Dichloropropene [3,4,5]	0.200	0.5	1	50
Ethanol [7]		N/A	1000	50000
Ethyl acetate		10.0	20	1000
Ethylbenzene [3,4,5]	0.100	0.5	1	50
Ethyl ether [3,4]		2.5	5	250
4-Ethyltoluene [4]		2.0	4	200
Ethyl methacrylate [3,4]		5.0	10	500
Ethyl-Tert-Butyl-Ether [6]		2.0	4	200
Freon-113 [5]		10.0	20	1000
Hexachlorobutadiene [3,4]		0.5	5	250
2-Hexanone [3,4,5]	0.100	5.0	10	500
Iodomethane		5.0		
Isopropyl Alcohol (IPA)		25		
Isopropylbenzene [3,4,5]	0.100	0.5	1	50
p-Isopropyltoluene [3,4]		0.5	1	50
Methyl Acetate [5]	0.100	20	20	1000
Methylene chloride [3,4,5]	0.100	3.0	10	500
Methyl Cyclohexane [5]	0.100	20	4	200
Methyl Methacrylate		1.0		
4-Methyl-2-pentanone [3,4,5]	0.100	5.0	10	500
Methyl-tert-butyl-ether [3,4,5]	0.100	1.0	2	100
Naphthalene [3,4]		2.5	5	250
n-Butanol [5]		100	200	10000
n-Propylbenzene [3,4]		0.5	1	50
Pentachloroethane		2.0	N/A	N/A
Styrene [3,4,5]	0.300	1.0	2	100
Tert-Butyl Alcohol [5]		30	100	5000
Tertiary-Amyl Methyl Ether [6]		2.0	4	200
1,1,1,2-Tetrachloroethane [3,4]		0.5	1	50
1,2,4,5-Tetramethylbenzene [4]		2.0	4	200

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)(1)	RDL(µg/KG)(2)
1,1,2,2-Tetrachloroethane (3,4,5)	0.300	0.5	1	50
Tetrachloroethene (3,4,5)	0.200	0.5	1	50
Tetrahydrofuran (3)		10.0	20	1000
Toluene (3,4,5)	0.400	0.75	1	75
1,2,3-Trichlorobenzene (3,4,5)		2.5	5	250
1,2,4-Trichlorobenzene (3,4,5)	0.200	2.5	5	250
1,3,5-Trichlorobenzene (6)		2.0	5	250
1,1,1-Trichloroethane (3,4,5)	0.100	0.5	1	50
1,1,2-Trichloroethane (3,4,5)	0.100	0.75	1.5	75
Trichloroethene (3,4,5)	0.200	0.5	1	50
Trichlorofluoromethane (3,4,5)	0.100	2.5	5	250
1,2,3-Trichloropropane (3,4)		5.0	10	500
1,2,4-Trimethylbenzene (3,4)		2.5	5	250
1,3,5-Trimethylbenzene (3,4)		2.5	5	250
Vinyl acetate (3,4)		5.0	10	500
Vinyl chloride (3,4,5)	0.100	1.0	2	100
m/p-Xylenes (3,4,5)	0.100	1.0	2	100
o-Xylene (3,4,5)	0.300	1.0	2	100
1,4-Dioxane (5) SIM		3.0		
1,1,2,2-Tetrachloroethane SIM		0.1		

(1) Detection Limits are for Low-level Aqueous preserved samples.
(2) Detection Limits are for High-level Methanol preserved samples.
(3) Analyte reported by standard 8260 reporting list.
(4) Analyte reported by New York TCL reporting list.
(5) Analyte reported by New Jersey TCL reporting list.
(6) Analyte reported for New Hampshire in addition to standard 8260 reporting list.
(7) Analyte only reported for New York TCL report upon client request.

Note: Reporting Limits are based on standard 8260 reporting list, RL's may vary for New York and New Jersey reporting lists.

Table 2

QUALITY CONTROL ACCEPTANCE CRITERIA

Surrogate Spike Percent Recovery	Aqueous Limits		Soil Limits	
	Lower Control Limit	Upper Control Limit	Lower Control Limit	Upper Control Limit
1,2-Dichloroethane-d_4	70%	130%	70%	130%
4-Bromofluorobenzene	70%	130%	70%	130%
Toluene-d_8	70%	130%	70%	130%
Dibromofluoromethane	70%	130%	70%	130%

Table 3

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Table 4

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Target Compound	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
Acetone	2000	0.5	2	10	20	30	50	100	200
Acrolein	2000	0.5	2	10	20	30	50	100	200
Acrylonitrile	2000	0.5	2	10	20	30	50	100	200
Benzene	2000	0.5	2	10	20	30	50	100	200
Bromobenzene	2000	0.5	2	10	20	30	50	100	200
Bromochloromethane	2000	0.5	2	10	20	30	50	100	200
Bromodichloromethane	2000	0.5	2	10	20	30	50	100	200
Bromoform	2000	0.5	2	10	20	30	50	100	200
Bromomethane	2000	0.5	2	10	20	30	50	100	200
2-Butanone	2000	0.5	2	10	20	30	50	100	200
n-Butyl benzene	2000	0.5	2	10	20	30	50	100	200
sec-Butyl benzene	2000	0.5	2	10	20	30	50	100	200
tert-Butyl benzene	2000	0.5	2	10	20	30	50	100	200
Carbon disulfide	2000	0.5	2	10	20	30	50	100	200
Carbon tetrachloride	2000	0.5	2	10	20	30	50	100	200
Chlorobenzene	2000	0.5	2	10	20	30	50	100	200
Chloroethane	2000	0.5	2	10	20	30	50	100	200
2-Chloroethylvinyl Ether	2000	0.5	2	10	20	30	50	100	200
Chloroform	2000	0.5	2	10	20	30	50	100	200
Chloromethane	2000	0.5	2	10	20	30	50	100	200
o-Chlorotoluene	2000	0.5	2	10	20	30	50	100	200
p-Chlorotoluene	2000	0.5	2	10	20	30	50	100	200
Cyclohexane	2000	0.5	2	10	20	30	50	100	200
Cyclohexanone	2000	0.5	2	10	20	30	50	100	200
Dibromochloromethane	2000	0.5	2	10	20	30	50	100	200
1,2-Dibromo-3-chloropropane	2000	0.5	2	10	20	30	50	100	200
1,2-Dibromoethane	2000	0.5	2	10	20	30	50	100	200
Dibromomethane	2000	0.5	2	10	20	30	50	100	200
1,2-Dichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,3-Dichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,4-Dichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,4-Dichlorobutane	2000	0.5	2	10	20	30	50	100	200
trans-1,4-Dichloro-2-butene	2000	0.5	2	10	20	30	50	100	200
Dichlorodifluoromethane	2000	0.5	2	10	20	30	50	100	200
1,1-Dichloroethane	2000	0.5	2	10	20	30	50	100	200
1,2-Dichloroethane	2000	0.5	2	10	20	30	50	100	200
1,1-Dichloroethene	2000	0.5	2	10	20	30	50	100	200
cis-1,2-Dichloroethene	2000	0.5	2	10	20	30	50	100	200
trans-1,2-Dichloroethene	2000	0.5	2	10	20	30	50	100	200
1,2-Dichloropropane	2000	0.5	2	10	20	30	50	100	200
1,3-Dichloropropane	2000	0.5	2	10	20	30	50	100	200
2,2-Dichloropropane	2000	0.5	2	10	20	30	50	100	200
1,1-Dichloropropene	2000	0.5	2	10	20	30	50	100	200

Table 4 (continued)

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Target Compound	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
cis-1,3-Dichloropropene	2000	0.5	2	10	20	30	50	100	200
trans-1,3-Dichloropropene	2000	0.5	2	10	20	30	50	100	200
p-Diethylbenzene	2000	0.5	2	10	20	30	50	100	200
Diisopropyl Ether	2000	0.5	2	10	20	30	50	100	200
1,4-Dioxane (non-SIM)	10000	100	400	1000	2000	3000	4000	5000	6000
Ethanol	10000	100	200	300	500	1000	2500	5000	N/A
Ethyl Acetate	2000	0.5	2	10	20	30	50	100	200
Ethylbenzene	2000	0.5	2	10	20	30	50	100	200
Ethyl ether	2000	0.5	2	10	20	30	50	100	200
Ethyl methacrylate	2000	0.5	2	10	20	30	50	100	200
Ethyl Tert-Butyl Ether	2000	0.5	2	10	20	30	50	100	200
4-Ethyltoluene	2000	0.5	2	10	20	30	50	100	200
Freon-113	2000	0.5	2	10	20	30	50	100	200
Halothane	2000	0.5	2	10	20	30	50	100	200
Hexachlorobutadiene	2000	0.5	2	10	20	30	50	100	200
2-Hexanone	2000	0.5	2	10	20	30	50	100	200
Iodomethane	2000	0.5	2	10	20	30	50	100	200
Isopropyl Alcohol (IPA)	10000	2.5	10	50	100	150	250	500	1000
Isopropylbenzene	2000	0.5	2	10	20	30	50	100	200
p-Isopropyltoluene	2000	0.5	2	10	20	30	50	100	200
Methyl Acetate	2000	0.5	2	10	20	30	50	100	200
Methylene Chloride	2000	0.5	2	10	20	30	50	100	200
Methyl Cyclohexane	2000	0.5	2	10	20	30	50	100	200
Methyl Methacrylate	2000	0.5	2	10	20	30	50	100	200
4-Methyl-2-pentanone	2000	0.5	2	10	20	30	50	100	200
Methyl-tert-butyl-ether	2000	0.5	2	10	20	30	50	100	200
Naphthalene	2000	0.5	2	10	20	30	50	100	200
n-Butanol	5000	2.5	10	50	100	150	250	500	N/A
n-Propylbenzene	2000	0.5	2	10	20	30	50	100	200
Pentachloroethane	1000	0.5	2	10	20	30	50	100	200
Styrene	4000	1	4	20	40	60	100	200	400
Tert-Butyl alcohol	10000	2.5	10	50	100	150	250	500	1000
Tertiary-Amyl Methyl Ether	2000	0.5	2	10	20	30	50	100	200
1,1,1,2-Tetrachloroethane	2000	0.5	2	10	20	30	50	100	200
1,1,2,2-Tetrachloroethane	2000	0.5	2	10	20	30	50	100	200
Tetrachloroethene	2000	0.5	2	10	20	30	50	100	200
Tetrahydrofuran	2000	0.5	2	10	20	30	50	100	200
1,2,4,5-Tetramethylbenzene	2000	0.5	2	10	20	30	50	100	200
Toluene	2000	0.5	2	10	20	30	50	100	200
1,2,3-Trichlorobenzene	2000	0.5	2	10	20	30	50	100	200

Table 4 (continued)

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Target Compound	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
1,2,4-Trichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,3,5-Trichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,1,1-Trichloroethane	2000	0.5	2	10	20	30	50	100	200
1,1,2-Trichloroethane	2000	0.5	2	10	20	30	50	100	200
Trichloroethene	2000	0.5	2	10	20	30	50	100	200
Trichlorofluoromethane	2000	0.5	2	10	20	30	50	100	200
1,2,3-Trichloropropane	2000	0.5	2	10	20	30	50	100	200
1,2,4-Trimethylbenzene	2000	0.5	2	10	20	30	50	100	200
1,3,5-Trimethylbenzene	2000	0.5	2	10	20	30	50	100	200
Vinyl acetate	2000	0.5	2	10	20	30	50	100	200
Vinyl chloride	2000	0.5	2	10	20	30	50	100	200
m/p-Xylenes	4000	1	4	20	40	60	100	200	400
o-Xylene	4000	1	4	20	40	60	100	200	400
1,4-Dioxane (SIM)	100	0.5	2	10	20	30	50	100	200
1,1,2,2-Tetrachloroethane (SIM)		0.05	0.1	0.2	0.5	1.0	2.0	5.0	10.0

Target Compounds	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
Internal Standards									
Fluorobenzene	2500	10	10	10	10	10	10	10	10
Chlorobenzene-d5	2500	10	10	10	10	10	10	10	10
1,4-Dichlorobenzene-d4	2500	10	10	10	10	10	10	10	10
Surrogates									
Dibromofluoromethane	2500	10	10	10	10	10	10	10	10
1,2-Dichloroethane-d4	2500	10	10	10	10	10	10	10	10
Toluene-d8	2500	10	10	10	10	10	10	10	10
4-Bromofluorobenzene	2500	10	10	10	10	10	10	10	10

- For Low Level Soil analysis, the calibration levels are the same in µg/Kg units.
- For High Level Soil analysis, the calibration levels are at 50x the levels listed due to sample preparation requirements.

TABLE 5

8260C Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Fluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Dichlorodifluoromethane	Toluene-d8 (surr)	Isopropylbenzene
Chloromethane	Toluene	Bromoform
Vinyl Chloride	Ethyl Methacrylate	1,4-dichloro-2-butane
Bromomethane	Trans-1,3-dichloropropene	1,1,2,2,-tetrachloroethane
Chloroethane	1,1,2-trichloroethane	4-bromofluorobenzene (surr)
Trichlorofluoromethane	2-hexanone	1,2,3-trichloropropane
Ethyl Ether	1,3-dichloropropane	trans-1,4-dichloro-2-butene
Freon 113	Tetrachloroethene	n-propylbenzene
Acrolein	Chlorodibromomethane	Bromobenzene
Acetone	1,2-dibromoethane	4-ethyltoluene
Ethanol	Chlorobenzene	1,3,5-trimethybenzene
1,1,-dichloroethene	1,1,1,2-tetrachloroethane	2-chlorotoluene
Tert-Butyl Alcohol	Ethylbenzene	4-chorotoluene
Methyl Acetate	p/m xylene	tert-butylbenzene
Carbon Disulfide	o xylene	1,2,4-trimethylbenzene
Methylene Chloride	Styrene	sec-butylbenzene
Acrylonitrile		p-isopropyltoluene
Methyl Tert Butyl Ether		1,3-dichlorobenzene
Halothane		1,4-dichlorobenzene
Trans-1,2-dichloroethene		n-butylbenzene
Diisopropyl Ether		p-diethylbenzene
Vinyl Acetate		1,2-dichlorobenzene
1,1-dichloroethane		1,2,4,5-tetramethylbenzene
Ethyl-Tert-Butyl-Ether		1,2-dibromo-3-chloropropane
2-butanone		1,3,5-trichlorobenzene
2,2-dichloropropane		1,2,4-trichlorobenzene
Cis-1,2-dichloroethene		Hexachlorobutadiene
Chloroform		Naphthalene
Bromochloromethane		1,2,3-trichlorobenzene
Tetrahydrofuran		Cyclohexanone
Dibromofluoromethane (surr)		1,3,5-Trichlorobenzene
1,1,1-trichloroethane		Pentachloroethane
Cyclohexane		
1,1-dichloropropene		
Carbon Tetrachloride		
Tertiary-Amyl Methyl Ether		
1,2-dichloroethane-d4 (surr)		
1,2-dichloroethane		
Benzene		
Trichloroethene		
Methyl Cyclohexane		
1,2-dichloropropane		
Bromodichloromethane		
1,4-Dioxane		
Dibromomethane		
2-Chloroethylvinyl Ether		
4-methyl-2-pentanone		
Cis-1,3-dichloropropene		
Iodomethane		
Methyl methacrylate		
n-Butanol		
Ethyl acetate		
Isopropyl Alcohol (IPA)		

TABLE 6
8260C Quantitation Ions

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Dichlorodifluoromethane	85	Ethyl Methacrylate	69
Chloromethane	50	Trans-1,3-dichloropropene	75
Vinyl Chloride	62	1,1,2-trichloroethane	83
Bromomethane	94	2-hexanone	43
Chloroethane	64	1,3-dichloropropane	76
Trichlorofluoromethane	101	Tetrachloroethene	166
Ethyl Ether	74	Chlorodibromomethane	129
Freon 113	101	1,2-dibromoethane	107
Acrolein	56	Chlorobenzene	112
Acetone	43	1,1,1,2-tetrachloroethane	131
1,1,-dichloroethene	96	Ethylbenzene	91
Tert-Butyl Alcohol	59	p/m xylene	106
Methyl Acetate	43	o xylene	106
Carbon Disulfide	84	Styrene	104
Methylene Chloride	76	Isopropylbenzene	105
Acrylonitrile	53	Bromoform	173
Methyl Tert Butyl Ether	73	1,4-dichloro-2-butane	55
Halothane	117	1,1,2,2,-tetrachloroethane	83
Trans-1,2-dichloroethene	96	1,2,3-trichloropropane	75
Diisopropyl Ether	45	Trans-1,4-dichloro-2-butene	53
Vinyl Acetate	43	n-propylbenzene	91
1,1-dichloroethane	63	Bromobenzene	156
Ethyl-Tert-Butyl-Ether	59	4-ethyltoluene	105
2-butanone	43	1,3,5-trimethybenzene	105
2,2-dichloropropane	77	2-chlorotoluene	91
Cis-1,2-dichloroethene	96	4-chorotoluene	91
Chloroform	83	tert-butylbenzene	119
Bromochloromethane	128	1,2,4-trimethylbenzene	105
Tetrahydrofuran	42	sec-butylbenzene	105
1,1,1-trichloroethane	97	p-isopropyltoluene	119
Cyclohexane	56	1,3-dichlorobenzene	146
1,1-dichloropropene	75	1,4-dichlorobenzene	146
Carbon Tetrachloride	117	n-butylbenzene	91
Tertiary-Amyl Methyl Ether	73	p-diethylbenzene	119
1,2-dichloroethane	62	1,2-dichlorobenzene	146
Benzene	78	1,2,4,5-tetramethylbenzene	119
Trichloroethene	95	1,2-dibromo-3-chloropropane	75
Methyl Cyclohexane	83	1,3,5-trichlorobenzene	180
1,2-dichloropropane	63	1,2,4-trichlorobenzene	180
Bromodichloromethane	83	Hexachlorobutadiene	225
1,4-dioxane	88	Naphthalene	128
Dibromomethane	93	1,2,3-trichlorobenzene	180
2-Chloroethylvinyl Ether	63	Ethanol	45
4-methyl-2-pentanone	58	Cyclohexanone	55
Cis-1,3-dichloropropene	75	Ethyl acetate	43

TABLE 6
8260C Quantitation Ions (continued)

Analyte	Quantiation Ion	Analyte	Quantiation Ion
Toluene	92	Iodomethane	142
Methyl methacrylate	69	n-Butanol	56
Pentachloroethane	167	Isopropyl Alcohol (IPA)	45

Table 7

List of 8260 Difficult Analytes:

1,1,2,2-Tetrachloroethane
1,2-Dibromo-3-chloropropane (DBCP)
1,4-Dioxane
2-Butanone
2-chloroethylvinyl ether
2-Hexanone
2,2-dichloropropane
4-Methyl-2-pentanone
Acetone
Bromoform
Bromomethane
Carbon disulfide
Chloroethane
Chloromethane
cis-1,3-Dichloropropene
Dichlorodifluoromethane (Freon 12)
Ethanol
Iodomethane
Isobutyl Alcohol
naphthalene
n-butanol
Styrene
Tert-Butyl Alcohol
Trichlorofluoromethane (Freon 11)
Isopropyl Alcohol (IPA)

Total Organic Carbon and Soot in Soil, Sediment

References: Perkin Elmer, "PE 2400 Series II CHNS/O Analyzer User's Manual," The Perkin-Elmer Corporation, April 1998.

USEPA, "Method 9060A Total Organic Carbon," in Test Methods for Evaluating Solid Waste, SW846, Revision 1 November 2004.

"Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability" published in Environmental Science and Technology, Vol. 31, No. 1, 1997 by Gustafsson, Haghseta, Chan, McFarlane and Gschwend.

"Reinterpreting Literature Sorption Data Considering Both Absorption into Organic Carbon and Adsorption onto Black Carbon" published in Environmental Science and Technology, Vol. 37, No. 1, 2003 by Accardi-Dey and Gschwend

1. Scope and Application

Matrices: This method is applicable for solid samples (soils, sediments, sludges).

Definitions: Refer to Alpha Analytical Quality Manual.

The organic carbon in a sample consists of a variety of organic compounds in various oxidation states. Some of these compounds may be oxidized by biological or chemical processes and can be measured by the Biochemical Oxygen Demand or Chemical Oxygen Demand tests. To measure the amount of organically bound carbon, the organic carbon molecules are broken down into single carbon units, and converted into a form that can be measured quantitatively.

The PE 2400 Series II CHNS/O Analyzer can simultaneously determine carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O) in organic material. In the CHN mode, the PE 2400 Series II CHNS/O Analyzer uses a combustion method to convert the sample elements to simple gases (CO_2, H_2O, and N_2). The resulting gases are homogenized and controlled to exact conditions of pressure, temperature, and volume. The homogenized gases are allowed to de-pressurize through a column where they are separated in a stepwise steady-state manner and detected as a function of their thermal conductivity and reported as a percentage for solid samples. Treated with hydrochloric acid prior to analysis, the Total Organic Carbon (TOC) content is determined by the amount of CO_2 in the sample. If the sample is analyzed without pretreatment, the Total Carbon (TC) content is determined. Total Inorganic Carbon can be determined by subtraction of the TOC from the TC values.

This method is applicable to the measurement of TOC and Soot in solid samples $\geq$ 100mg/Kg or $\geq$ 0.01%, dry weight. According to the instrument manufacturer, a sample of up to 70% carbon can be analyzed without dilution.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the TOC Analyzer and in the interpretation of the data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

An aliquot of solid sample is dried, homogenized, weighed into a tin capsule, pre-treated with hydrochloric acid and heated to 75°C to convert the inorganic carbon (*i.e.,* carbonate and bicarbonate in the form of CO_2) prior to analysis.

Organic carbon is measured using combustion and a carbon analyzer. The sample, of approximately 5-10 (masses less than 5 mg must be avoided since balance is not calibrated below 5 mg)mg corrected sample weight (a larger aliquot may be used for heterogeneous, or low- carbon samples to improve reproducibility), is oxidized in a pure oxygen environment, introduced into a furnace by a 60-slot Autosampler, then combusted. A greater mass may be weighed if the sample observations indicate low carbon content. As much as 20-25mg may be utilized. The carrier gas (O_2) is combined with the carbon content of the combusted sample to form CO_2. Elements, such as halogens and sulfur, are removed by scrubbing reagents in the combustion zone. A thermal conductivity detector then measures the CO_2. The amount of CO_2 derived from a sample is directly proportional to the concentration of organic carbon material in the sample.

For Soot, the sample will be transferred to an aluminum tin. The tins will be placed in an oven or muffle furnace for 24 hours at 375^{o}C. After sample is in the muffle furnace for 24hrs sample is reduced to a powder with a mortar and pestle, weighed into a tin capsule, then treated with acid then re-dried and analyzed on the TOC Analyzer.

2.1 Method Modifications from Reference

Samples are analyzed using 2 replicates unless otherwise specified by the Client. Hydrochloric acid is substituted for phosphoric acid. The use of this acid has been validated and documented by the laboratory.

3. Reporting Limits

The Practical Quantitation Limit (PQL) or Reporting Limit (RL) is 0.01 (or 100mg/Kg) for solid samples.

4. Interferences

4.1 To determine the TOC/Soot content, the inorganic fractions (carbonate and bicarbonate) must be removed prior to analysis by lowering the pH of the sample to $\leq$ 2. When removing the inorganic carbon, care must be taken in sample pretreatment to minimize the potential of loss of volatile organic carbon present in the sample.

4.2 Maintaining the samples at 4±2°C, and analyzing within the specified holding time minimizes bacterial decomposition and volatilization of the organic material.

4.3 Elements, such as halogens and sulfur, are removed by scrubbing reagents in the combustion zone. Large and/or complex organic molecules such as tannins, lignins, or humic acids may be oxidized slowly. If these compounds are suspected to be present, it is advisable to check the efficiency of the oxidation procedure with a selected representative sample, and adjust the analysis (sample size) as needed.

4.4 Oils from skin, or contact with any organic material, such as plastic containers and rubber tubing, can contaminate samples. Handle the weighing tins with tweezers.

4.5 Sample heterogeneity can be a major source of imprecision in TOC/Soot results. Recommend to clients to perform a client-specific duplicate, to evaluate precision and representativeness of the TOC/Soot results to the site.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. All relevant Material Safety Data Sheets (MSDSs) are kept on file.

Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting device and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

All standards and reagents shall be prepared in a hood while using the proper PPE.

Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintenance an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

All additional company safety practices shall be followed at all times as written in the *Chemical Hygiene Plan*.

All TOC standard solutions must be handled with caution. See pages 5-3 through 5-5 of the PE 2400 Series II CHNS/O Analyzer User's Manual for additional and specific Warnings and Precautions.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

A minimum of 5 grams of sample must be collected in a glass jar.

6.2 Sample Preservation

Soil samples must be refrigerated and maintained at 4°±2°C until drying and analysis.

Sediment samples are either processed within 28 days (or can be frozen (for programs with an EPA approved holding time extension for frozen storage) at -15C to -20C until processing.

6.3 Sample Shipping

The *Sample Receipt & Login SOP* (1559) describes how samples are normally shipped or obtained by the laboratory, precautions to be used in opening sample shipments, and sample storage conditions.

6.4 Sample Handling

Soil samples must be refrigerated and maintained at 4°±2°C until drying and analysis.

Sediment samples are either processed within 28 days or can be frozen at -15C to -20C until processing (for programs with an EPA approved holding time extension for frozen storage)

All solid samples must be analyzed within 28 days from the date of collection unless extended hold times are provided in client specific QAPP.

The *Sample Receipt & Login SOP* (1559) describes the responsibilities of sample custody including all proper documentation, verification, and tracking procedures, following Chain of Custody (COC) protocols and sample receipt procedures using the *Sample Receipt Checklist*, which includes the check for proper sample preservation and cooler temperature verification.

Internal COC procedures for sample tracking include the use of sample tracking logbooks. These procedures are also described in the *Sample Receipt & Login SOP* (1559).

7. Equipment and Supplies

7.1 Instrument: Perkin Elmer 2400 Series II CHNS/O Analyzer with computer and PE software

7.2 Detector: Thermal Conductivity

7.3 Mortar and pestle

7.4 Oven: 75 +/- 2 °C

7.5 Muffle Furnace: Capable of maintaining temperature of 375°C

7.6 Dessicator

7.7 Volumetric Flasks: Class-A, various volumes

7.8 Microbalance: Perkin-Elmer, Model AD-6. See the Manufacturer User's Manual, Section 1.2, pages 3-4, for Microbalance and weighing instructions. The Microbalance calibration is verified every day using 5.0mg - 200mg weights, in the range of daily use, and be accurate within +/1%. If the balance needs to be re-calibrated, see the Manufacturer User's Manual SOP, Section 1.1, page 3, for details.

7.9 Tweezers: Anti-Magnetic

7.10 Tin Boats: 5 X 8 mm.

7.11 Polypropylene beakers: 50 mL, for weighing wet samples.

7.12 Glass Beads: Filter Aid 400, High-density; from Perkin Elmer or 3M. Used as the CCB/Method Blank media. Beads must be muffled at 375°C prior to use to remove any trace carbon.

7.13 Gas-tight syringes: Various measuring sizes, including 10μL for direct sample spiking

7.14 Transfer pipets and/or Eppendorf pipettor for delivering HCl: Polypropylene.

7.15 Printer : OKIDATA Microline 320 Turbo-9 Pin or equivalent

Note: All maintenance records including routine upkeep and outside service visits are maintained in the instrument maintenance logbooks. See the Manufacturers User's Manual for detailed troubleshooting instructions and routine maintenance, as needed.

8. Reagents and Standards

Use reagent grade chemicals for all reagents. All reagents and standards are stored at room temperature and expire 6 months from preparation, unless otherwise indicated below.

8.1 Deionized (DI) water: All references to DI water refer to ASTM Type II reagent grade water which has been boiled, cooled and capped to eliminate CO_2. Water must be replaced weekly.

8.2 10% Hydrochloric Acid solution: Add 300mL of concentrated HCl (34-37%) to 700mL of DI water. Mix thoroughly and cool to room temperature before use. Store in a glass bottle and keep at room temperature. This solution is stable for 6 months.

8.3 Potassium Hydrogen Phthalate (KHP), CCV or K-Factor for TOC:

CCV- 10,000mg/L equivalent to 10,000mg/Kg Carbon, or 1.0 % TOC:

Dry potassium hydrogen phthalate (KHP) (primary standard grade) at 110°C for 1 hour. Cool to room temperature in a dessicator. Dissolve 2.128g of KHP (primary standard grade) in ASTM DI water, and dilute to 100mL in a volumetric flask. This standard is good for 6 months, stored at room temperature. Discard solution if discoloration or any signs of bacterial growth are observed.

8.4 Potassium Hydrogen Phthalate (KHP) Spiking Solution/MS/ICV: 10,000mg/L equivalent to 10,000mg/Kg Carbon, or 1.0 % TOC. From a separate source than the solution in Section 8.3, Sigma Aldrich or equivalent.

Dry KHP (primary standard grade) at 110°C for 1 hour. Cool to room temperature in a dessicator. Dissolve 2.128g of KHP (primary standard grade) in ASTM DI water, and dilute to 100mL in a volumetric flask. *Use a separate source or different lot of KHP from that used to prepare the calibration standards.*

This stock may be stored up to six months at room temperature. Discard solution if discoloration or any signs of bacterial growth are observed. Use at 1.0 % TOC concentration for solid samples, and at 10,000mg/L for aqueous samples.

8.5 Solid Standard Reference Materials: Standard Reference Material (SRM) NIST 1944, a separate source from the calibration standard that is certified at the concentration of 4.4% TOC for solid samples. SRM 1650b for Soot, this material has no certified value, however has a reference value of 78% soot carbon. ("Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability" published in Environmental Science and Technology, Vol. 31, No. 1, 1997 by Gustafsson, Haghseta, Chan, McFarlane and Gschwend.)

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 Method Blank: A method blank must be analyzed once per every 20 samples or per TOC/Soot batch, whichever is more frequent. The Blank consists of 30-50mg of Filter Aid acidified with HCl and dried. (Section 7.11).

TOC must not be detectable in the method blank at a concentration greater than the reporting limit for the given matrix (Section 3).

Corrective Action: Analysis of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Department Manager, if the samples associated with the out of control method blank are non-detect for TOC/Soot, or if sample TOC/Soot concentrations are greater than 10X the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

9.1.2 Continuing Calibration Blank: A CCB must be analyzed immediately after every CCV. The CCB is 30-50mg of Filter Aid acidified with HCl and dried. (Section 7.10).

The CCB concentration must not be greater than the reporting limit for the given matrix (Section 3).

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the CCB still exceeds the control limits, re-calibrate and/or re-analyze a fresh

blank. All samples associated with the out of control CCB must be re-analyzed (since the last acceptable CCB). Exceptions may be made with approval of the Department Manager if the samples associated with the out of control method blank are non-detect for TOC/Soot or if sample TOC/Soot concentrations are greater than 10X the blank levels. In such cases, the sample results are accepted without corrective action for the high CCB and the client is notified in a project narrative associated with the sample results.

9.1.3 Instrument Blank: An Instrument Blank must be analyzed during Calibration.run2. The Instrument Blank consists of 30-50mg of Filter Aid acidified with HCl and dried at 75°C. (Section 7.10).

9.2 Laboratory Control Sample (LCS)

Laboratory control sample (LCS) must be from a second source or lot number to verify the accuracy of the standard curve. The LCS is analyzed along with the samples. A LCS must be analyzed once per every 20 samples or per TOC batch, whichever is more frequent. For *solid* samples, the LCS is the NIST SRM at 4.4% TOC. The LCS is treated identically as samples with HCl and dried at 75°C.

LCS consists of approximately 5.0mg of SRM 1944 with a true value of 4.4% TOC (Section 8.6). This SRM is NOT evaluated for Soot since no certified concentration for Soot is published.

The acceptable recovery QC range is 75%-125% for the LCS.

Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-calibrate and re-analyze the LCS and all associated samples. Samples cannot be analyzed until an acceptable LCS is obtained. Exceptions may be made with approval of the Department Manager if the samples associated with the out of control LCS are also associated with a matrix spike that is in control. This is an acceptable measure of accuracy of the analytical procedures. An explanation of this out of control LCS recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

Second source analyzed at the beginning of the run. Sample matrix of ICV is a 1.0% carbon solution of KHP.

The acceptable recovery QC range for the ICV is 80-120%.

The **ICV** is prepared by adding 10μL of *Spiking Solution* (Section 8.4), (with a 1% TOC true value). The weight recorded is the Calibration Solution only. (10μL of solution should weigh approximately 10mg).

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the ICV still exceeds the control limits, re-calibrate and re-analyze all samples since last acceptable ICV.

9.4 Continuing Calibration Verification (CCV)

A CCV must be analyzed at a minimum of every 10 burns and at the close of an analytical sequence. This standard monitors instrument performance throughout the duration of the analytical run. Sample matrix of CCV is a 1.0% carbon solution of KHP.

The **CCV** is prepared by adding 10μL of *Calibration Solution* (Section 8.3), (with a 1% TOC true value). The weight recorded is the Calibration Solution only. (10μL of solution should weigh approximately 10mg).

The acceptable recovery QC range for the CCV is 80-120%.

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the CCV still exceeds the control limits, re-calibrate and re-analyze all samples since last acceptable CCV.

9.5 Matrix Spike

A matrix spike must be performed once per 10 samples (10% frequency). When project specifications dictate, a Matrix Spike Duplicate (MSD) may also need to be performed at the same frequency as the MS. Prepare the MS according to Section 8.4 for the given matrix.

A **Matrix Spike** sample is spiked with 10μL of *Spiking Solution* (Section 8.4), at a 1.0 % TOC true value. The weight recorded is the sample weight prior to spiking.

The acceptable recovery QC range is 75%-125% for the MS/MSD. Calculate the %RPD as in Section 9.6 when analyzing a MS/MSD pair. The acceptable %RPD is $\leq$ 25% for solid samples.

Corrective Action: If sample concentration exceeds 4X the spike amount, narrate. Repeat analysis once to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the criteria and the LCS is compliant; include a project narrative with the results to the client noting that there may be potential matrix effects on the accuracy or precision of the TOC/Soot results as evidenced by matrix spike recovery or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses (matrix duplicate) must be performed once per 20 samples (5% frequency).

Acceptable relative percent difference (RPD) of duplicates is $\leq$ 25%. Acceptance criterion is not applicable to sample concentrations less than 5X the reporting limit. Calculate RPD as follows:

$$\%RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = highest result
R2 = lower result

Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to the client noting that there may be potential matrix effects on the precision of the TOC/Soot results as evidenced by the matrix duplicate RPD exceedance.

9.7 Method-specific Quality Control Samples

High level Initial Calibration Verification (HLICV - linearity check):

This sample consists of 4% carbon solution (high calibration point).

Aliquot 50 µL using a syringe into a tin boat, record weight and analyze. (Equivalent to a 5 mg sample containing 40% TOC).

The acceptable recovery QC range for the HLICV is 80-120%.

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the HLICV still exceeds the control limits, re-calibrate.

9.8 Method Sequence

- Calibration Curve generation
- ICV
- ICB
- HLICV
- Method Blank
- LCS
- Laboratory Duplicate
- Matrix Spike
- Samples 1 – 8
- CCV
- CCB
- Samples 9 – 18
- CCV
- CCB

10. Procedure

10.1 Equipment Set-up

Samples are prioritized by the Department Manager for analysis based on hold time and client due date. Section 15.5 outlines the steps for final TOC reporting that will contain the sample analysis final results.

The analyst initials, date, sample ID #'s, sample weights (solids), or volumes (aqueous), of all Standards, QC samples and field samples, are entered into the TOC logbook. Include the standard, LCS and MS IDs and concentrations. Use TOC weight spreadsheet to record all information.

10.1.1 Pretreatment for Total Organic Carbon:

This procedure is used to remove the inorganic compounds in the sample prior to the determination of Total Organic Carbon. If Total Carbon is to be determined, addition of the acid solution is omitted.

10.1.1.1 Aliquot 10-15g of wet solid sample into a weighing vessel. Remove shells, rocks and other large non-representative materials with tweezers. Record weight of vessel and vessel + wet sample.

10.1.1.2 Heat for 4 hours at 75 +/- 2 °C. Return sample to oven if additional drying time is required(in ½ hour increments) Record time in and out of oven. Cool sample to room temperature in dessicator. Record weight of vessel + dry sample.

10.1.1.3 Add 10% HCl (Section 8.2) dropwise to dry sample to induce effervescence (removal of inorganic carbon). Do not use excessive acid.. Only add enough acid to exhaust the effervescence. In lieu of additional acid, use DI water to wet the sample in order to form a slurry if necessary, to distribute the acid throughout the sample. Record the volume of HCl used for each sample.

10.1.1.4 Return the sample to the oven. Heat for 4 hours at 75 +/- 2 °C. Return sample to oven if additional drying time is required(in ½ hour increments) Record time in and out of oven. Cool to room temperature in dessicator. Record final weight of dry sample + vessel. The change in weight of dry sample after acidification is used to adjust the weight of the aliquots used for analysis.

10.1.1.5 Take a sub-aliquot of approximately 2-3 g of dry sample and perform moisture content using SM2540D. The corrected sample weight (CSW, see Section 11.4) is corrected using this result (see Section 11.4 for calculations)

10.1.1.6 Grind sample with mortar and pestle to homogenize. Place ground sample into a 4 mL vial.

10.1.1.6.1 If Soot analysis is required, take a portion of the dried sample aliquot(prior to acidification), approximately 5-10 gm, and place into a muffle furnace at 375°C for 24 hours. Once the sample aliquot is cooled to room temperature, proceed to the acid addition step, Section 10.1.1.3. Samples for Soot are read analytically in the same way as Routine TOC.

10.1.1.7 Weigh (and record weight of) each replicate into a tin capsule (see Section 10.3 for appropriate weight range). Record the weight and use the TOC weight spreadsheet to calculate the corrected sample weight (CSW) which is entered into the analyzer.

10.2 Initial Calibration

On the accompanying computer, or "PC", Perkin Elmer software has been loaded to collect and save sample and standard analysis data. Create a file to store the data from each day's analytical sequence by clicking on the PE 2400 icon, go to "new" and "save" the file "as" the name of the date of the analysis (*i.e.,* 060503.chn). Data from each standard or sample analysis will be automatically stored in this file for future reference

10.2.1 Follow the instrument Manufacturers User's Manual for instrument set up and see Sections below for an outline of the initial, daily calibration procedure.

See the Manufacturers User's Manual, page 5-69, for the default instrument conditions when in the CHN operating mode.

10.2.2 *Routine* TOC/Soot Calibration Method:

TOC analyses use K-Factors (3 repeat analyses of a 1.0% TOC standard, Section 8.3) and Blanks, calculated and averaged by the instrument when the calibration is being performed. Follow the instrument Manufacturer's User's Manual, page 4-67. Calibration of the instrument using K-Factors and Blanks is performed once per day of analysis.

Load the Autosampler (Section 10.3.2) for combustion to analyze Blanks and Standards in the order listed below. All "Blanks" and "K-Factors" are prepared in the same way. Analyze the standards following the procedure in Section 10.3.

- **Instrument "Primer"** (~3.5mg of SRM 1944, with true value of 4.4% TOC) only if new column.
- **Blank 1** (30-50mg of Filter Aid) only if new column
- **K-Factor 1** (10µl of *Calibration Solution*, Section 10.3,)
  The weight recorded is the Calibration Solution, only.
- **Blank 2** (30.0mg of Filter Aid)
- **K-Factor 2** (10µl of *Calibration Solution*, Section 10.3,)
  The weight recorded is the Calibration Solution, only.
- **K-Factor 3** (10µl of *Calibration Solution*, Section 10.3,)
  The weight recorded is the Calibration Solution, only.

The K-Factors must reproduce from the mean value within +/- 15% of known K factor for each instrument for carbon. The calculation involves the comparison among the latest K-Factor run, the prior K-Factor run, and the current running average K-Factor. An "out of tolerance" message will be displayed if this criterion is not met.

The ICV/LCS and Method Blank are prepared to run immediately following the third K-Factor, given the instrument is “in tolerance”.

Corrective Action: If, at any time during the 6–step process of calibration, one of the K-Factors or Blanks fail, repeat analysis of failing sample.

10.3 Equipment Operation and Sample Processing

Prepare a Calibration per Section 10.2.

An ICV/ICV and LCS/Method Blank are analyzed immediately following the third K-Factor of the Routine Calibration, provided the instrument is “in tolerance”. The LCS and method blank must be run with the same number of replicates that are required for the samples in the run.

Remove all pretreated samples from the oven, and allow to cool in a dessicator.

Weigh approximately 5-10mg (to an accuracy of 0.01 mg) (masses less than 5 mg must be avoided since balance is not calibrated below 5 mg) of sample into a tin boat using the Perkin-Elmer Microbalance, Model AD-6, which is linked to the instrument. Samples that are known to be high in TOC may use a lesser sample aliquot. Samples where it is observed that the TOC may be low, weight as much as 20-25mg. Record the weights in the TOC/Soot logbook. Matrix Duplicate and Matrix Spike samples are weighed as separate samples, with their own replicates. As noted in Section 10.1.1.6, use the TOC weight spreadsheet to correct the sample weights for all replicates and re-analyses.
Any observations regarding the sample composition must be noted in the TOC/Soot logbook such as: sandy, contains shells or rocks, contains grass or roots, is light/white in coloration, etc.

Analyze a CCV/CCB (Section 9.4 and 9.1.3) after every 10 “burns”, and at the end of the analytical sequence.

If following the *Lloyd Kahn* Calibration method, analyze the first set of batch QC samples, the ICV/LCS and Method Blank, followed by field samples with a CCV/CCB analyzed every 10 “burns”, and at the end of the analytical sequence.

10.3.1 Multiple Sample Instrument Loading:

10.3.1.1 Each tin boat is carefully folded with anti-magnetic tweezers, and placed into its respective holding tray position. (See the Manufacturer User’s Manual SOP, Section 1.2, page 4, for tin boat folding details.) Weights, sample tray position and Autosampler location are recorded in the TOC/Soot logbook. The analyst must be very careful when transferring samples. Documentation of sample consistency is very important. See Figure 1 for an example logbook page.

10.3.1.2 Adjust the Autosampler tray to begin sampling at auto-slot #1 and carefully transfer the folded tins containing the samples to the correct auto-slot, using tweezers. All Instrument Calibration Standards (Blanks, K-Factors and/or *Lloyd Kahn* Calibration Standards) are loaded first. Immediately following will be the first batch QC samples, the ICV/LCS and Method Blank for the run, followed by field samples. Rotate the Autosampler in a *counter clockwise* manner.

10.3.1.3 A maximum of 60 tins can be held on the Autosampler at one time. The instrument only allows for 100 total samples (including calibration/blank samples) to be programmed for any given run.

10.3.2 Sample Analysis

10.3.2.1 Select the *Auto Run* option on the keypad on the instrument. The following menu appears:

AUTO RUN NO. 1
1B 2K 3S
where: B = Blank
K = K-Factor
S = Sample

Assuming the *Instrument Standardization* occupies auto-slots 1-6, Enter "3" for Sample, then select "Enter". Enter the weight of the "Primer" Standard (position 1) and select "Enter".

10.3.2.2 You will be prompted for AUTO RUN NO. 2 as in Section 10.3.3.1, except now there is a function "4NP" which allows the analyst to stop/erase everything by selecting this feature.

- Enter "1" for the Blank. The weight will not be asked for.
- Enter "2" for the first K-Factor, then select "Enter". Enter the weight of your K-Factor (the standard weight should be ~ 10mg). Select "Enter".
- Enter "1" for the second Blank, then "Enter".
- Enter "2" for the second K-Factor, then "Enter". Enter the weight, then "Enter".
- Enter "2" for the third K-Factor, then "Enter". Enter the weight, then "Enter".

10.3.2.3 All remaining sample weights are added by entering "3" for Sample, and then by entering the sample weight. Continue adding all information to the run sequence, using the keypad. When entering sample identifications, and letters are necessary, refer to the Manufacturer's User's Manual, page 3-4, for instructions for converting numbers to letters.

10.3.2.4 When all sequence information is entered and complete, select "Start". The analyses will begin. The computer will continue to display the "AUTO RUN" menu during sample analysis.

10.3.2.5 The Standard and Sample data prints out as it is collected on the line printer, and is also collected on the linked computer using the PE 2400 software. The data on the computer is saved in a file named as the date of the analysis (*i.e.,* 060503.chn) for future reference, as noted in 10.2.

10.3.2.6 When all analyses are complete, refer to the Manufacturers User's Manual SOP, Section 3.1, page 7, for instructions to "Shut Down" the instrument.

10.3.2.7 Evaluate all batch QC samples first, to confirm or exclude the need for re-analyses. The ICV/LCS/HLICV, Method Blank, CCB/CCV samples must meet the acceptance criteria in Section 9. Follow the corrective action for any QC failures.

10.3.2.8 Evaluate the field samples, LCS/SRM, and MS/Duplicates next. If replicate "burns" vary by more than 30%, re-weigh two additional aliquots similar to the initial weights used, and re-analyze the sample. For a 2-replicate analysis, this provides a total of four burns/samples. If the second set of replicates also has RPD > 30%, report the first set. If the re-analysis has an RPD which passes, report the re-analysis. Three and four replicate analysis may be requested, and the average of the multiple burns is reported, unless otherwise indicated by the client. Analysts should use judgement in cases where there may have been an error with the autosampler or other factor (incorrect loading, etc.) which may have led to RPD failure. The Lloyd Kahn method requires that quadruplicate analysis be performed on one sample per batch of up to 20 samples and that the RSD must meet precision criteria of 3 times the standard deviation of laboratory generated sample data. One sample per batch of up to 20 must be analyzed in quadruplicate. RSD criteria is 40%. This value was generated using 30 data points of quadruplicate analysis of various concentrations and multiple instruments. The RSD was calculated for each sample in quadruplicate, and the RSD limit is based on 3 times the average standard deviation. Failure to meet this criteria requires that the entire batch be re-analyzed.

10.4 Continuing Calibration

A CCV must be analyzed as outlined in Section 10.3.3 at a minimum of every 10 burns and at the close of an analytical sequence.

10.5 Preventive Maintenance

The reduction column is changed after every 100-200 runs. The combustion column is replaced as needed.

11. Data Evaluation, Calculations and Reporting

11.1 Procedures for data and record management for TOC/Soot analysis must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP, 08-01. All records are stored in such a manner as to be safe and accessible for at least 10 years.

11.2 The relevant TOC/Soot laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* Work Instruction 1556, and all record keeping and document control practices.

11.3 Sample results for TOC/Soot are directly reported from the instrument printout.

11.4 Calculations: The following calculations are applied by the instrument when determining the percentage of Total Organic Carbon or Soot: Note: sample weights are corrected for sample mass changes due to acidification prior to analysis and entered into the instrument software as the corrected weights, therefore, no correction is required after analysis.

Corrected Sample Weight(CSW): Weight that is entered at the instrument, corrected for sample mass change after acidification.

CSW = aliquot wt (mg) * total sample dry wt (g) (prior to acidification) * dry wt.@ (75°C)
Total sample dry wt (g) (post acidification) *dry wt.@(105°C)

Carbon Blank (CB): Used to make all other determinations, namely, K-Factor and weight percent calculations. When run alternately with samples, the instrument averages the blank values.

Carbon Blank (CB) = Carbon Read – Nitrogen Read

Carbon K-Factor (C KF): K-Factor, or detector calibration factor, is determined when a known standard is analyzed to calibrate the analyzer in terms of micrograms of carbon. This calibration factor is then used to determine unknowns.

C KF = ((CR – NR) – CB x 100) / (CSW x C Theory Wt. %)

where: CR = Carbon Read
NR = Nitrogen Read
CB = Carbon Blank
CSW= Corrected Sample Weight (mg) (using corrected weight recorded in Section 10.3)
C Theory Wt % = 1% (based on the concentration of the K-Factor standard)

Carbon (mg/Kg):

Carbon (mg/Kg) = (((CR – NR) – CB) x 1000) / (SW x C KF)

where: CR = Carbon Read
NR = Nitrogen Read
CB = Carbon Blank
CSW = Corrected Sample Weight (mg)
C KF = Carbon K-Factor

To convert to % C divide result in mg/Kg by 10000

Example Calculation of Percent TOC/Soot from the instrument, converted to mg of TOC/Soot:

If the result from the instrument is 1.083% and the sample weight is 10.76mg:

1.083% = 100 x [**X** / 10.76mg] (to solve for **X,** divide each side by 100)
0.01083 = **X** / 10.76mg (multiply each side by 10.76mg)
0.116mg = **X**

11.5 Reporting Results

The following procedures must be followed for reporting of TOC/Soot results:

Go to the next page in the bound TOC/Soot logbook.

Retain all computer printouts for the standard and sample analyses of this sequence.

Print the sequence from the computer and review it to make sure that no weight or sample ID transcription errors were made.

Calculate the recoveries for the QC samples (IVC/LCS, CCV, MS and RPD between the duplicate samples) and ensure the Method Blank and CCB meet the criteria. Remember any "QC Failures" require corrective action. See Section 9.0 or 12.0.

After reviewing the final logbook entries, make a copy of the logbook page for secondary review.

Go to the LIMS and "batch" and "associate" the set of QC samples and field samples that were just analyzed.

Enter the Final Results into the LIMS report sheet and check for transcription errors.

Transfer all associated sample paperwork to the Department Manager for the second level review and approval.

All TOC/Soot results are reportable without qualification if analytical holding times are met, preservation (including pH and cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference was not suspected during analysis of the TOC/Soot samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis.

QC Parameter	Acceptance Criteria
Initial Calibration Curve, *Lloyd Kahn*	r $\geq$ 0.995 and slope ± 10% the historical curves
Initial Calibration Verification	80-120% of True Value
Linearity check (high level ICV)	80-120% of True Value
Method Blank	< reporting limit for matrix
Laboratory Control Sample	75-125% R
Matrix Duplicate	25%RPD for results <5x reporting limit
Replicate burns (each sample)	30% RPD
Quadruplicate analysis(1 per batch)	< 40 % RSD
Matrix Spike	75-125% R
Matrix Spike Duplicate if requested	75-125%R/25%RPD
Continuing Calibration Verification	80-120% of True Value
Continuing Calibration Blank	< reporting limit for matrix

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 outlines sample batch QC acceptance criteria. If non-compliant TOC/Soot results are to be reported, the Department Manager and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager is notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

The *Hazardous Waste and Sample Disposal* SOP (1797) must be referenced for disposal of used standards, solvents, acids, reagents or other chemicals.

Once satisfactory TOC results have been generated, the sample containers must be scanned back into LIMS and returned to the appropriate sample refrigerator and held for 30 days.

All waste generated must be transferred to the waste disposal area. Dispose all solid samples in solid waste container. Aqueous samples must be poured into a 55-gallon drum marked acidic/non-chlorinated waste.

Refer to the *Chemical Hygiene Plan* and the *Hazardous Waste & Sample Disposal SOP* (1797) for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

PE 2400 Series II CHNS/O Analyzer User's Manual

Sample Receipt & Login SOP (1559)

Perkin-Elmer, Model AD-6 Manufacturer User's Manual

Document Control SOP 1729

Laboratory Notebook Usage Work Instruction (WI 1556)

SOP/1732 MDL Generation

SOP/1739 IDC Generation

Hazardous Waste & Sample Disposal SOP (1797)

16. Attachments

None.

Total and Amenable Cyanide

References: **Method 9010C / 9012B,** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 2 and Revision 3 2004

SM 4500CN-CEG. Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 10-204-00-1-A, Lachat Instruments, 6645 West Mill Road, Milwaukee, WI 53218, 1994.

Method 9014 (Modified). SW-846, Test Methods for Evaluating Solid Waste: Physical / Chemical Methods, EPA SW-846, Update III, 1997.

1. Scope and Application

Matrices: This method is applicable to waters, liquids, solids, soils and sludges.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

The following SOP is a reflux-distillation procedure used to extract soluble cyanide salts and many insoluble cyanide complexes from wastes and leachates. It is based on the decomposition of nearly all cyanides by a reflux distillation procedure using a strong acid and a magnesium catalyst. Cyanide, in the form of hydrocyanic acid (HCN) is purged from the sample and captured into an alkaline scrubber solution. The concentration of cyanide in the scrubber solution is then determined by flow injection analysis on a Lachat Analyzer.

This method was designed to address the problem of “trace” analyses (<1000ppm). The method may also be used for “minor” (1000ppm – 10,000ppm) and “major” (>10,000ppm) analyses by adapting the appropriate sample dilution. However, the amount of sodium hydroxide in the standards and the sample analyzed must be the same.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer

This method is restricted to use by or under the supervision of analysts experienced in the operation of the distillation unit and/or the Lachat Instrument, and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide concentration in the absorbing solution is then determined colorimetrically by Lachat flow injection analysis.

2.1 Method Modifications from Reference

The sample size used is 50mL. The Midi distillation unit has demonstrated the ability to achieve the same RDL using 50mL instead of 500mL sample volume. Refer to EPA Method 335.4.

Modification for Method 9014: An automated determination of cyanide using the Lachat instrument is used instead of manual spectrophotometric determination.

Modification for amenable cyanide: Analysis is not prepped under amber light.

3. Detection Limits

The Reported Detection Limit for aqueous samples is 0.005mg/L; soil and solid samples is 1mg/Kg.

4. Interferences

4.1 Instrumental

None.

4.2 Parameters

4.2.1 Interferences are eliminated or reduced by using the distillation procedure. However, chlorine and sulfide are interferences. Refer to Section 9.1.

4.2.2 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under test conditions to generate HCN. The possibility of interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation. Nitrate and nitrite are interferences when present at levels higher than 10mg/L and in conjunction with certain organic compounds.

4.2.3 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10mg/L were not found to interfere.

4.2.4 Fatty acids, detergents, surfactants, and other compounds may cause foaming during the distillation when they are present in high concentrations. Add anti-foaming agent to the sample during the distillation procedure (Section 9.2).

4.2.5 Carbonates and aldehydes are possible interferences

5. Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following chemicals have the potential to be highly toxic or hazardous. For detailed explanations consult the MSDS:

- Cyanide
- Sulfuric acid
- Pyridine
- Chloramine-T

6. Sample Collection, Preservation, and Handling

6.1 Sample Collection

Samples are collected in plastic or glass containers. All containers must be thoroughly cleaned and rinsed.

Oxidizing agents such as chlorine decompose most cyanides. Testing for chlorine must be done in the field prior to sample preservation.

6.2 Sample Preservation

Prior to preservation, samples must be tested for chlorine (Section 6.1).

Aqueous samples are preserved with 50% sodium hydroxide in the field to a pH $\geq$ 12 at the time of collection.

Samples and distillates are stored in the refrigerator at 4 ±2 °C.

6.3 Sample Handling

When properly preserved, cyanide samples are stored for up to 14 days prior to sample preparation steps.

Distillates must be analyzed within 14 days of distillation. Samples must be analyzed within 14 days of receipt.
Note: for MCP-TCN samples must be analyzed within 24h after distillation.

7. Equipment and Supplies

7.1 Cyanide Midi Distillation Unit: Lab Crest, BGL or comparable midi distillation unit. With reaction vessels, collection vessels, cold fingers and impingers.

7.2 pH paper: Range 1-14

7.3 Lead Acetate Paper

7.4 Vacuum source

7.5 50mL centrifuge tubes: New, plastic, with caps.

7.6 KI starch paper: Residual Chlorine sensitivity

7.7 Class A volumetric flasks: 25, 50, 100, 500 and 1000mL

7.8 Graduated cylinders: 50mL glass or plastic

7.9 Eppendorf pipettor or pipets: 0.5, 1, 2, and 5mL

7.10 Lachat 8000 Flow Analyzer: Including Quick Chem software, autosampler, pump and accessories.

7.11 Balance: Capable of weighing to 0.0001gram

7.12 Beakers: 100mL

7.13 Chiller

7.14 Stir plate

7.15 Stir bars

8. Standards and Reagents

Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

8.1 Standards and Reagents for Distillation

8.1.1 Reagent Water: All references to water in this method refer to Deionized Water (DI) from Alpha's water treatment system.

8.1.2 Ascorbic Acid, $C_6H_8O_6$: Powder. Store at room temperature. Expires upon manufacturer's specified date.

8.1.3 Sodium hydroxide solution (1N), NaOH: In a 1L volumetric flask, dissolve 40g of NaOH. Bring to volume with DI water. Store at room temperature. Expires one month from date of preparation.

8.1.4 Sulfamic acid (0.4N), H_2NSO_3H: In a 1L volumetric flask, dissolve 40g H_2NSO_3H. Bring to volume with DI water. Store at room temperature. Expires 6 months from date of preparation.

8.1.5 Sulfuric acid (1:1), H_2SO_4: To a 1L volumetric flask, add 500mL DI water. Slowly and carefully add 500mL of concentrated H_2SO_4. Store at room temperature. Expires one month from date of preparation.

8.1.6 Magnesium chloride solution (2.5M), $MgCl_2 \cdot 6H_2O$: In a 1L volumetric flask, dissolve 510g of $MgCl_2 \cdot 6H_2O$. Bring to volume with DI water. Store at room temperature. Expires 6 months from date of preparation.

8.1.7 LCS, 1000ppm cyanide stock solution: Commercially available standard with a certificate of analysis and from a different source than the Lachat calibration standards. Purchased from Ricca, Catalog # 2543-32. Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.1.8 LCS 10ppm cyanide working solution: Pipet 1mL of 1000ppm cyanide stock solution(Section 8.1.7) into a 100mL volumetric flask. Add 10mL of 1N NaOH (Section 8.1.3). Bring to volume with DI water. Prepare each day of use.

8.1.9 1000ppm Stock Spiking Solution: 1000ppm cyanide standard available commercially with a certificate of analysis. This is from a different source than the LCS (8.1.7). Purchased from LabChem Inc., Catalog # LC13545. Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.1.10 10ppm Working Cyanide Spiking Solution: Pipet 1mL of the 1000ppm Stock Spiking Solution (Section 8.1.9) into a 100mL volumetric flask. Add 10mL 1N NaOH (Section 8.1.3). Bring to volume with DI water. Prepare fresh each day of use.

8.1.11 pH 4 Acetate Buffer solution: In a 500mL volumetric flask, dissolve 410g of sodium acetate trihydrate. Bring to volume with DI water. Adjust to pH of 4.5 with acetic acid (Section 8.1.13). Store at room temperature. Expires 6 months from date of preparation.

8.1.12 Lead Carbonate Powder, [Pb (CO3)]

8.1.13 LCS 0.5 ppm Cyanide Working Solution: Pipet 5mL of the 10ppm Working Cyanide Spiking Solution (Section 8.1.10) into a 100mL volumetric flask . Add 1mL of 10N NaOH (Section 8.1.16). Bring to volume with DI water. Prepare each day of use.

8.1.14 Concentrated Acetic Acid: Store at room temperature. Expires upon manufacturer's specified date.

8.1.15 Ottawa Sand

8.1.16 Sodium hydroxide solution (10N), NaOH: In a 1L volumetric flask, dissolve 400g of NaOH. Bring to volume with DI water. Store at room temperature. Expires 6 months from date of preparation.

8.1.17 Total Cyanide SRM: ERA catalog # 541. Store in room temperature. Expires upon manufacturer's specified date.

8.1.18 Calcium Hypochlorite Solution: Dissolve 5g Ca(OCl)2 in 100mL Deionized water. Store in an amber colored bottle in the dark. Expires monthly.

8.2 Standards and Reagents for Lachat Analysis

8.2.1 Helium gas: To prevent bubble formation, degas all solutions except the standards with helium. Use He at 140kPa (20 lb/in^2) through a helium degassing tube (Lachat part number 50100). Bubble He vigorously through the solution for one minute.

8.2.2 Reagent 1. Carrier, 0.1N Sodium Hydroxide: In a 1L plastic container add 10mL of 10N NaOH (Section 8.1.16). Bring to 1L volume with DI. Store at room temperature. Prepare fresh bi-weekly.

8.2.3 Reagent 2. Acetate Buffer, 2.68M: In a 1L volumetric flask, dissolve 163g spdium acetate trihydrate (acetic acid, sodium salt trihydrate, $CH_3CO_2NA•H_2O$) in approximately 800mL of water. Add 40mL of acetic acid to solution. Dilute to the mark and invert to mix. Store at room temperature. Prepare fresh monthly.

8.2.4 Reagent 3. Chloramine-T: Dissolve 2.0g chloramine-T hydrate in 500mL DI. Prepare fresh daily.

8.2.5 Reagent 4. Pyridine-Barbituric Acid Reagent: Under a fume hood, place 15g barbituric acid in a 1L beaker and add 100mL water, rinsing down the sides of the beaker to wet the barbituric acid. Add 75mL pyridine (C_5H_5N) while stirring and mix until the barbituric acid dissolves. Add the 15mL concentrated hydrochloric acid (12M HCl) and mix. Store at room temperature. Prepare fresh weekly.

8.2.6 0.5ppm Calibration standard: Pipet 5mL of the 10ppm working cyanide spiking solution (Section 8.1.10) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.7 0.2ppm Calibration standard: Pipet 2mL of the 10ppm working cyanide spiking solution (Section 8.1.10) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.8 0.1ppm Calibration standard: Pipet 1mL of the 10ppm working cyanide spiking solution (Section 8.1.10) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use. This calibration standard is also used as the Continuing Calibration Verification sample.

8.2.9 0.04ppm Calibration standard: Pipet 5mL of the 0.2ppm calibration standard (Section 8.2.7) into a 25mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.10 0.02ppm Calibration standard: Pipet 1mL of the 0.5ppm calibration standard (Section 8.2.6) into a 25mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.11 0.01ppm Calibration standard: Pipet 10mL of the 0.02ppm calibration standard (Section 8.2.10) and 10mL of 0.1N NaOH into a container and mix. Prepare each day of use.

8.2.12 0.004ppm Calibration standard: Pipet 5mL of 0.04ppm calibration standard (Section 8.2.9) into a 50mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.13 0.1ppm ICV standard: Pipet 1mL of the 10ppm LCS cyanide working solution (8.1.8) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

9. Procedure

9.1 Screening for Chlorine and Sulfide Interference

9.1.1 Chlorine Interference

Oxidizing agents, such as chlorine, decompose most cyanides. Test by placing a drop of sample on a strip of potassium iodide (KI) - starch paper previously moistened with acetate buffer solution, pH 4. If positive indication is noted, then treat an aliquot of sample with Ascorbic Acid (Section 8.1.2). Repeat this test until the KI paper is negative. Immediately inform the Department Supervisor of this interference.

Manganese dioxide, nitrosyl chloride, etc., if present also may cause discoloration of the test paper.

9.1.2 Sulfide Interference
Oxidized products of sulfide convert CN- to SCN- rapidly, especially at high pH. Test for S^{-2} by placing a drop of sample on lead acetate test paper previously moistened with acetic acid buffer solution, pH 4 (Section 8.1.11). Darkening of the paper indicates presence of S^{-2}. Add powdered lead carbonate [Pb (CO3)] in 1g increments to the whole sample volume. Re-test with acetate paper. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper. Record in the sample prep logbook the amount of lead carbonate added to the sample.

9.2 Distillation

9.2.1 Add 50mL of shaken liquid sample, or 1gram of a well-homogenized solid sample and 50mL of DI, to the 50mL reaction vessel.

9.2.2 For the Liquid High LCS, fill one 50mL reaction vessel with 50mL DI. For the soil High LCS, add 1g Ottawa Sand (Section 8.1.15) and 50mL of DI. After the system has been charged with air, add 1mL of 10ppm LCS cyanide working solution (8.1.8) to the closed system. (Final concentration equals 0.2mg/L.)

For the Liquid Low LCS, fill one 50mL reaction vessel with 50mL of DI. For a soil Low LCS, add 0.2-0.3 g of SRM (sec 8.1.17) and 50mL of DI. Record exact SRM weight.For liquid samples: After the system has been charged with air, add 0.5mL of 10ppm LCS cyanide working solution (Section 8.1.8) to the closed system. (Final concentration equals 0.1mg/L.) **Don't add liquid Cyanide Standard for soil samples!** Final LCS soil concentration will change based on SRM lot
Samples for Method 9010C/9012B: Prepare a LCS Duplicate along with the LCSs described above.

9.2.3 For the method blank for liquid samples, fill one 50mL reaction vessel with 50mL of DI. For the method blank for soil samples, fill a 50mL reaction vessel with 1g of Ottawa Sand (Section 8.1.15) and 50mL DI.

9.2.4 For the matrix spike, fill a 50mL reaction vessel with 50mL of sample that has been chosen to be spiked. For soil samples, use 1g of soil and add 50mL of DI water. After the system has been charged with air, add 1mL of 10ppm working cyanide spiking solution (8.1.10) to the closed system.

Samples for Method 9010C/9012B: Prepare a Matrix Spike Duplicate (MSD) in the same manner as the MS, as described above.

9.2.5 For the duplicate, fill a 50mL reaction vessel with a duplicate aliquot of 50mL, or 1g soil and 50mL DI water of a sample that has been chosen to be duplicated.

9.2.6 Into the receiver or scrubber tube add 5mL of a 1N NaOH solution and add 40mL of DI water.

9.2.7 Arrange tubes in the distillation unit noting in the logbook which sample is in which glassware. The glassware is numbered and consistently placed in the same position in the distillation unit.

9.2.8 Assemble the unit completely. Turn on the pump. There must be gas bubbling in each tube. Check to make sure all connections are tight and bubbles are flowing at an equal rate in each sample tube. If not, adjust flow rate with the knobs in front of each receiver tube and/or check lines to ensure they are not obstructed.

9.2.9 Add 5mL of 0.4N sulfamic acid (8.1.4) to each sample tube and rinse the closed 50mL reaction vessel with a squirt of DI. No residue is to be left of the vessel wall.

9.2.10 Add 5mL of 1:1 H_2SO_4 (8.1.5) to each sample tube and rinse the closed 50mL reaction vessel with a squirt of DI. No residue is to be left on the vessel wall. Turn on the heat. Samples are to come to a boil on all of the midi-still units.

9.2.11 After 2 minutes of heating, add 2mL of 2.5M $MgCl_2$ Solution (8.1.6) to each sample tube, followed by a rinse with DI. If foaming occurs, an additional 2mL of $MgCl_2$ Solution may be added. If foaming continues, stop the distillation for that sample and reduce the sample size by 2 – 5x (as determined by the severity of the foaming). Contact the Inorganics Supervisor for guidance.

9.2.12 Turn on the chiller.

9.2.13 Set the timer-dial on the distillation unit to “110”.

9.2.14 After 110 minutes the unit will shut off; leave the chiller running for an additional 30 minutes while the tubes cool down.

9.2.15 Pour contents of the scrubber tube into a new, labeled, centrifuge tube (Section 7.5). Carefully rinse the scrubber tube with DI water and add rinseate to the centrifuge tube to bring to 50mL volume. Cap and refrigerate for later analysis by the Lachat Instrument.

9.3 Initial Calibration of Lachat Instrument

9.3.1 Allow 15 minutes for heating unit to warm up to 60 °C.

9.3.2 Prepare a series of 7 calibration standards (Sections 8.2.6 – 8.2.12) and a 0.1N NaOH blank. Alternatively, calibration standards may be prepared by auto-diluting a 0.5ppm calibration standard (Section 8.2.6). Perform this function per the Lachat manufacturer’s instructions for the Quick Chem 8000.

9.3.3 Set up manifold as shown in Table 1.

9.3.4 Input data system parameters as shown in Table 2.

9.3.5 Place standards and blank in the autosampler, per the manufacturer’s instructions. Input the information required by the data system, such as concentration, replicates and QC scheme.

9.3.6 Inject the standards, per the manufacturer’s instructions.

9.3.7 Prepare a standard curve by plotting instrument response against standard concentration values. A calibration curve is fitted to the calibration solution concentration/response data using the computer. The calibration coefficient of the curve must be greater than or equal to 0.995 before sample analysis can begin.

9.4 Standardization (Continuing Calibration Verification)

9.4.1 After the calibration has been established, it must be verified by the analysis of an Initial Calibration Verification Standard (ICV) (Section 8.2.13). The ICV of 0.1ppm must be made from a different source than the calibration standards. If the measurements exceed ±10% of 0.1ppm, the analysis is terminated. See Section 10.6 for Corrective Actions.

9.4.2 A Blank and a Continuing Calibration Verification (CCV) sample (Section 8.2.8) are analyzed after every 10 injections. The CCV measurements cannot exceed ±10% of the CCV value of 0.1ppm and the blank result must be less than the reporting limit of 0.005 mg/L. See Section 10.6 for CCV Corrective Actions and Section 10.2 for Blank corrective actions.

9.5 Lachat Analysis

9.5.1 Following initial calibration and standardization, (Section 9.3 and 9.4), place the samples in the autosampler, per the manufacturer's instructions. Input the information required by the data system, such as concentration, replicates and QC scheme.

9.5.2 Inject the samples, per the manufacturer's instructions.

9.5.3 The data system calculates sample concentration using the regression equation. Results are mg/L for Aqueous samples and mg/Kg for soil and solid samples.

9.5.4 If sample concentrations are greater than the highest calibration standard, the distilled sample is diluted with 0.1N sodium hydroxide (NaOH) diluent (Section 8.2.2), and reanalyzed. When the automated diluter is used, 0.1N NaOH is also used. **Do not dilute distilled samples or standards with DI water.**

9.6 Preventative Maintenance

Preventative maintenance is recorded in the instrument maintenance logbook and is performed on the Lachat instrument as follows:

Daily:

1) Clean the autosampler
2) Clean the surfaces on the auto-dilutor
3) Prime the dilutor with fresh DI water
4) Clean the pump surfaces
5) Clean the detector with DI and dry with Kim-Wipes
6) Clean the instrument surfaces with DI, wipe clean with a paper towel

Bi-weekly:

1) Clean the injection ports with DI. Take apart the injection valve and inspect it for corrosion. Make sure that the valve connectors are tight and the o-rings are not worn. If the O-rings look worn replace with new ones.
2) Perform a scan disk and disk de-fragmentation on the computer.

Monthly:

1) Using DI water, clean the unions and the tees that are associated with the manifold.
2) Delete Temporary files on the computer, and clear the hard drive of all unnecessary files.

Every 6 months:

1) Replace the o-rings in the injection valve.
2) Replace the o-rings in the manifold
3) Back up the files on the computer. Delete backed files on the hard drive.

9.7 Calculations

9.7.1 The Lachat data system calculates sample concentration using the regression equation.

9.7.2 Report only those values that fall between the lowest and the highest calibration standards.

9.7.3 Report results in mg CN/L for liquids and in mg CN/kg for soils.

9.8 Amenable Cyanide Prep

9.8.1 Add 25mL, or 1g and 25mL of DI (8.1.1), to a 100mL beaker with a stir bar.

9.8.2 Add 1mL of 10ppm LCS cyanide working solution (8.1.8) to beaker.

9.8.3 Prep one sample in duplicate.

9.8.4 Add 1-2mL of calcium hypochlorite solution (8.1.18) to all samples and QC under the hood.

9.8.5 Check for the presence of chlorine by placing a drop of the sample on a KI-starch paper (7.6). It should turn blue if there is sufficient chlorine.

9.8.6 Check samples every 15 minutes for one hour for the presence of chlorine and add more calcium hypochlorite if needed.

9.8.7 After one hour of digesting, add ascorbic acid (8.1.2) until KI strip no longer turns blue.

9.8.8 Bring sample volume up to 50mL and distill following steps 9.2.6 thru 9.2.15.

10. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

10.1 Demonstration of Capability

Refer to Alpha SOP/ 1734 and 1739 for DOC information.

10.2 Blank

A minimum of one method blank is distilled and analyzed per batch of 20 or less samples. The Method Blank is utilized to determine if contamination or any memory effects are occurring. (Section 9.2.1) The blank result must be less than the reporting limit of 0.005 mg/L for liquids and 1 mg/kg for soils. If the blank result is outside of acceptance criteria, it is injected another time. If failure continues, sample analysis is terminated and the source of the problem is found and corrected. All samples analyzed since the last acceptable blank analysis must be reanalyzed.

10.3 Laboratory Control Samples (LCS) / Laboratory Control Sample Duplicate (LCSD)

Distill and analyze two LCSs per batch of 20 samples. A Low LCS is analyzed at 0.1mg/L and a high LCS is analyzed at 0.2mg/L. (Section 9.2.2)

LCS measurements for Method SM 4500 CN-CE must be within ±10%. For Method 9010C/9012B, the LCS measurements must be within ± 15% for liquids and +/- 20% for soils.

Samples for Method 9010C/9012B: LCSDs are distilled and analyzed along with the LCSs, as described above. The RPD between LCS and LCSD must be $\leq$ 20% for liquids and $\leq$35% for soils.
For soil samples: LCS and LCSD recovery must be within vendor specified acceptance criteria (it will be different for different lots of SRM)

If any LCS fails acceptance criteria for either % Recovery or RPD, analysis is terminated and samples are redigested and analyzed.

10.4 Matrix Spike

Distill and analyze one spike per batch of 20 samples. For Method 9010C/9012B distill and analyze one spike per batch of 10 samples.
For Method 9010C/9012B, the % Recovery must be within ±20% for liquids and ±35% for solids. For SM 4500CN-CE, the % Recovery must be within ±10%. (Section 9.2.4).

Samples for Method 9010C/9012B: A Matrix Spike Duplicate (MSD) is distilled and analyzed along with the MS, as described above. The RPD between MS and MCSD must be $\leq$ 20% for liquids and $\leq$35% for soils.

10.5 Duplicates

Analyze one duplicate sample for every 20 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process. (Section 9.2.5)

The RPD must be 20% or less for liquids and 35% or less for soils and solids. See Section 12 for Corrective Action if these criteria are not met.

10.6 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

The Initial Calibration Verification Standard (ICV) (Section 8.2.13) is analyzed immediately following the calibration to verify the curve. If the measurements exceeds ±10% of 0.1ppm, the analysis is terminated and recalibration must occur. An acceptable result for the ICV must be obtained prior to any sample analysis.

The Continuing Calibration Verification Standard (CCV) (Section 8.2.8) is analyzed after every 10 injections. The CCV measurements cannot exceed ±10% of the of 0.1ppm. If the CCV is not within acceptance criteria, the standard is injected again. If failure continues, sample analysis is terminated and the source of the problem is found and corrected. All samples analyzed since the last acceptable calibration verification must be reanalyzed.

10.7 Control Limits

Refer to SOP/ 1734.

10.8 Analytical Sequence

The analytical sequence is:

- Screening of samples for chlorine and sulfide
- Prep of amenable cyanide, if needed
- Distillation:
    - Samples
    - LCS Low
    - LCS High
    - Blank
    - Matrix Spike
    - Duplicate
- Analysis:
    - Calibration and Standardization of Lachat Instrument
    - CCV
    - CCB
    - ICV
    - ICB
    - 10 samples
    - CCV
    - CCB
    - 10 samples
    - CCV
    - CCB
    - Calculation of sample cyanide concentration

11. Method Performance

Refer to SOP/ 1732 for MDL/LOD/LOQ information. Refer to SOP/ 1734 and 1739 for DOC information.

12. Corrective Actions

Holding time exceedence and improper preservation are noted on the nonconformance report form. The analyst narrates the nonconformance when the project is turned in for review. The narration must state what the nonconformance was and any corrective action taken.

Perform routine preventative maintenance according to Section 9.6. Record all maintenance in the instrument logbook. Notify the Department Manager if the instrument problems are not routine in nature. The Department Manager determines whether the problem can be corrected with in-house technical staff or if the instrument vendor should be contacted to schedule service. All service calls are documented in the Instrument logbook, and a copy of the service report is given to the Department Manager.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. If any part of batch quality control does not meet acceptance criteria, the Department Manager is notified. If enough sample remains and holding time has not expired, then the batch is redistilled and reanalyzed. If there is not sufficient sample remaining to allow redistillation, then that analysis is repeated and both sets of data are reported with the nonconformance narrated on the final report.

If either the ICV, ICB, Method Blank, LCS, LCSD, CCV, or CCB recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The raw data is noted. If the standard fails again, the problem must be found and corrected. The CCV/CCB standard is remade and reanalyzed. If the standard passes, all samples analyzed since the previous passing standard are reanalyzed. The raw data is noted and all data associated with the failing standard must have one line drawn through the data, indicating its unusability.

If the standard fails after instrument maintenance, the instrument is recalibrated. A new ICV/ICB is performed, and all samples analyzed since the previous passing CCV/CCB are reanalyzed.

If following reanalysis of the LCS, it is found to still be outside acceptance criteria, the entire sample batch must be redistilled and reanalyzed. If the %RPD between the LCS/LCSD fails after reinjection, then the entire sample batch must be redistilled and reanalyzed.

If the Method Blank fails it is re-poured and reinjected. If failure continues, the associated sample data is evaluated as follows: Sample results below the detection limit may be reported with a narrative included. If samples have positive results, and the results are greater than 10x the concentration found in the method blank, the data may be reported with a narrative included. Any positive samples with results less than 10x the concentration found in the method blank must be redistilled and reanalyzed.

If the Matrix Spike recovery does not meet acceptance criteria, and the LCS recovery is acceptable, matrix interference may be assumed. The associated data may be reported with a narrative included.

If sample Duplicates are outside of the acceptance criteria, the analyst examines the sample for homogeneity. If the sample is not homogenous, this is narrated on the final report. Clean, homogenous samples are redistilled and reanalyzed within holding time.

Sample nonconformance regarding a Matrix Spike recovery or a duplicate %RSD is narrated on the final report along with the corrective action(s) taken.

13. Pollution Prevention

See Chemical Hygiene Plan for pollution prevention operations.

14. Waste Management

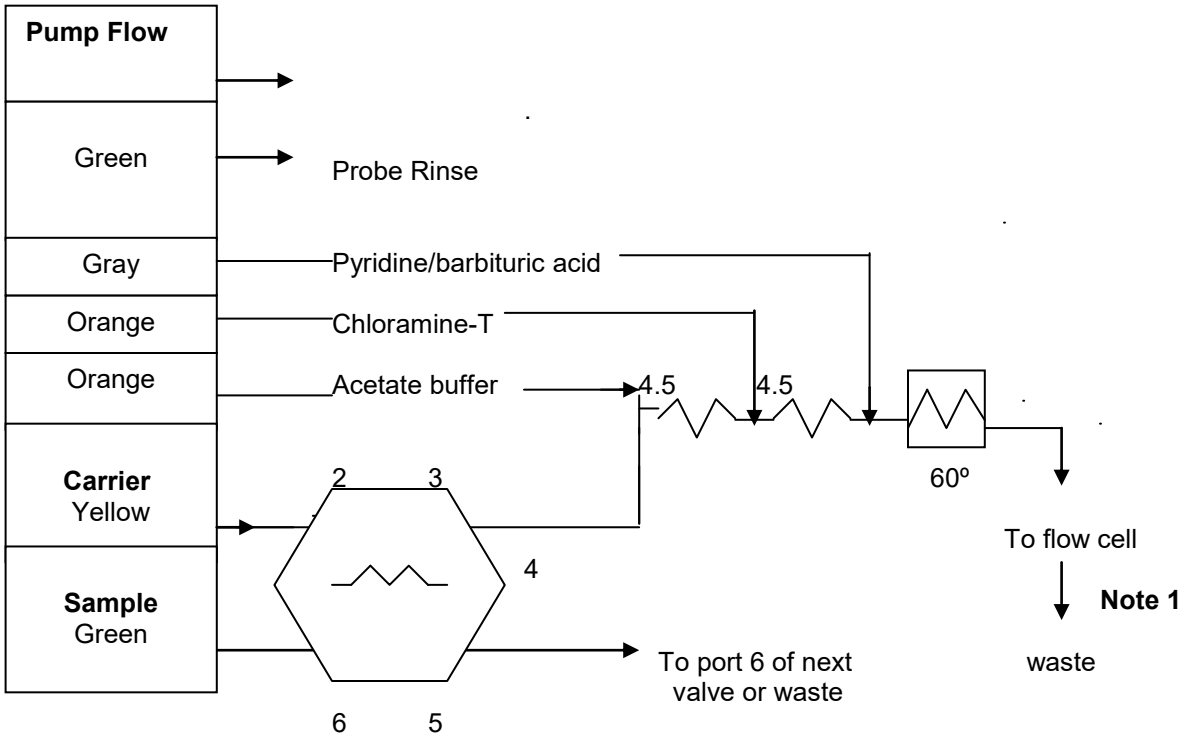
See Chemical Hygiene Plan for waste handling and disposal.

15. Attachments

TABLE 1: Cyanide Manifold Diagram

TABLE 2: Data System Parameters for QC 8000

TABLE 1
Cyanide Manifold Diagram



Sample Loop = 150cm x 0.8mm i.d. Interference Filter = 570nm
QC8000 Sample loop = 150cm x 0.8mm i.d.

CARRIER is 0.1 N sodium hydroxide solution.

All manifold tubing is 0.8mm (0.030 in) i.d. This is 5.2µL/cm.

4.5 is 70.0cm of tubing on a 4.5cm coil support

APPARATUS: An injection valve, flow cell, a 10mm path length flow cell, and a colorimetric detector module are required.

The box  shows 650cm of tubing wrapped around the heater block at the specified temperature.

Note 1: 2 meter back pressure loop, 0.52mm i.d.

TABLE 2
Data system Parameters for QC 8000

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample Throughput: 80 samples/hour, 45 s/sample
Pump Speed: 35
Cycle Period: 45

Analyte Data:

Concentration Units: mg CN^-/L
Peak Base Width: 39 s
% Width Tolerance: 100
Threshold: 25000
Inject to Peak Start: 42 s
Chemistry: Direct

Calibration Data:

Levels	1	2	3	4	5	6	7	8
Concentration ug/50mL	25	10	5	2	1	0.5	0.2	0

Calibration Fit Type: 1st Order Polynomial
Calibration Rep. Handling: Replace
Weighting Method: 1/X
Concentration Scaling: None
Force Through Zero: No

Sampler Timing:

Min. Probe in Wash Period: 14 s
Probe in Sample Period: 20 s

Valve Timing:

Load Time: 0.0 s
Load Period: 20 s
Inject Period: 25 s

D

Agency Comments on Initial Versions of SAP/QAPP

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MacLeod, Steven

From: Thein, Jeff <Jeff.Thein@dep.nj.gov>
Sent: Tuesday, November 01, 2016 9:27 AM
To: MacLeod, Steven
Subject: RE: Raritan Bay Sediment Study

Hi Steve,

It is necessary to test for cyanide. We only require that you use a NJ Certified laboratory so I can't speak to the test method.

Jeff

From: MacLeod, Steven [mailto:SMacLeod@ene.com]
Sent: Monday, October 31, 2016 2:18 PM
To: Thein, Jeff
Cc: Kelly, Megan; Mochrie, Sara; Horner, Scott (Scott.Horner@williams.com)
Subject: RE: Raritan Bay Sediment Study

Hi Jeff,
Were you able to confirm if EPA Method 8270D SIM is an acceptable method for analyzing PCB congeners for this Project?

Also, please confirm whether it is necessary to test for cyanide. This analyte is included under the "metals" list in the NJDEP 1997 dredge manual appendix.

Thanks,
Steve

ecology and environment, inc.

Steven MacLeod, *Environmental Scientist*
368 Pleasant View Drive, Lancaster, NY 14086
Office Phone: 716-684-8060 x3907 • Work Cell: 716-462-0845
smacleod@ene.com • www.ene.com

From: MacLeod, Steven
Sent: Thursday, October 27, 2016 5:22 PM
To: 'Thein, Jeff' <Jeff.Thein@dep.nj.gov>
Cc: Kelly, Megan <megan.kelly@dep.nj.gov>; Mochrie, Sara <SMochrie@ene.com>; Horner, Scott (Scott.Horner@williams.com) <Scott.Horner@williams.com>
Subject: RE: Raritan Bay Sediment Study
Importance: High

Jeff,
We would like to analyze PCB congeners using EPA method 8270D SIM. The lab we are using (Alpha Analytical) is certified in NJ to analyze PCBs as aroclors using method 8082A, and they are certified to analyze PAHs using method 8270D. However, the lab indicates that New Jersey does not offer certification for analyzing PCB congeners using the 8270D SIM method. The lab's PCB congener reporting limit using 8270D SIM is 0.8 microgram/Kg, which is below the 1 microgram/Kg PCB reporting limit listed in the NJDEP 1997 manual.

Is it acceptable to use the 8270D SIM method for PCB congener analysis? Attached in support of using this method is the lab's SOP for 8270D and results from standard reference material (SRM) tests for PCBs performed for the US Army Corps of Engineers between 2013 and 2016 – the SRM results demonstrate the range of PCB congener recoveries achieved using 8270D SIM.

Thank you,
Steve

ecology and environment, inc.

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From: Thein, Jeff [mailto:Jeff.Thein@dep.nj.gov]
Sent: Wednesday, October 19, 2016 4:30 PM
To: MacLeod, Steven <SMacLeod@ene.com>
Cc: Kelly, Megan <megan.kelly@dep.nj.gov>; Mochrie, Sara <SMochrie@ene.com>; Horner, Scott (Scott.Horner@williams.com) <Scott.Horner@williams.com>
Subject: RE: Raritan Bay Sediment Study

Steve,

I don't know the testing method but we would require a NJ certified lab conduct the analysis.

The congener list is correct.

Jeff

From: MacLeod, Steven [mailto:SMacLeod@ene.com]
Sent: Wednesday, October 19, 2016 10:43 AM
To: Thein, Jeff
Cc: Kelly, Megan; Mochrie, Sara; Horner, Scott (Scott.Horner@williams.com)
Subject: RE: Raritan Bay Sediment Study

Jeff,
Extending on my question from yesterday, can you also clarify what set of PCB congeners should be tested?
The 1997 NJDEP manual says "These congener specific results will be converted to a total PCB value by multiplying the sum of the 22 individual congeners by a factor of 2…"
We note that the following 22 PCB congeners are identified in an example sample list in the attached USACE/EPA Regional Testing Manual.
Are these the congeners that NJDEP would like reported?
Congener nos. 8, 18, 28, 44, 49, 52, 66, 87, 101, 105, 118, 128, 138, 153, 170, 180, 183, 184, 187, 195, 206, 209

Thanks again,
Steve
716-684-8060 (office)
716-462-0845 (cell)

From: MacLeod, Steven
Sent: Tuesday, October 18, 2016 7:53 AM
To: 'Thein, Jeff' <Jeff.Thein@dep.nj.gov>
Cc: 'Kelly, Megan' <megan.kelly@dep.nj.gov>; Mochrie, Sara <SMochrie@ene.com>; 'Horner, Scott

(Scott.Horner@williams.com)' <Scott.Horner@williams.com>
Subject: RE: Raritan Bay Sediment Study

Hi Jeff,
The 1997 manual says "the Department will require congener specific based analysis for PCBs using the Sloan method, NOAA Technical Memorandum NOS ORCA-71 or its equivalent. This is the same methodology that the USEPA employs…".
This appears outdated. Does NJDEP have a currently preferred (or required) PCB testing method? Perhaps EPA Method 8082A (SW-846)?

Thanks,
Steve

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From: MacLeod, Steven
Sent: Friday, October 14, 2016 5:04 PM
To: 'Thein, Jeff' <Jeff.Thein@dep.nj.gov>
Cc: Kelly, Megan <megan.kelly@dep.nj.gov>; Mochrie, Sara <SMochrie@ene.com>; Horner, Scott (Scott.Horner@williams.com) <Scott.Horner@williams.com>
Subject: RE: Raritan Bay Sediment Study

Jeff,
Thanks for the quick response earlier this week.
Based on your feedback, we will proceed with our current sampling approach to test the vibracores in roughly 3-foot sections. Sediment that extends at least 6 inches deeper than the proposed trench depth will be tested as part of a 3-foot section in each vibracore.

Regards,
Steve

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From: Thein, Jeff [mailto:Jeff.Thein@dep.nj.gov]
Sent: Wednesday, October 12, 2016 9:24 AM
To: MacLeod, Steven <SMacLeod@ene.com>
Cc: Kelly, Megan <megan.kelly@dep.nj.gov>; Mochrie, Sara <SMochrie@ene.com>; Horner, Scott (Scott.Horner@williams.com) <Scott.Horner@williams.com>
Subject: RE: Raritan Bay Sediment Study

Steve,

- VOC testing would not be required.

- Congener analysis would be appropriate for sub sea plow or jet-sled as the material is kept on the sea floor and is therefore a concern for aquatic species

- Sampling is not required for any samples that have greater than 90% sand

- Samples should be taken to the proposed depth. Due to inaccuracies in dredging, we can allow a 1 to 2 feet of overdredge so the vibracore sample should be the project depth plus your projected overdredge, if any.

- While the trench is proposed to be backfilled with clean material, the material pushed out to create the trench would still be exposed, correct? If material removed from the trench will be exposed, the bottom 6 inches from the cores shall be sampled.

Jeff

From: MacLeod, Steven [mailto:SMacLeod@ene.com]
Sent: Tuesday, October 11, 2016 6:10 PM
To: Thein, Jeff
Cc: Kelly, Megan; Mochrie, Sara; Horner, Scott (Scott.Horner@williams.com)
Subject: FW: Raritan Bay Sediment Study
Importance: High

Jeff,
Thank you for the feedback. Please clarify the following points:

- Attachment 1 of Appendix B references a large number of volatiles. However, the text of the introduction language of Appendix B says "the volatile organic compounds list… will be required on a case by case basis" (Section a on Page 92 of 124). **Please verify whether testing for the entire list of VOCs is required along all portions of the route in NJ waters** (i.e., both near the Morgan shoreline and the area near Ambrose channel north of Sandy Hook).
- Attachment 1 of Appendix B identifies Aroclor testing, but the introduction language of Appendix B says congener analyses will be required instead for in-water placement (i.e., "where aquatic species impacts are a concern") (Section b on Page 92 of 124). **Please confirm that aroclor testing (rather than congener analysis) is acceptable in areas where a subsea plow and/or jet-sled might be used.** We will plan to perform congener analysis for any areas where clamshell dredging may occur, regardless of the ultimate placement/disposal site.
- Appendix C (Dredging material data form) suggests that (chemistry) testing is not required for samples with greater than 90% sand (i.e., grain size diameter >0.0625 mm). **Please confirm that this exception applies.**
- The current sampling plan calls for chemical analysis of vibracore sections 3 feet in length (or less) partly to ensure sufficient sample volume for all analyses. The depth of vibracores will extend at least 2 feet below the max proposed dredge depth. We feel this satisfies your office's requirement for testing material 6-inches below the anticipated trench depth. **Please confirm that the proposed sampling depths/intervals are acceptable.** (*Note that once installed, the pipe/trench would be backfilled with clean, compatible material, such that the trench bottom and side-walls would only temporarily be exposed to the water column.*)

If you like, we would be happy to discuss any of these issues by phone. Your prompt reply would be greatly appreciated to facilitate the commencement of sampling and testing this month.

Sincerely,
Steve

Steven MacLeod, *Environmental Scientist*
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From: Thein, Jeff [mailto:Jeff.Thein@dep.nj.gov]
Sent: Tuesday, October 11, 2016 3:32 PM
To: MacLeod, Steven <SMacLeod@ene.com>
Subject: Raritan Bay Sediment Study

Hi Steve, I'm with the Office of Dredging and Sediment Technology and with respect to the sampling that would be required for the proposed project, please note that we are still evaluating whether we would allow re-profiling for pipe placement. The use of a clamshell bucket to remove material and place adjacent to the trench is not an approach that we've allowed. In the event that we approved some type of plowing operation, we would require bulk sediment chemistry testing on the parameters listed in Attachment 1 & 2 in Appendix B of the attached Technical Manual. In addition to sampling cores taken to the project depth, our office would also require the bottom 6 inches of each core to be sampled as that bottom 6 inches is representative to the material that will be exposed by the trenching.

Jeff

MacLeod, Steven

From: Thein, Jeff <Jeff.Thein@dep.nj.gov>
Sent: Tuesday, January 03, 2017 2:43 PM
To: MacLeod, Steven
Subject: RE: NESE Project Sediment Study Reporting

Categories: Agency Communication

Hi Steve,

DVD with excel spreadsheets is fine.

Jeff

From: MacLeod, Steven [mailto:SMacLeod@ene.com]
Sent: Tuesday, January 03, 2017 2:22 PM
To: Thein, Jeff
Cc: Kelly, Megan; Foster, Ruth; Clements, Blake; Martinez, Chris; Winston, Webb; Mochrie, Sara
Subject: NESE Project Sediment Study Reporting

Hello Jeff,
Transco completed its offshore sediment sampling effort for the Northeast Supply Enhancement Project on December 20th. The geotechnical, benthic, and chemical labs will be processing the samples over the next several weeks. We plan to submit a combined environmental sampling report to NJDEP with results of the sampling effort.

Does NJDEP have a preferred format for submittal of the bulk sediment chemistry lab data, which would be an appendix to the combined sampling report (e.g., DVD with PDF tables and/or Excel spreadsheets)?

Thank you,
Steve

ecology and environment, inc.

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www.dec.ny.gov

October 5, 2016

Timothy Powell
Transcontinental Gas Pipeline Company, LLC
2800 Post Oak Boulevard
Houston, Texas 77056

RE: Northeast Supply Enhancement Project; Raritan Bay and Lower New York Bay Sediment Study

Dear Mr. Powell:

This is in response to the submission on behalf of Transcontinental Gas Pipeline Company, LLC (Transco) from Sara Mochrie, Ecology and Environment (E&E), dated July 29, 2016 regarding the above referenced application. The submission was a response to the Notice of Incomplete Application issued by the New York State Department of Environmental Conservation (DEC) on June 2, 2016. The most recent submission included four attachments, including revised study area figures that show the new proposed route for the project.

New Proposed Route

As indicated in the conference call on August 24, 2016 with Transco and E&E representatives, DEC staff has significant concerns regarding the new proposed route, specifically miles 2-8. The original route proposed by Transco, which closely paralleled the NY/NJ state line through Raritan Bay, did not require any special provisions for either surfclams or hard clams. The new route, however, takes a significant jog north between mile 2 (about the NY/NJ line) and mile 8 (south of Great Kills Harbor/Crookes Point). This area is the most productive hard clam area in NY's portion of Raritan Bay and has been the area of an intensive shellfish transplant harvest program (not currently active, but likely to be revived in the future). It is also the site of DEC's hard clam

sampling for QPX (quahog parasite unknown). Transco has advised DEC that the proposed route change was primarily the result of consultation with the U.S. Coast Guard and Harbor Operations regarding anchorage areas in the New York Harbor area.

During the August 24th conference call DEC agreed to provide Transco with additional technical information related to the abundance of hard clams in the proposed project area. DEC also suggested during the call that Transco review this new resource information and determine if the route can be relocated to avoid or minimize the natural resource impacts identified by the DEC. (DEC provided the technical information on August 26, 2016).

On September 28, 2016, DEC staff met again with Transco regarding the new proposed route. During this meeting Transco took the opportunity to present the information (illustrated in a project map) that resulted in selecting the new proposed route. Transco also stated that the various competing resources illustrated in the project map provided little opportunity for any other significant route changes to avoid the hard shell clam area in New York.

Sediment and Benthic Sampling

DEC is aware that Transco wants to commence sediment and benthic sampling in the near term to take advantage of favorable ocean and weather conditions. Transco has indicated that this information is needed in order for it to analyze the new proposed route, both from an environmental and technical standpoint. As currently proposed, the sampling activity qualifies for coverage under DEC's statewide Water Quality Certificate because the test trench excavation originally proposed in the application has been eliminated. As such, an individual DEC approval is not required. However, Transco is seeking initial DEC concurrence regarding the new proposed route in order to avoid expending valuable resources sampling multiple routes.

Given the fact that the project is still only in the pre-application stage of the FERC review process and that an environmental analysis has yet to be completed on the project DEC cannot provide its concurrence on the new proposed route. At this time, DEC can only suggest that if Transcontinental Pipeline undertakes the sediment and benthic sampling along the new proposed route that it revise its sampling plan to include the whole area identified as "Special Permit Harvest Area".

This additional information will ultimately allow the Federal Energy Regulatory Commission, in its environmental review, to examine alternative routes that potentially avoid or minimize impacts to this important resource area. This information will also allow the DEC to more effectively review the new proposed route and any alternatives.

The DEC reserves its right to provide additional comments regarding the route and any alternatives upon review of the sediment and benthic sampling.

Thank you for the opportunity to comment on the Sediment and Benthic Sampling plan. If you have any questions regarding the above comments please contact me.

Sincerely,

Christopher M. Hogan
Deputy, Chief Permit Administrator

Cc: C. DeQuillfeldt
D. Barnes
K. Woodfield
D. English
S. Mochrie
S. McCleod
W. Little

MacLeod, Steven

From: Hogan, Chris M (DEC) <chris.hogan@dec.ny.gov>
Sent: Wednesday, September 07, 2016 1:24 PM
To: MacLeod, Steven
Cc: Mochrie, Sara
Subject: RE: Transco Raritan Bay and Lower NY Bay Sediment Study

Categories: Agency Communication

As indicated in the meeting last week the DEC has significant concerns regarding the new proposed route. The DEC realizes that Williams is seeking to mobilize the sediment sampling operations soon, but it does not make much sense to conduct surveys on a route that may ultimately change. Staff met internally this morning to discuss the project. I am revising the DEC letter to reflect what we discussed. In summary, DEC staff believe that Williams should propose additional alternatives based on the new surf calm population data provided by DEC. In addition, the alternative of utilizing the originally proposed route should be explored by either; 1) installing the line to the depth required by the ACOE; or 2) getting the ACOE to agree to a lesser depth.

Below are the DEC's comments on the sampling plan that will also be incorporated into the letter. The Department does not recommend Williams undertaking the sampling along the route as it is currently proposed until further discussion of alternatives can take place. The Department is willing to meet again in person but Williams must come to the meeting prepared with the information discussed above.

Sampling Plan Comments

Below are DEC staff's comments regarding the Sampling Plan;

1. Sediment core samples should be collected to the depth of disturbance for cable/pipeline installations and not to two feet below the bottom of the potential trench (as listed on page 28.)
2. Dividing each core sample into 3 foot increments, as proposed on page 36, is acceptable to NYSDEC. If any changes are made at the time of sampling NYSDEC should be notified and consulted.
3. Provide the total area of the offshore study area and an estimate of the total offshore area that will ultimately be disturbed by the project.
4. New York State requires all samples collected in NY to be analyzed by a NYSDOH ELAP certified lab.
5. On the table the sample sites should be labeled the same as they are on the sample location map. VCA6 vs VC6.
6. Provide information regarding the steepest angle (vertical:horizontal ratio) that the pipe can be laid to get underneath the deep navigation channels like the West Reach, Chapel Hill South Channel, and Ambrose Channel. It is likely that the pipe will have to be laid deeper when approaching and leaving these channels so the cores should be deeper as well to categorize the sediment that will be disturbed.
7. Provide information regarding the total area of the corridor? This information is helpful for evaluating the appropriate number of samples. Update details regarding the length of the new route, the exact length in New York and confirm whether the study area is still 5000 feet.

From: MacLeod, Steven [mailto:SMacLeod@ene.com]
Sent: Tuesday, August 30, 2016 6:07 PM
To: Hogan, Chris M (DEC) <chris.hogan@dec.ny.gov>
Cc: SMochrie@ene.com
Subject: RE: Transco Raritan Bay and Lower NY Bay Sediment Study

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Hi Chris, Any update on when we can get those SAP/QAPP comments? I know NYSDEC staff mentioned they were minor, but we have limited time left to review and incorporate them prior to contractor mobilization.

Thank you,
Steve

ecology and environment, inc.

Steven MacLeod, *Environmental Scientist*
368 Pleasant View Drive, Lancaster, NY 14086
Office Phone: 716-684-8060 x3907 • Work Cell: 716-462-0845
smacleod@ene.com • www.ene.com

From: Hogan, Chris M (DEC) [mailto:chris.hogan@dec.ny.gov]
Sent: Tuesday, August 23, 2016 9:55 AM
To: MacLeod, Steven <SMacLeod@ene.com>
Cc: Mochrie, Sara <SMochrie@ene.com>
Subject: RE: Transco Raritan Bay and Lower NY Bay Sediment Study

I was out yesterday. I can revise the comments and get them to you. However, Marine Resources has identified significant issues with the new route. Specifically, with miles 2-8. I think it would make sense to have a conference call to discuss before Williams proceeds with sampling. It does not make much sense to sample for an area where the DEC may have objections.

From: MacLeod, Steven [mailto:SMacLeod@ene.com]
Sent: Friday, August 19, 2016 11:04 AM
To: Hogan, Chris M (DEC) <chris.hogan@dec.ny.gov>
Cc: SMochrie@ene.com
Subject: RE: Transco Raritan Bay and Lower NY Bay Sediment Study

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Hi Chris,
Could you provided an updated estimate on when NYSDEC will be able to provide comments on the sampling plan? Do you wish to discuss any aspects of the plan before providing the written comments?

Thank you,
Steve

ecology and environment, inc.

Steven MacLeod, *Environmental Scientist*
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smacleod@ene.com • www.ene.com

From: Hogan, Chris M (DEC) [mailto:chris.hogan@dec.ny.gov]
Sent: Monday, August 15, 2016 9:10 AM
To: MacLeod, Steven <SMacLeod@ene.com>
Cc: Mochrie, Sara <SMochrie@ene.com>
Subject: RE: Transco Raritan Bay and Lower NY Bay Sediment Study

I have a conference call with staff tomorrow. DOW has prepared comments regarding the sediment sampling portion of the plan. However, the plan was also sent to the DEC's dredge team and they commenced their own review. The purpose of the meeting is to coordinate the two efforts. Because this is not a dredge project my intent was for Karen and Diane to take the lead. I need to get everyone on the same page. I will be able to send the comments to you tomorrow PM after that meeting.

Charlie has been on vacation so I do not have comments on the benthic monitoring plan. I may have to get comments to you on that portion of the document later. I assume the benthic plan is consistent with the Rockaway Lateral plan.

From: MacLeod, Steven [mailto:SMacLeod@ene.com]
Sent: Friday, August 12, 2016 5:02 PM
To: Hogan, Chris M (DEC) <chris.hogan@dec.ny.gov>
Cc: SMochrie@ene.com
Subject: RE: Transco Raritan Bay and Lower NY Bay Sediment Study

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Chris,
I just realized that I have recently sent messages to your older e-mail address (cmhogan@gw.dec.state.ny.us). I did not receive any return-replies, but please let me know if these messages are not automatically forwarded and I will re-transmit. My apologies for any inconvenience.

Have a good weekend.

-Steve

From: MacLeod, Steven
Sent: Thursday, August 11, 2016 1:48 PM
To: Chris Hogan (cmhogan@gw.dec.state.ny.us) <cmhogan@gw.dec.state.ny.us>
Cc: Mochrie, Sara (SMochrie@ene.com) <SMochrie@ene.com>
Subject: RE: Transco Raritan Bay and Lower NY Bay Sediment Study

Hi Chris,
Could you tell us when we can expect to receive NYSDEC comments on the Sampling and Analysis Plan/QAPP for the Northeast Supply Enhancement Project offshore sediment study?

Thank you,
Steve

ecology and environment, inc.

Steven MacLeod, *Environmental Scientist*
368 Pleasant View Drive, Lancaster, NY 14086
Office Phone: 716-684-8060 x3907 • Work Cell: 716-462-0845
smacleod@ene.com • www.ene.com

From: MacLeod, Steven
Sent: Friday, July 29, 2016 8:35 PM
To: Chris Hogan (cmhogan@gw.dec.state.ny.us) <cmhogan@gw.dec.state.ny.us>
Cc: deQuillfeldt, Charles (charles.dequillfeldt@dec.ny.gov) <charles.dequillfeldt@dec.ny.gov>; Horner, Scott (Scott.Horner@williams.com) <Scott.Horner@williams.com>; Gavelek, Jim (Jim.Gavelek@Williams.com) <Jim.Gavelek@Williams.com>; 'Martinez, Chris' <Chris.Martinez@williams.com>; Clements, Blake <Blake.Clements@williams.com>; Kellogg, Stephen (Stephen.Kellogg@Williams.com) <Stephen.Kellogg@Williams.com>; Maraglio, Matthew (DOS) (Matthew.Maraglio@dos.ny.gov) <Matthew.Maraglio@dos.ny.gov>; 'CR@dos.ny.gov' <CR@dos.ny.gov>; Fronckowiak, Megan L. (MFronckowiak@ene.com) <MFronckowiak@ene.com>; Mochrie, Sara (SMochrie@ene.com) <SMochrie@ene.com>; 'Naomi Handell' (Naomi.J.Handell@usace.army.mil) <Naomi.J.Handell@usace.army.mil>; 'dawn.mcreynolds@dec.ny.gov' <dawn.mcreynolds@dec.ny.gov>; karen.woodfield@dec.ny.gov; diane.english@dec.ny.gov; katie.axt@dec.ny.gov
Subject: Transco Raritan Bay and Lower NY Bay Sediment Study

Mr. Hogan,
Please find attached a response to the 2nd NOIA issued by NYSDEC on June 2, 2016 regarding the sediment sampling activities associated with Transco's Northeast Supply Enhancement Project.

A hard copy of this document will be sent to your office early next week.

Sincerely,
Steve



Steven MacLeod, *Environmental Scientist*
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smacleod@ene.com • www.ene.com

MacLeod, Steven

From: Barnes, Debra (DEC) <debra.barnes@dec.ny.gov>
Sent: Friday, October 14, 2016 8:47 AM
To: MacLeod, Steven
Cc: Carden, Wade E (DEC); Horner, Scott (Scott.Horner@williams.com); Mochrie, Sara; Fronckowiak, Megan L.
Subject: RE: NESE benthic sampling

Categories: Agency Communication

Steve,

Your email accurately captures our telephone conversation regarding benthic sampling in Raritan Bay. After the sampling has been completed, we can determine if any further site specific sampling would be needed to quantify the population distribution of hard clams along the proposed project route.

Thank you.

Debbie

From: MacLeod, Steven [mailto:SMacLeod@ene.com]
Sent: Thursday, October 13, 2016 5:34 PM
To: Barnes, Debra (DEC) <debra.barnes@dec.ny.gov>
Cc: Carden, Wade E (DEC) <wade.carden@dec.ny.gov>; Horner, Scott (Scott.Horner@williams.com) <Scott.Horner@williams.com>; SMochrie@ene.com; Fronckowiak, Megan L. <MFronckowiak@ene.com>
Subject: NESE benthic sampling

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Hi Debbie,

Expanding on the Sampling and Analysis Plan (SAP/QAPP v0.2) for the Northeast Supply Enhancement (NESE) Project, Transco may collect additional sediment samples in the NY waters near Staten Island that have previously been part of NYSDEC's hard clam transplantation program.

Per our conversation last Friday (10/7), You indicated that sediment grab samples (Smith-Mcintyre or equivalent) would be sufficient for providing a general comparison of shellfish densities along different NESE Project route alternatives through this special-permit harvest area.

You noted that the type of hydraulic clam dredge survey conducted by NJDEP is more robust than surface sediment grab samples, accounting for patchiness of shellfish beds, but you suggested that we just stick with the grab method proposed in the SAP/QAPP at this stage.

Please confirm that this accurately reflects your input on the matter.

Thanks,
Steve



Steven MacLeod, *Environmental Scientist*
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MacLeod, Steven

From: Hogan, Chris M (DEC) <chris.hogan@dec.ny.gov>
Sent: Thursday, November 10, 2016 3:18 PM
To: MacLeod, Steven
Subject: RE: Transco Sediment Study Update (USACE File No. NAN-2016-00908-EHA)

Categories: Agency Communication

Steve- Below our the comments from our Sediment Management Unit in DOW.

We have reviewed the proposed Enclosure 3 NESE SAP Test Tables and have the following comments. No sites in NY should have analyses only for the 22 PCB congeners unless the results will be multiplied by 2. It would be preferable if sites in NY waters are analyzed for PCB aroclors as well as the 22 congeners. All the metals analyzed by method 6010 b (which includes all the metals listed below) should also be reported in NY. In addition, some sediment at each NY sample location should be archived for future dioxin/furan analysis if necessary.

In the E+E letter dated September 20, 2016, which was a response to our comment letter, is an incorrect interpretation of our comment. Our comment stated that sediment core samples should be collected to the depth of disturbance for the cable/pipeline installations and not to two feet below the bottom of the potential trench. The Response was:

Response: We conservatively interpret this statement to mean vibracore samples should be collected at least two feet below the bottom of the potential trench or to the maximum depth of disturbance, whichever is greater. Transco will collect vibracore samples to depths that sufficiently reflect the maximum anticipated depth of disturbance. These sample depths are reflected in Appendix B, Tables 1 and 2 of the SAP/QAPP, except that the vibracore depths at the anticipated HDD entry/exit points (VC 1, VC 54 and VC 55) will be increased from 12 feet to 20 feet.

The goal of our comment is to assure that the collected samples accurately define the material to be disturbed and to preclude potentially diluting any results with underlying glacial till or non-contaminated material. Therefore, core samples should only be collected to a depth that will characterize the material to be disturbed and not two feet below the bottom of the potential trench - unless that material two feet below will definitely be disturbed by the installation. The sampling plan proposed to divide each core into three foot increments (page 36) which should be acceptable for defining the material. However, to reiterate our original comment on the plan, if there is any deviation from this proposed segmentation of the cores, DEC must be notified and consulted first. Based on the approved plan each core will be segmented and no core will be completely homogenized

Aluminum
Antimony
Arsenic
Barium
Beryllium
Boron
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron

Lead
Lithium
Magnesium
Manganese
Mercury
Molybdenum
Nickel
Phosphorus
Potassium
Selenium
Silica (SiO)
Silver
Sodium
Strontium
Thallium
Tin
Titanium
Vanadium
Zinc

From: MacLeod, Steven [mailto:SMacLeod@ene.com]
Sent: Friday, November 04, 2016 8:39 PM
To: 'Naomi Handell' (Naomi.J.Handell@usace.army.mil) <Naomi.J.Handell@usace.army.mil>; Hogan, Chris M (DEC) <chris.hogan@dec.ny.gov>; Maraglio, Matthew (DOS) <Matthew.Maraglio@dos.ny.gov>; Allan Zaretsky (DCP) <AZARETSKY@planning.nyc.gov>
Cc: Mendez, Rosa E (DOS) <Rosa.Mendez@dos.ny.gov>; deQuillfeldt, Charles none (DEC) <charles.dequillfeldt@dec.ny.gov>; Barnes, Debra (DEC) <debra.barnes@dec.ny.gov>; MMarrel@planning.nyc.gov; Woodfield, Karen (DEC) <karen.woodfield@dec.ny.gov>; English, Diane M (DEC) <diane.english@dec.ny.gov>; Horner, Scott (Scott.Horner@williams.com) <Scott.Horner@williams.com>; Clements, Blake <Blake.Clements@williams.com>; SMochrie@ene.com; Eakin,Megan <MEakin@ene.com>; Martinez, Chris <Chris.Martinez@williams.com>
Subject: Transco Sediment Study Update (USACE File No. NAN-2016-00908-EHA)

ATTENTION: This email came from an external source. Do not open attachments or click on links from unknown senders or unexpected emails.

Greetings,

This message is a courtesy update on the offshore sediment study that Transco is conducting in support of the Northeast Supply Enhancement Project. Several sample site coordinates in New York and New Jersey have been adjusted since July to account for further engineering design analysis and the results of Phase I underwater archaeological surveys for all sample sites. The current sample sites are identified in the attached tables (Enclosure 1) and figures (Enclosure 2), which are revisions to the tables and figures that were submitted on July 29, 2016. Transco began collecting core boring samples in New Jersey waters in early October; Transco is awaiting additional New Jersey agency approval and suitable weather windows to complete the core boring activities. Transco anticipates collecting vibracore samples starting November 14th, or soon thereafter.

In addition to adjustments to previous site coordinates, new sites have been added. In particular, Transco has added vibracore sample sites VC70 through VC87 (in New York waters). These sites were added in response to a request from the New York State Department of Environmental Conservation (Bureau of Marine Resources) to consider an alternative to the currently preferred route. Further, Transco has determined that up to five additional core borings may be necessary in or near Ambrose Channel (in New Jersey waters) to fully inform the HDD engineering analysis. These potential core boring sites are identified as BHA 21 through BHA 25-ALT in the attached table and drawings.

The historic preservation offices (HPO) in New York and New Jersey have been notified of the Phase I archaeological survey results, and concurrence has been obtained as necessary.

Please note that since July 2016 Transco has also adjusted the list of sediment chemistry analyses that will be performed at vibracoring sites. These adjustments were made to account for guidance from the New Jersey Department of Environmental Protection (Office of Dredging and Sediment Technology) and information in the April 2016 U.S. Army Corps of Engineers (New York District) document titled *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal.* Attached are two tables listing the currently planned chemical analyses; the applicable vibracore sample sites are listed at the top of each table (Enclosure 3).

Please feel free to contact me or Sara Mochrie at 716-684-8060 if you have any questions about this message.

Sincerely,
Steve MacLeod

ecology and environment, inc.

Steven MacLeod, *Environmental Scientist*
368 Pleasant View Drive, Lancaster, NY 14086
Office Phone: 716-684-8060 x3907 • Work Cell: 716-462-0845
smacleod@ene.com • www.ene.com

MacLeod, Steven

From: Hogan, Chris M (DEC) <chris.hogan@dec.ny.gov>
Sent: Tuesday, November 15, 2016 9:07 AM
To: MacLeod, Steven; Mochrie, Sara
Subject: Responses

Categories: Agency Communication

See responses. Sorry for the delay.

1. In NY waters where clamshell dredging will not occur, we plan to collect one sample per vibracore from the 3-foot interval where dioxins are most likely be present, as determined by the onboard geologist. Samples with substantial silt/clay content will be analyzed immediately for dioxins, while samples with little or no silt/clay will be archived. ***If you meant for us to archive a dioxin sample for each 3-foot vibracore interval (e.g., 4 samples for each 12-ft vibracore), please advise.*** Please archive a sample from each segment. If there is high dioxin found in the part of the core that is analyzed, the archived segments can then be analyzed to determine the extent of the contamination. If nothing shows up in the first analysis, the archived segments can be discarded.
2. We assume that you want the dioxin sample archived approximately 4 to 6 months - long enough for NYSDEC to receive and review the other sediment chemistry results, and determine if the follow-up dioxin testing is warranted. ***If you had a different time period in mind, please advise.*** We agree with the proposed timeframe and the rationale behind it.
3. We intend to test for metals using method 6020A rather than 6010C (the latest update to method 6010B). We understand that 6020A is better at detecting certain analytes in marine sediment that are subject to interference by 6010B/C. ***If NYSDEC prefers 6010C, please explain the reason.*** Method 6020A is acceptable.
4. We propose to exclude the following four analytes from the "metals" list that you provided because they are problematic:
   Boron, Lithium, Phosphorous, and Silica.
   In particular, the laboratory currently under contract is not certified in New York to analyze these four analytes using method 6010C. Further, it does not appear there are any ecological screening criteria in New York State for these analytes. ***Please advise if (and how) these analytes are critical to NYSDEC evaluation of the Project.*** We agree that boron, lithium, phosphorous, and silica can be deleted from the list of analytes.

Christopher M. Hogan
Chief, Major Project Management Unit
Department of Environmental Conservation
Division of Environmental Permits
625 Broadway, 4th Floor
Albany, NY 12233-1750
(518) 402-9151
chris.hogan@dec.ny.gov

E

Complete Sediment Chemistry Data Tables and Grain Size Curves

This appendix is provided separately on DVD.

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F

Sediment Chemistry Laboratory Reports

This appendix is provided separately on DVD.

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G Complete Water Quality Data Tables

This appendix is provided separately on DVD.

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H Benthic Laboratory Results

This appendix is provided separately on DVD.

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Data Usability Summary Reports

This appendix is provided separately on DVD.

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J

Grain Size Curves

This appendix is provided separately on DVD.

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TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC

NORTHEAST SUPPLY ENHANCEMENT PROJECT

ENVIRONMENTAL REPORT

J-2. ERRATA AND ADDENDUM

NOVEMBER 2026

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Fall/Winter 2016 Offshore Environmental Sampling Report for the Northeast Supply Enhancement Project

NEW JERSEY, NEW YORK

Errata Sheets

August 2017

Prepared for:

Transcontinental Gas Pipe Line Company, LLC

Prepared by:

ECOLOGY AND ENVIRONMENT, INC.
368 Pleasant View Drive
Lancaster, New York 14086

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B-10 Sample Sites and Samples where Metals Exceeded Class A and/or Class C Thresholds in the Alternative Group..... 3-3

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1 Introduction

Transcontinental Gas Pipe Line Company, LLC. (Transco), an indirect wholly owned subsidiary of Williams Partners L.P. (Williams), is proposing the Northeast Supply Enhancement (NESE) Project (Project) to expand its existing interstate natural gas pipeline system in Pennsylvania and New Jersey and its existing offshore natural gas pipeline system in New Jersey and New York waters. The Project capacity is fully subscribed by two entities of National Grid: Brooklyn Union Gas Company (d/b/a [doing business as] National Grid NY) and KeySpan Gas East Corporation (d/b/a National Grid), collectively referred to herein as "National Grid."

To provide the incremental 400,000 dekatherms per day (Dth/d) of capacity, Transco plans to expand portions of its system from the existing Compressor Station 195 in York County, Pennsylvania, to the Rockaway Transfer Point in New York State waters. As defined in executed precedent agreements with National Grid, the Rockaway Transfer Point is the interconnection point between Transco's existing Lower New York Bay Lateral (LNYBL) and existing offshore Rockaway Delivery Lateral (RDL).

Transco filed its application for a Certificate of Public Convenience and Necessity with the Federal Energy Regulatory Commission (FERC) on March 27, 2017 (Certificate Application), and FERC assigned the Project Docket No. CP17-101. On June 27, 2017, Transco submitted its Offshore Environmental Sampling Report as Appendix J of Transco's Joint Permit Application to the U.S. Army Corp of Engineers (USACE) and New York State Department of Environmental Conservation. Transco submitted its Offshore Environmental Sampling Report to FERC on June 30, 2017.

The following information consists of corrections to information initially presented in the Offshore Environmental Sampling Report. Please refer to the Offshore Environmental Sampling Report for the unchanged information. All revised information is listed in **red bold text** and removed features marked as ~~**strike through text**~~. The following items have corrections and are included in this document:

- Text for pesticides in Group B;
- Text for PCB Aroclors along the Alternative Route;

- Figures A-6, A-7, A-8, A-17, and A-18; and
- Tables B-5 and B-10.

2 Sediment Chemistry Results

2.3.2 Group B – New York, Page 1-10

Pesticides

Possible Clamshell Dredge Sample Sites

Of the pesticides tested for in samples from sample sites where clamshell dredging may occur, 12 have available Class A and/or Class C thresholds. A total of 8 pesticides (aldrin, endosulfan sulfate, endrin, O,P'-DDD, O,P'-DDE, P,P'-DDD, P,P'-DDE, and P,P'-DDT) were detected in at least one sample in Group B. (See Table B-7 for a list of the sample sites where pesticides were detected.) Of the detected pesticides, only endrin and the sum of DDT + DDE + DDD have available Class A and Class C thresholds, and only the sum of DDT + DDE + DDD exceeded the Class A threshold. The sum of DDT + DDE + DDD exceeded the Class A threshold in the 0- to 3-foot samples at VC6 and VC7; all samples from VC16 and VC17; and 0- to 3 foot **and 3- to 6 foot** sample at VC41-ALT. Concentrations that exceeded the Class A threshold ranged from ~~8.6~~ **3.9** µg/kg to 99.0 µg/kg.

2.3.5 Alternative Route, Page 1-21

Polychlorinated Biphenyls (PCBs)

Non-Clamshell Dredge Sample Sites

Three Aroclor compounds (Aroclor 1016, Aroclor 1254, and Aroclor 1260) were detected in at least one sample from the alternative route (see Table 2-11 for the sample sites where PCBs were detected). Class A thresholds were exceeded only for the sum of Aroclor compounds in the following samples: 0- to 3 feet at VC70 and VC76~~ and 9- to 12 feet at VC73 and VC74~~. Concentrations that exceeded the Class A threshold ranged from 300 µg/kg to 554 µg/kg.

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3 Tables

Table B-5 Sample Sites and Samples in Group A Exceeding ER-L and/or ER-M Thresholds for Metals

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded ER-L (mg/kg)	Exceeded ER-M (mg/kg)		
Arsenic	8.37-~~108~~66.6	~~N/A~~ 98.7-108.0	VC1-ALT	0-3 ft, ***3-6 ft,*** 6-9 ft, 9-12 ft, 12-15 ft, 15-16 ft
			VC1B-ALT	0-3 ft, ***3-6 ft,*** 6-9 ft, 9-12 ft, 12-15 ft
			VC2	0-3 ft, 6-9 ft
			VC3-ALT	0-3 ft, 3-6 ft, 6-9 ft, 9-10.5 ft
			VC4	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
Barium	--	82.5-135	VC1-ALT	***0-3 ft, 3-6 ft***
			VC1B-ALT	***0-3 ft, 3-6 ft***
Cadmium	1.62-2.36	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Chromium	97.6-145	N/A	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	0-3 ft, 3-6 ft
Cobalt	--	10.1-11.6	VC1-ALT	***3-6 ft, 12-15 ft, 15-16 ft***
			VC1B-ALT	***12-15 ft***
			VC3-ALT	***3-6 ft, 6-9 ft, 9-10.5 ft***
			VC4	***0-3 ft, 3-6 ft***
Copper	237-~~504~~ 256.0	~~N/A~~ 307.0-504.0	VC1-ALT	***0-3 ft,*** 3-6 ft
			VC1B-ALT	***0-3 ft***, 3-6 ft
Lead	47.4-~~235~~ 214.0	~~N/A~~ 235.0	VC1-ALT	0-3 ft, 3-6 ft
			VC1B-ALT	***0-3 ft***, 3-6 ft
			VC4	0-3 ft
Manganese	--	274-436	VC1-ALT	***3-6 ft, 6-9 ft, 9-12 ft***
			VC1B-ALT	***3-6 ft, 9-12 ft, 12-15 ft***
			VC3-ALT	***0-3 ft, 3-6 ft, 6-9 ft, 9-10.5 ft***
			VC4	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft***
Nickel	24.3-40.7	N/A	VC1-ALT	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16 ft
			VC1B-ALT	0-3 ft, 3-6 ft, 9-12 ft, 12-15 ft
			VC3-ALT	0-3 ft, 3-6 ft, 6-9 ft, 9-10.5 ft
			VC4	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
Selenium	--	1.22-18.6	VC1-ALT	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16 ft***
			VC1B-ALT	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft***
			VC2	***0-3 ft***
			VC3-ALT	***0-3 ft, 3-6 ft, 6-9 ft, 9-10.5 ft***
			VC4	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft***
Silver	3.06-4.58	N/A	VC1-ALT	0-3 ft, ***3-6 ft***
			VC1B-ALT	***0-3 ft,*** 3-6 ft
Vanadium	--	70.4-83.3	VC1-ALT	***3-6 f***
			VC1B-ALT	***3-6 ft***
Zinc	227-336	N/A	VC1-ALT	03- ft, 3-6 ft
			VC1B-ALT	03- ft, 3-6 ft
Mercury	0.294-0.32	2.53-5.28	VC1-ALT	***0-3 ft, 3-6 ft***
			VC1B-ALT	***0-3 ft, 3-6 ft,*** 6-9 ft
			VC4	0-3 ft

Key:
-- = ER-L thresholds were not available.
N/A = Detected concentrations did not exceed the ER-M thresholds.

* Sample depths where ER-M thresholds were exceeded are in bold and italics.

Table B-10 Sample Sites and Samples where Metals Exceeded Class A and/or Class C Thresholds in the Alternative Group

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded Class A (mg/kg)	Exceeded Class C (mg/kg)		
Arsenic	8.27-49.6	N/A	VC70	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC71	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC72	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC73-ALT	0-3 ft, 9-12 ft
			VC74	6-9 ft
			VC75	0-3 ft
			VC76	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC77	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, VC12-15.6 ft
			VC78	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, VC12-15 ft, VC15-17 ft
			VC79	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, VC12-16 ft
			VC80	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, VC12-15 ft, VC15-16.5 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, VC12-14.75 ft
			VC82	0-3 ft, 6-9 ft, 9-12 ft
Cadmium	1.23-3.36	N/A	VC70	0-3 ft
			VC76	0-3 ft
			VC80	3-6 ft, 6-9 ft
			VC81	3-6 ft, 6-9 ft, 9-12 ft
Chromium	85.2-198	N/A	VC70	0-3 ft
			VC76	0-3 ft
			VC77	0-3 ft
			VC80	3-6 ft, 6-9 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
Copper	36.4-230	N/A	VC70	0-3 ft
			VC75	0-3 ft
			VC76	0-3 ft
			VC77	0-3 ft
			VC78	0-3 ft
			VC80	0-3 ft, 3-6 ft, 6-9 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC82	0-3 ft
Lead	67.9-194	223	VC70	0-3 ft
			VC75	0-3 ft
			VC76	0-3 ft
			VC77	0-3 ft
			VC78	0-3 ft, 6-9 ft
			VC80	0-3 ft, ***3-6 ft***, 6-9 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC82	0-3 ft
Nickel	22.3-48.4	N/A	VC70	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC71	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC72	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC75	0-3 ft
			VC76	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
			VC77	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15.6 ft
			VC78	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-17 ft
			VC79-ALT	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-16 ft
			VC80	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16.5 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-14.75 ft
			VC82	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft, 12-16 ft

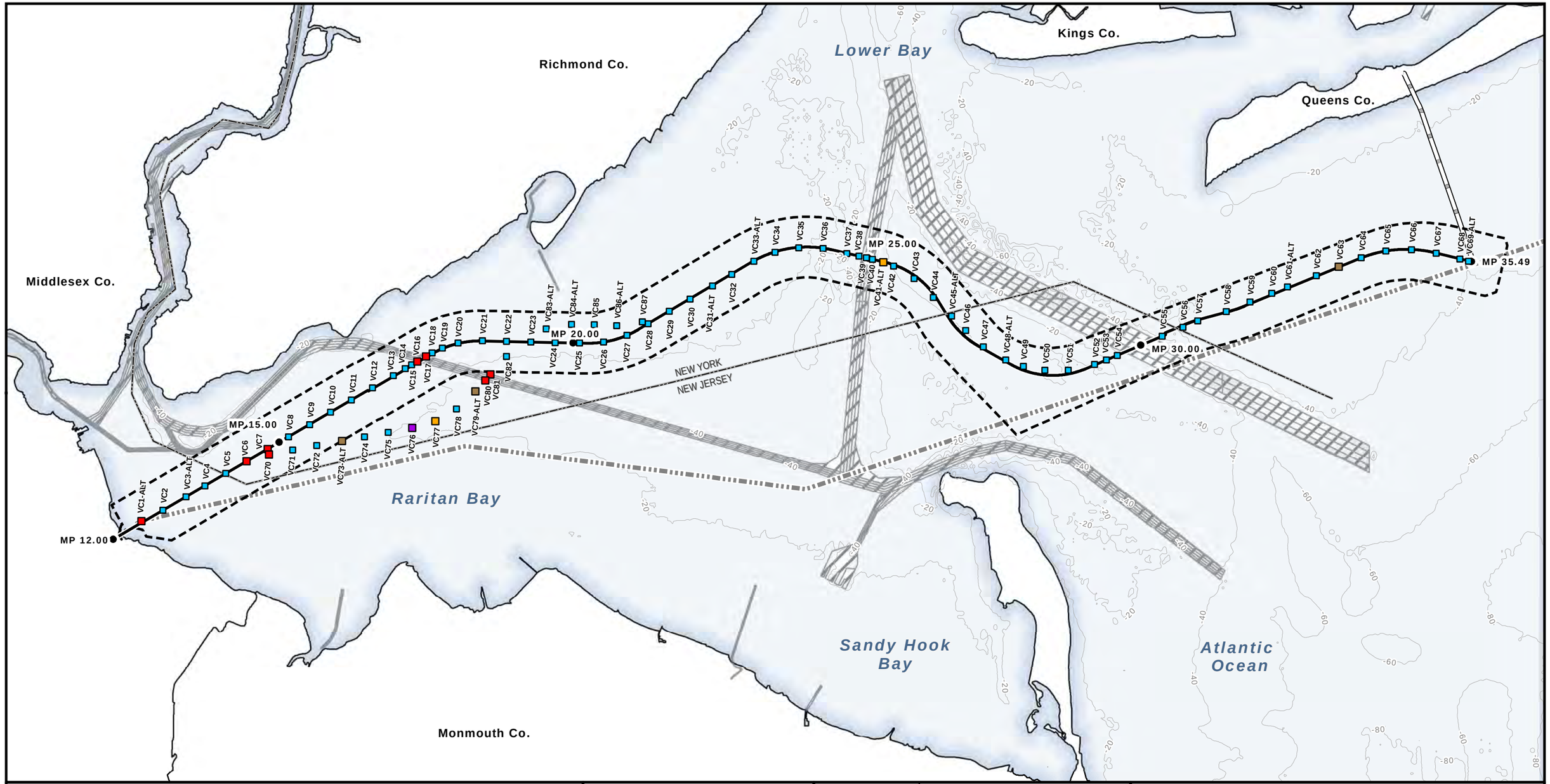
Table B-10 Sample Sites and Samples where Metals Exceeded Class A and/or Class C Thresholds in the Alternative Group

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded Class A (mg/kg)	Exceeded Class C (mg/kg)		
Silver	1.68-2.74	3.98-7.09	VC70	***0-3 ft***
			VC76	***0-3 ft***
			VC77	0-3 ft
			VC78	0-3 ft
			VC80	0-3 ft, ***3-6 ft, 6-9 ft***
			VC81	0-3 ft, ***3-6 ft, 6-9 ft, 9-12 ft***
Zinc	190-404	N/A	VC70	0-3 ft
			VC76	0-3 ft
			VC77	0-3 ft
			VC78	0-3 ft
			VC80	0-3 ft, 3-6 ft, 6-9 ft
			VC81	0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft
Mercury	0.191-0.337	**0.976**~~1.03~~ 3.49	VC70	***0-3 ft,*** 3-6 ft
			VC73-ALT	0-3 ft
			VC75	***0-3 ft***
			VC76	***0-3 ft***
			VC77	***0-3 ft,*** 3-6 ft
			VC78	***0-3 ft***
			VC79	0-3 ft
			VC80	***0-3 ft***
			VC81	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft,*** 12-14.75 ft
			VC82	***0-3 ft***

Key:
mg/kg = milligram per kilogram
* Sample depths where Class C thresholds were exceeded are in bold and italics.
N/A = Detected concentrations did not exceed the Class C thresholds.

4 Figures

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Legend

Pesticide Exceedances*

- 0
- 1
- 2
- 3
- 4

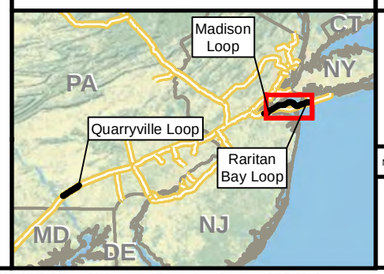
- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

*Number of analyte exceedances for lowest threshold of either the NJDEP Ecological Screening Criteria or NYSDEC Sediment Guidance Values.

N

0 1 Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE
	A-6

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE CONTAMINATION RESULTS
PESTICIDES
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
A	8/1/2017	MK		1000891		

DRAWN BY: MK	DATE: 8/1/2017	ISSUE FOR BID: N/A	SCALE: 1:95,000
CHECKED BY:	DATE:	ISSUE FOR CONSTRUCTION: N/A	Project features ver15
APPROVED BY:	DATE:	DRAWING NUMBER: Figure A-6	SHEET 1 OF 1
WO: 1000891		12:55 PM 8/1/2017	

Data Sources: Williams 2017; E&E 2017; ESRI 2012, 2017; NJDEP Digital Data Series Bathymetric Data 2006; NOAA ENC, Approach Scale, data downloaded 2016; USGS 2005; NYS Office of Information Technology Services GPO 2016.

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02:1000891.0020.05-Figure A-7 Pesticide Exceedances at Depth.ai-8/1/17

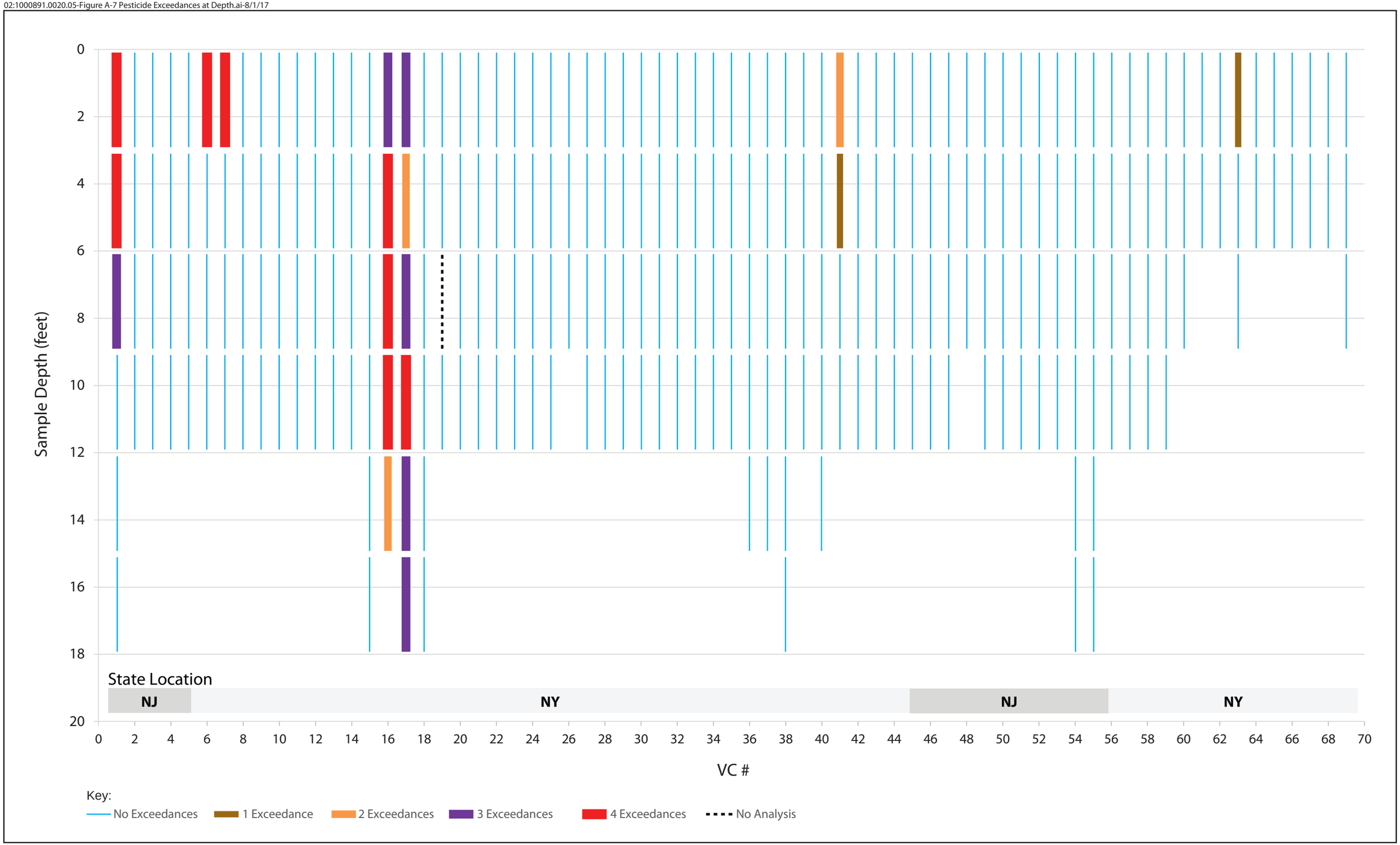
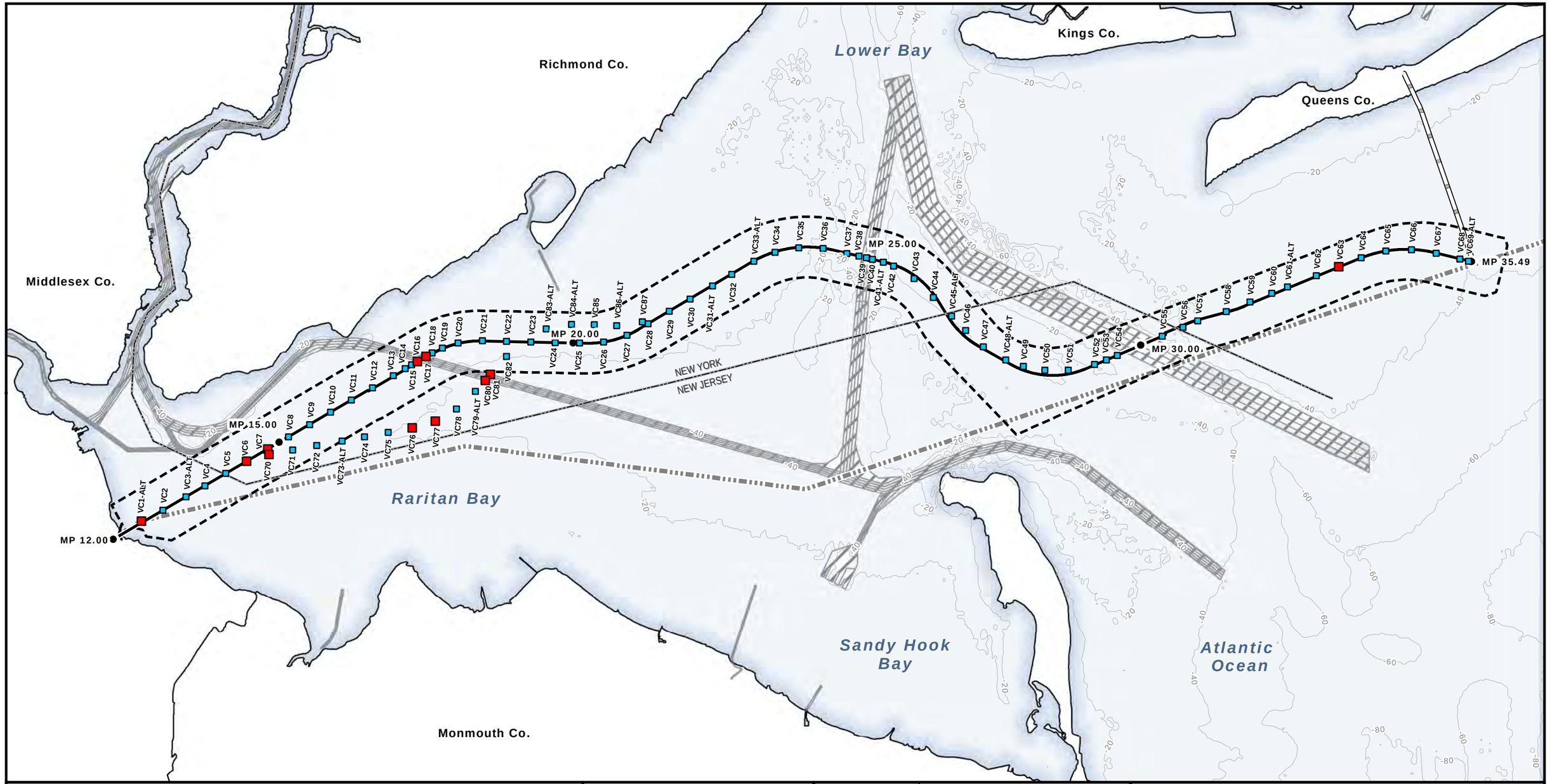


Figure A-7: Pesticide Exceedances at Depth Along the Preferred Route



Legend

PCB Aroclor Exceedances*

- 0
- 1
- 2 - 3

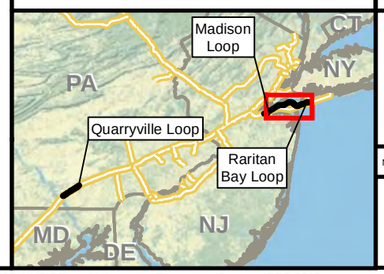
- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

*Number of analyte exceedances for lowest threshold of either the NJDEP Ecological Screening Criteria or NYSDEC Sediment Guidance Values.

N

0 1 Miles

SITE LOCATION



DRAWING NO.

REFERENCE TITLE

A-8

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE CONTAMINATION RESULTS
PCB AROCLORS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
A	8/1/2017	MK		1000891		

DRAWN BY: MK	DATE: 8/1/2017	ISSUE FOR BID: N/A	SCALE: 1:95,000
CHECKED BY:	DATE:	ISSUE FOR CONSTRUCTION: N/A	Project features ver15
APPROVED BY:	DATE:	DRAWING NUMBER: Figure A-8	SHEET 1 OF 1
WO: 1000891		12:52 PM 8/1/2017	

Data Sources: Williams 2017; E&E 2017; ESRI 2012, 2017; NJDEP Digital Data Series Bathymetric Data 2006; NOAA ENC, Approach Scale, data downloaded 2016; USGS 2005; NYS Office of Information Technology Services GPO 2016.

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02:1000891.0020.05-Figure A-17 Pesticide Exceedances at Depth-Alternative Route.ai-8/1/17

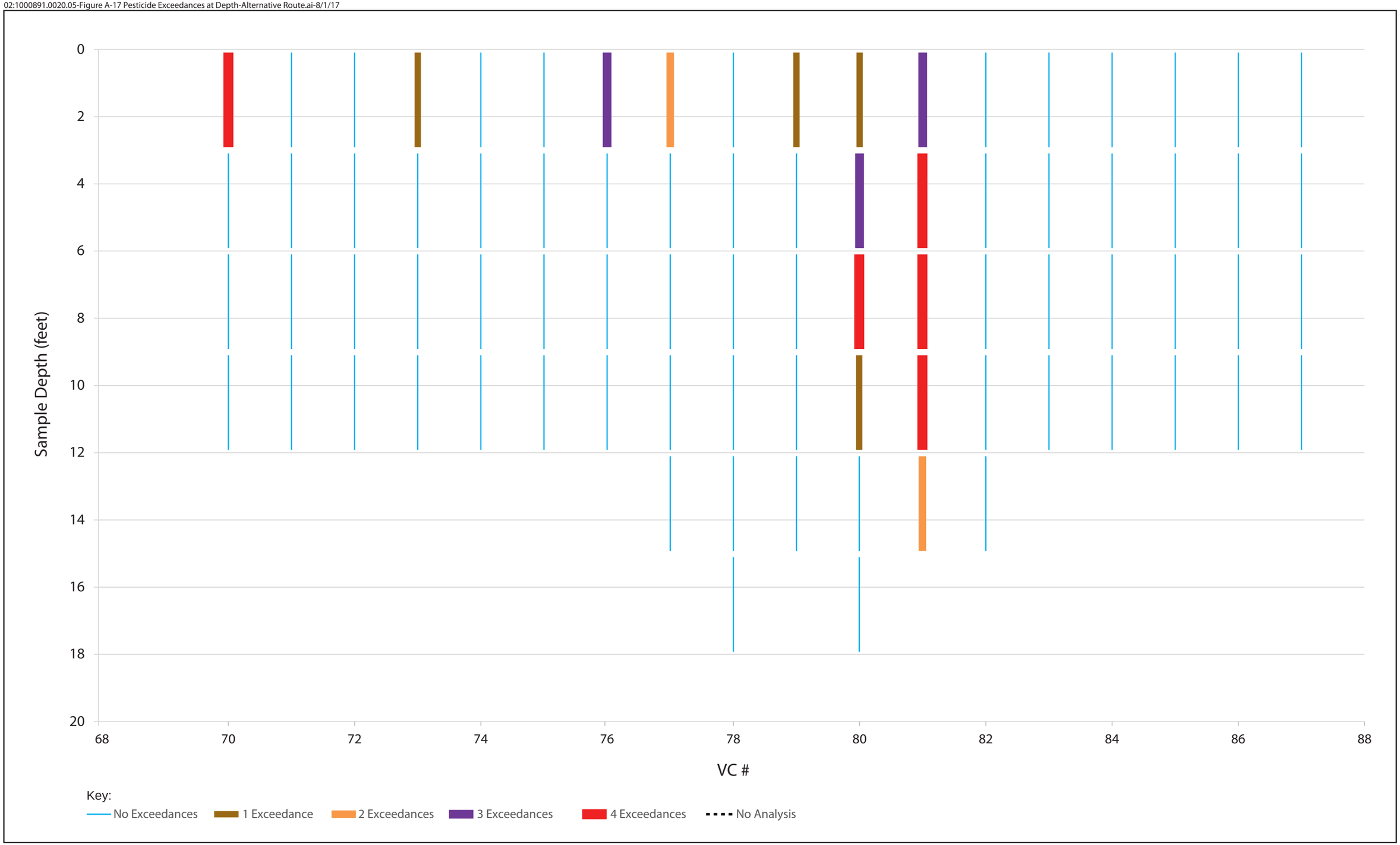


Figure A-17: Pesticide Exceedances at Depth Along Alternative Route

02:1000891.0020.05-Figure A-18 PCB Aroclor Exceedances at Depth-Alternative Route.ai-8/1/17

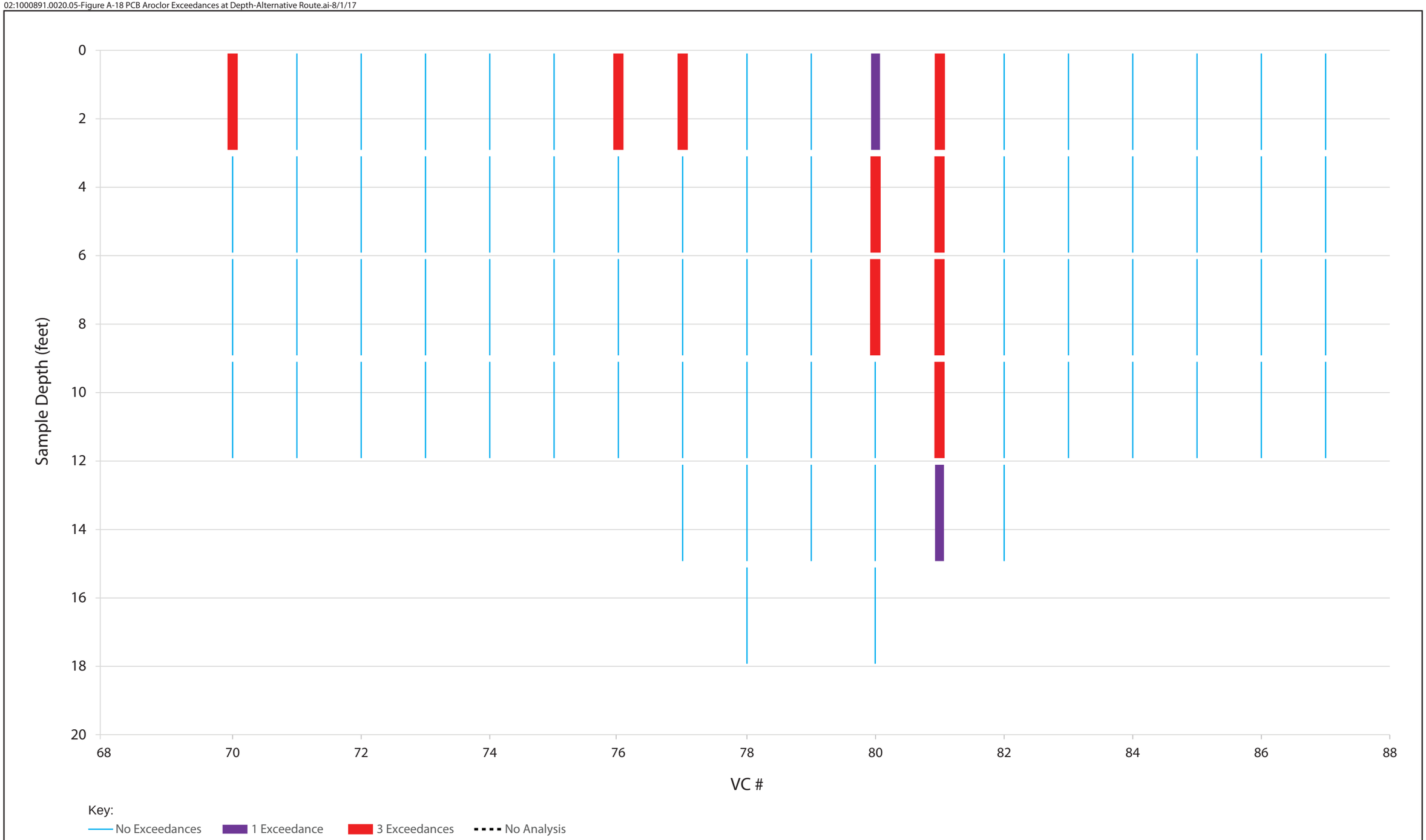


Figure A-18: PCB Aroclor Exceedances at Depth Along Alternative Route

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Addendum to the Fall/Winter 2016 Offshore Environmental Sampling Report for the Northeast Supply Enhancement Project New Jersey, New York

September 2017

Prepared for:

Transcontinental Gas Pipe Line Company, LLC

Prepared by:

ECOLOGY AND ENVIRONMENT, INC.
368 Pleasant View Drive
Lancaster, New York 14086

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List of Abbreviations and Acronyms

CFR	Code of Federal Regulations
COC	chemicals of Concern
ER-L	Effects Range Low (ER-L)
ER-M	Effects Range Median (ER-M)
E & E	Ecology and Environment, Inc.
EPA	U.S. Environmental Protection Agency
ESC	environmental screening criteria
ETRA	Environmental Toxicology and Risk Assessment
FERC	Federal Energy Regulatory Commission
HARS	historic remediation site
HDD	horizontal directional drill
NJDEP	New Jersey Department of Environmental Protection
NYCDEP	New York City Department of Environmental Protection
NYSDEC	New York State Department of Environmental Conservation
Project	Northeast Supply Enhancement Project
QA/QC	quality assurance/quality control
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan
SGVs	sediment guidance values
SRP	Site Remediation Program
TOGS	Technical and Operational Guidance Series
Transco	Transcontinental Gas Pipe Line Company, LLC
USACE	U.S. Army Corps of Engineers
U.S.C.	United States Code

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1 Introduction

1.1 Project Description

Transcontinental Gas Pipe Line Company, LLC (Transco), has filed an application with the Federal Energy Regulatory Commission (FERC) for a Certificate of Public Convenience and Necessity (Certificate) for the Northeast Supply Enhancement Project (Project) (Docket No. CP17-101-000). The Offshore Environmental Sampling Report was prepared to facilitate FERC's review and development of an environmental impact statement for the Project and to support the corresponding state-level environmental evaluation of the Project. On June 27, 2017, Transco submitted its Offshore Environmental Sampling Report as Appendix J of Transco's application for a U.S. Army Corps of Engineers (USACE) permit. On June 30, 2017, Transco submitted its Offshore Environmental Sampling Report to FERC. See the Offshore Environmental Sampling Report for a Project Description.

1.2 Scope of Work

Transco conducted a supplemental field survey and laboratory analysis in May 2017 to evaluate the physical and chemical characteristics of sediment at the proposed anode sled installation and to further evaluate chemical data along the Raritan Bay Loop route. The survey was conducted to support evaluation of the route for both engineering design and environmental review purposes. Laboratory analysis was completed for a focused list of contaminants, specifically metals, based on historical contamination documented in the area. Transco contracted Ecology and Environment, Inc. (E & E) to support the environmental permitting effort for the Project. The Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP) developed by E & E and reviewed by regulatory agencies for the previous offshore survey was used for this supplemental field survey. The SAP/QAPP prepared for the offshore component of the Project is provided in Appendix C of the Offshore Environmental Sampling Report. Agency comments on initial versions of the SAP/QAPP are provided in Appendix D of the Offshore Environmental Sampling Report. Transco contracted Alpine Ocean (a Gardline company) to conduct the survey and arrange for sediment testing at onshore laboratories.

Figure A-1 shows the locations of the sites sampled along the proposed Raritan Bay Loop route during the 2017 offshore study.

1.3 Previous Investigations

See the Offshore Environmental Sampling Report for a discussion of previous investigations.

1.4 Spring 2017 Survey Results

This report presents the results of sample collection and analyses supporting the sediment chemistry evaluation for the Project. Field data collected as part of the spring 2017 survey is included in the appendices. Appendix A contains figures referred to in the body of this report; Appendix B contains the tabularized laboratory results for all chemical parameters analyzed and grain size statistics; Appendix C contains the sediment chemistry laboratory reports; Appendix D contains the Data Usability Summary Reports; Appendix E contains grain size statistics; and Appendix F contains the previously unsubmitted core logs for samples collected in the 2016 survey. Supplemental vibracore logs from the May 2017 sampling will be provided in a Supplemental Information Filing anticipated to be submitted September 29, 2017.

2 Sediment Sampling

The SAP/QAPP developed to evaluate the site-specific sediment conditions along the proposed pipeline route was designed specifically to address chemicals of concern (COCs) identified by the New York State Department of Environmental Conservation (NYSDEC) and the New Jersey Department of Environmental Protection (NJDEP). These chemicals would be of concern if found above threshold levels. The threshold values indicate where there could be harmful effects to aquatic life.

2.1 Thresholds for Chemicals of Concern

As introduced in the Offshore Environmental Sampling Report, thresholds for COCs in sediments sampled in New York State waters were taken from the Technical and Operational Guidance Series (TOGS) 5.1.9 document, *In-Water and Riparian Management of Sediment and Dredged Material* (NYSDEC 2004) and the *Screening and Assessment of Contaminated Sediment* (NYSDEC 2014). Thresholds for COCs in sediments sampled in New Jersey waters were taken from *The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters* (NJDEP 1997). Additional guidance on COCs within the offshore Project area was taken from the USACE/U.S. Environmental Protection Agency's (EPA) *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal* (2016). These agency documents were developed to provide guidance on the statutory and regulatory requirements for dredging activities and to promote uniformity in the certification and/or permitting of dredging projects.

In New York, under TOGS 5.1.9, the NYSDEC Division of Water established three classes of sediment quality thresholds for dredged material (sediment guidance values [SGVs]). This system is based upon the concentrations of contaminants within the dredged sediments, in contrast to the EPA's system, which is based upon bioaccumulation and biotoxicity. The three classes of dredged sediment provided in TOGS 5.1.9 are Class A, B, and C, where Class A has no appreciable contamination and Class C has high contamination. See the Offshore Environmental Sampling Report for further discussion.

In New Jersey, the NJDEP's Site Remediation Program (SRP), Environmental Toxicology and Risk Assessment (ETRA) Unit, developed ecological screening criteria (ESC) for surface water, sediment, and soil. These ESCs were developed by the ERTA Unit using multiple sources, and the most conservative criterion for each contaminant was used. The ESC are presented in terms of Effects Range

Low (ER-L) and Effects Range Median (ER-M) levels for saline waters. The sediment and soil standards presented are not promulgated and are intended as screening values for ecological assessments.

The EPA's Marine Protection, Research, and Sanctuaries Act provides the criteria for disposal of dredged sediments in the ocean. Under 40 Code of Federal Regulations (CFR) 227, Criteria for the Evaluation of Permit Applications for Ocean Dumping of Materials, there are three conditions that qualify dredged materials for ocean dumping without further testing. Two of these conditions are applicable to dredging materials proposed for placement at offshore sites, i.e., the historic area remediation site (HARS). They are further discussed in the Offshore Environmental Sampling Report.

For materials that do not meet the conditions for ocean dumping without further testing, 40 CFR 227 prohibits ocean dumping of dredged materials containing anything greater than trace amounts of organohalogen compounds; mercury and mercury compounds; cadmium and cadmium compounds; oil of any kind or in any form; or known carcinogens, mutagens, or teratogens or any materials suspected of being such. Materials not excluded by this condition are subject to toxicity and bioaccumulation testing and must meet the conditions determined by the USACE/EPA guidance document. Based on the required testing and review by USACE/EPA, material may be approved for disposal at the HARS. A HARS plan must be reviewed and approved for this activity. Transco is considering disposal at the HARS as an option under consideration based on results from the offshore survey for management of dredged material during construction.

2.2 Sampling Methods

During the initial sampling effort, deep core samples (core boring) extending up to 125 feet below the seafloor were collected from nine sample sites, all of which are within New Jersey waters. These deep core sites are located near the New Jersey shoreline and across the Ambrose Channel where Transco proposes to install the pipeline using horizontal directional drill (HDD). The deep core depths represent the maximum depths for HDD installation. Initially, deep core samples were analyzed only for geotechnical properties for use in the engineering design of the HDDs. In 2017, core samples from deep core sites BHA7-ALT and BHA9-ALT were analyzed for metals. These two cores were included for laboratory analysis due to the documented historical contamination in the area. Table 2-1 notes the depths sampled for analysis of metals and the number of samples collected at these two deep-core sample sites.

Table 2-1 Deep Core Samples Include in Analysis

Sample Site [a]	State	Maximum Sample Depth (ft)[b]	No. Of Samples Collected
BHA7-ALT	***NJ***	***17***	***7***
BHA9-ALT	***NJ***	***17***	***6***

[a] Site in bold and italics indicate locations where clamshell dredging might occur.
[b] Depths provided represent actual recovery at each sample site.

Key:
ft = feet

As part of the spring 2017 effort, shallow core samples (vibracores) were collected at six sites in New Jersey waters, to a maximum depth of 12 feet below the seafloor (see Table 2-2). Five of the sample sites were located between two previously sampled sites (VC1-ALT and VC2) along the proposed route, and the sixth sample was located at the location where the anode sled will be installed (see Figure A-1 for the locations of the supplemental shallow core sample sites). The shallow core sample sites are identified in Table 2-2, and were reviewed and cleared by the New Jersey State Historic Preservation Office prior to sampling.

Table 2-2 Vibracore Sample Locations

Sample Site [a]	State	Coring Depth (ft)[c]	No. Off Samples Collected
VC88	***NJ***	***10***	***5[b]***
VC89	***NJ***	***10***	***5[b]***
VC90	***NJ***	***10***	***5[b]***
VC91	***NJ***	***9.6***	***3***
VC92	***NJ***	***10***	***5[b]***
VC93	***NJ***	***10***	***4***

[a] Site in bold and italics indicate locations where clamshell dredging might occur.
[b] A duplicate sample at one depth interval was collected at this site.
[c] Depths provided represent actual recovery at each sample site.

Key:
ft = feet

The shallow core samples were collected in areas where the pipeline would be installed by a clamshell dredge, where the HDD entry/exit pits would be excavated using a clamshell dredge, and where the anode sled would be installed.

Once retrieved, the sediment core soil types were classified according to the United Soil Classification System, and sediment samples were collected from the core and shipped to a laboratory for chemical analysis. Each core was separated into 3-foot sections for shallow core samples. Distinct layers more than 1-foot thick were separated as unique sub-samples subject to sediment chemistry analyses if they appeared contaminated, based on the field geologist's evaluation of appearance and/or odor. The minimum 1-foot thickness provided sufficient sam-

ple volume for all sediment chemistry analyses. A total of 27 samples were collected, as identified in Table 2-2. The tests performed, method, and quantities of samples collected at sample sites in New Jersey waters are summarized in Table 2-3. The list of chemical analyses performed is based upon the NYSDEC TOGS 5.1.9 and the NJDEP Site Remediation Program ER-L and Effects Range Median ER-M ecological screening criteria. A summary of the results are discussed below, and the complete analytical results are provided in Appendix C of the Offshore Environmental Sampling Report.

Table 2-3 Selection of Sediment Chemistry Tests for Metals Detected that Exceeded State Thresholds

Test Description	EPA Method	Unit	NYSDEC Required Method Detection Limits	NJDEP or USACE Required Reporting Limits[1]	NJDEP Effects Range Low (ER-L)[2]	NJDEP Effects Range-Median (ER-M)[3]	NYSDEC Class A	NYSDEC Class C
Arsenic	EPA 6020A	mg/Kg	3^	1	8.2	70	8.2	70
Barium	EPA 6020A	mg/Kg	--	40	--	48	--	--
Cadmium	EPA 6020A	mg/Kg	1	1	1.2	9.6	1.2	9.6
Chromium	EPA 6020A	mg/Kg	--	1	81	370	81	370
Cobalt	EPA 6020A	mg/Kg	--	10	--	10	--	--
Copper	EPA 6020A	mg/Kg	5^	1	34	270	34	270
Lead	EPA 6020A	mg/Kg	2^	0.6	47	218	47	220
Manganese	EPA 6020A	mg/Kg	--	3	--	260	--	--
Mercury	EPA 7474	mg/Kg	0.2^	0.1	0.15	0.71	0.15	0.71
Nickel	EPA 6020A	mg/Kg	--	1	21	52	21	52
Selenium	EPA 6020A	mg/Kg	--	1	--	1	--	--
Silver	EPA 6020A	mg/Kg	--	1	1	3.7	1	3.7
Vanadium	EPA 6020A	mg/Kg	--	10	--	57	--	--
Zinc	EPA 6020A	mg/Kg	--	4	150	410	150	410

Notes:

[1] Unshaded cells present reporting limits from USACE/EPA *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal* (2014). Shaded cells present reporting limits from *The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters* (1997). If both documents provide reporting limits, only the lowest (most restrictive) reporting limits are shown here.

[2] NJDEP Ecological Screening Criteria (2009)

[3] NYS TOGS 5.1.9 (2004) and NYS *Screening and Assessment of Contaminated Sediment* (2014).

Key:

-- = Guidance value not available

^ = If the value found is less than the method detection limit, the laboratory reporting limit is listed.

mg/Kg = milligrams per kilogram

Quality assurance/quality control (QA/QC) samples were collected in accordance with the SAP/QAPP (Appendix C of the Offshore Environmental Sampling Report). Analytical data have been validated for samples collected as part of this sampling effort. Field QC samples included, at a minimum, one sediment duplicate per matrix per 20 samples for each analysis. Field equipment blanks were used at a rate of one per equipment set per day. Equipment blanks were only used

on equipment sets that were subject to decontamination. Dedicated or disposable equipment did not require the use of equipment blanks.

The results of the chemistry analysis for metals in vibracore samples is presented below.

2.3 Sediment Chemistry Results

Each sediment sample was analyzed for 27 metals, including mercury (see Appendix C of the Offshore Environmental Sampling Report for a complete list of these metals). Of these metals, 16 have available ER-L and/or ER-M thresholds. Within this sample set, all of the 27 metals tested for were detected in the 2017 survey. Sediment samples from BHA7-ALT and BHA9-ALT were analyzed for 26 of the 27 metals introduced above; the holding time for mercury analysis had been exceeded and, therefore, the samples were not analyzed for mercury. Of these metals, 15 have available ER-L and/or ER-M thresholds. Table 2-4 lists the number of metals detected at each sample site. Thirteen of the detected metals exceeded available NJDEP thresholds. The metals detected within this sample set that exceeded ER-L and/or ER-M thresholds are presented in Table 2-5. Table 2-6 presents the number of exceedances of metals for thresholds from both New Jersey and New York at each sample site.

Table 2-4 Number of Metals Detected at Sample Sites

Sample Site	Number of Metals Detected
VC88	25
VC89	25
VC90	25
VC91	25
VC92	21
VC93	24
BHA7-ALT	26
BHA9-ALT	24

Table 2-5 Sample Sites and Samples Exceeding ER-L and/or ER-M Thresholds for Metals

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded ER-L (mgKkg)	Exceeded ER-M (mg/Kg)		
Arsenic	8.37-66.6	94.6-118	VC88	6-9 ft, 9-10 ft
			VC89	6-9 ft, 9-10 ft
			VC90	3-6 ft, 6-9 ft, 9-10 ft
			VC91	3-6 ft, 6-9.6 ft
			BHA7-ALT	0-2 ft, ***2-4 ft, 4-6 ft,*** 6-8 ft, 8-10 ft, 15-17 ft

Table 2-5 Sample Sites and Samples Exceeding ER-L and/or ER-M Thresholds for Metals

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded ER-L (mgKkg)	Exceeded ER-M (mg/Kg)		
Barium	--	49.6-84.2	BHA7-ALT	***0-2 ft, 2-4 ft, 4-6 ft, 6-8 ft***
Cadmium	1.55	N/A	BHA7-ALT	2-4 ft
Chromium	87.3-136	N/A	BHA7-ALT	2-4 ft, 4-6 ft
Cobalt	--	10.1-34.8	VC88	***9-10 ft***
			VC89	***9-10 ft***
			VC90	***9-10 ft***
			BHA7-ALT	***15-17 ft***
			BHA9-ALT	***6-8 ft, 8-10 ft, 10-12 ft***
Copper	138-145	294	BHA7-ALT	0-2 ft, ***2-4 ft***, 4-6 ft, 6-8 ft
Lead	104-163	N/A	BHA7-ALT	0-2 ft, 2-4 ft, 4-6 ft, 6-8 ft
Manganese	--	265-380	VC88	***6-9 ft, 9-10 ft***
			VC89	***6-9 ft, 9-10 ft***
			VC90	***6-9 ft, 9-10 ft***
			BHA7-ALT	***0-2 ft, 4-6 ft, 8-10 ft, 15-17 ft***
Nickel	22.2-40.7	N/A	VC88	6-9 ft, 9-10 ft
			VC89	6-9 ft, 9-10 ft
			VC90	6-9 ft, 9-10 ft
			VC91	6-9.6 ft
			BHA7-ALT	0-2 ft, 2-4 ft, 4-6 ft, 6-8 ft, 15-17 ft
			BHA9-ALT	8-10 ft
Selenium	--	1.19-18.6	VC88	***3-6 ft, 6-9 ft, 9-10 ft***
			VC89	***0-3 ft, 3-6 ft, 6-9 ft, 9-10 ft***
			VC90	***0-3 ft, 3-6 ft, 6-9 ft, 9-10 ft***
			VC91	***0-3 ft, 3-6 ft, 6-9.6 ft***
			VC92	***0-3 ft, 3-6 ft, 6-9 ft, 9-10 ft,***
			VC93	***3-6 ft***
			BHA7-ALT	***0-2 ft, 2-4 ft, 4-6 ft, 6-8 ft, 8-10 ft, 10-12 ft, 15-17 ft***
			BHA9-ALT	***6-8 ft, 8-10 ft, 10-12 ft***
Silver	1.86-2.44	3.87	BHA7-ALT	0-2 ft, ***2-4 ft***, 4-6 ft, 6-8 ft
Vanadium	--	57.1-66.2	BHA7-ALT	***2-4 ft, 4-6 ft, 6-8 ft***
Zinc	207-276	N/A	BHA7-ALT	0-2 ft, 2-4 ft, 4-6 ft, 6-8 ft

* Sample depths where ER-M thresholds were exceeded are in bold and italics.

Key:
-- = ER-L thresholds were not available.
N/A = Detected concentrations did not exceed the ER-M thresholds.

Table 2-6 Count of Exceedances in New York and New Jersey per Number of Available SGVs and ESCs

Analyte Group	VC 88		VC 89		VC 90		VC 91	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY
Metals[1]	2/16	1/9	5/16	2/9	5/16	2/9	5/16	2/9

Analyte Group	VC 92		VC93		BHA7-ALT		BHA9-ALT	
	NJ	NJ	NJ	NY	NJ	NY	NJ	NY
Metals[1]	1/16	1/16	1/16	2/9	13/15	8/8	3/15	1/8

Key:
[1] .Highlighted cells indicate that ER-M or Class C thresholds also were exceeded.

Figure A-2 shows the number of exceedances at each sample site along the preferred route for metals based upon the lowest available threshold of either New Jersey or New York. Figure A-3 shows the number of exceedances for metals at each depth sampled along the preferred route between 0 ft and 10 ft for shallow core samples and 0 ft and 17 ft for deep core samples based upon the lowest available threshold of either New Jersey or New York.

2.4 Sediment Grain Size Results

The vibracore sample collected from VC93 was analyzed for grain size. The field geologist determined the number of depth intervals included in this analysis. This determination was based upon the observed uniformity of sediment type within the vibracore. Grain size analysis of these samples was conducted by the geotechnical laboratory using the sieve-based particle size analysis methodology (ASTM D6913) and/or a hydrometer (ASTM D7928). Results of the sediment grain-size analysis are presented in Table 2-7. Sediment grain size curves for VC93 are presented in Appendix E.

Table 2-7 Sediment Grain Sizes from VC93

Sample Depth	% Gravel (#4 Sieve)	% Sand (#200 Sieve)	% Fines (< #200 Sieve)	Water Content (%)	USCS Symbol
1.0"	0.4	98.6	1.0	22.9	SP
1.0'	0.0	98.7	1.3	21.9	SP
2.0'	0.0	97.5	2.5	20.3	SP
3.0'	5.7	95.1	4.9	21.4	SP
4.0'	0.0	83.4	16.6	25.3	SM
5.0'	0.0	92.3	7.7	24.4	SP-SM
6.0'	0.0	80.7	19.3	22.9	SM
7.0'	0.0	95.9	4.1	26.7	SP
8.0'	0.0	92.9	7.1	24.2	SP-SM
9.0'	0.0	94.4	5.6	23.3	SP-SM
10.0'	0.0	95.3	4.7	21.5	SP

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3 References

New Jersey Department of Environmental Protection (NJDEP). 1997. The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters. October 1997. Trenton, New Jersey.

_____. 2009. Ecological Screening Criteria. NJDEP Site Remediation Program. http://www.nj.gov/dep/srp/guidance/ecoscreening/ Accessed February 1, 2017.

New York State Department of Environmental Conservation (NYSDEC). 2004. Technical & Operational Guidance Series (TOGS) 5.1.9: In-Water and Riparian Management of Sediment and Dredged Material. November 2004. Division of Water. Albany, New York.

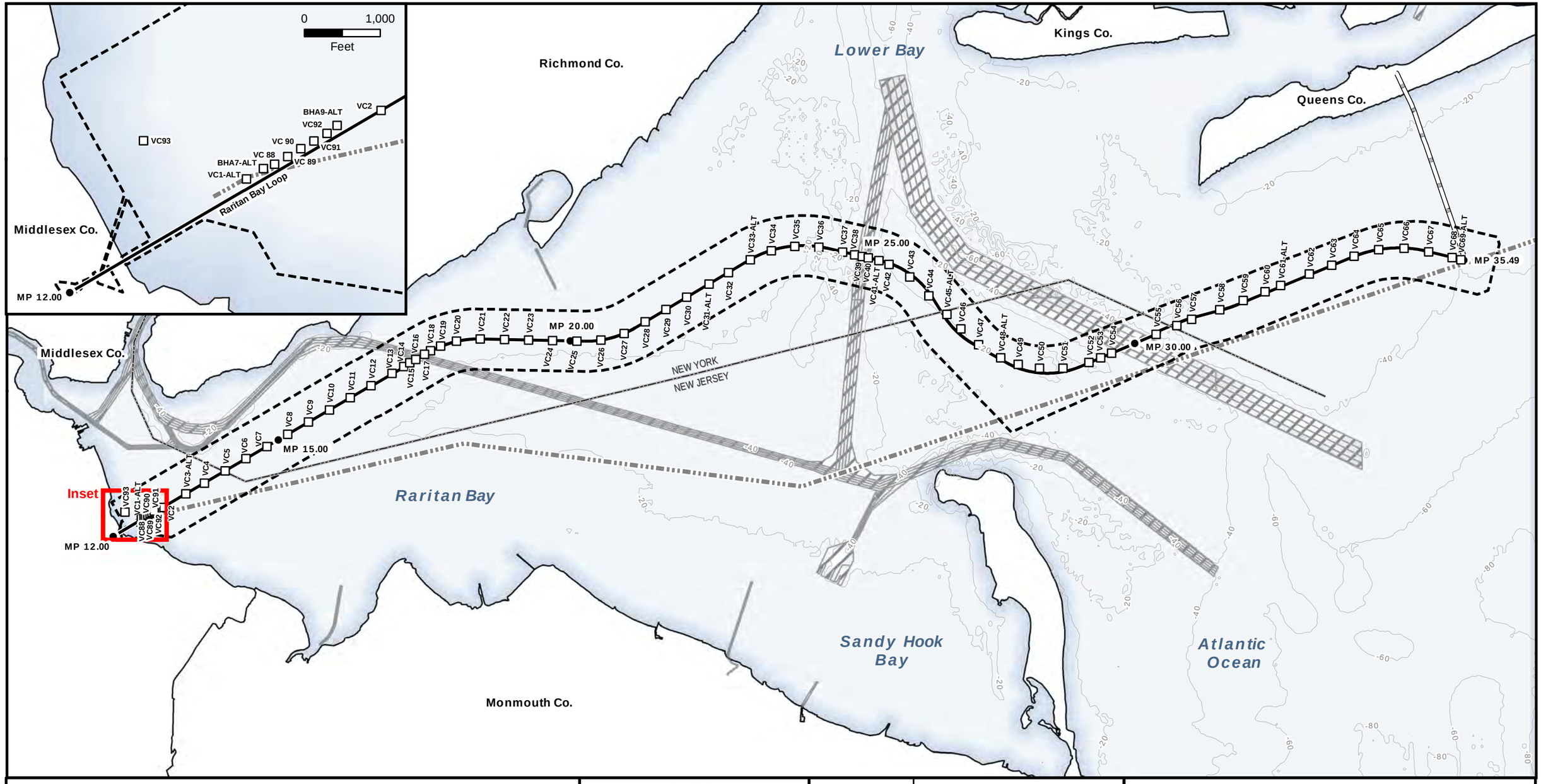
_____. 2014. Screening and Assessment of Contaminated Sediment. June 2014. Division of Fish, Wildlife and Marine Resources. Bureau of Habitat. Albany, New York.

U.S. Army Corp of Engineers (USACE), EPA. 2016. Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal. April 2016. USACE New York District. EPA Region 2. New York, New York.

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A Figures

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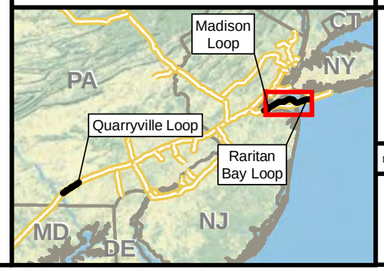
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- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

*Number of analyte exceedances for lowest threshold of either the NJDEP Ecological Screening Criteria or NYSDEC Sediment Guidance Values.

N

0 1 Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
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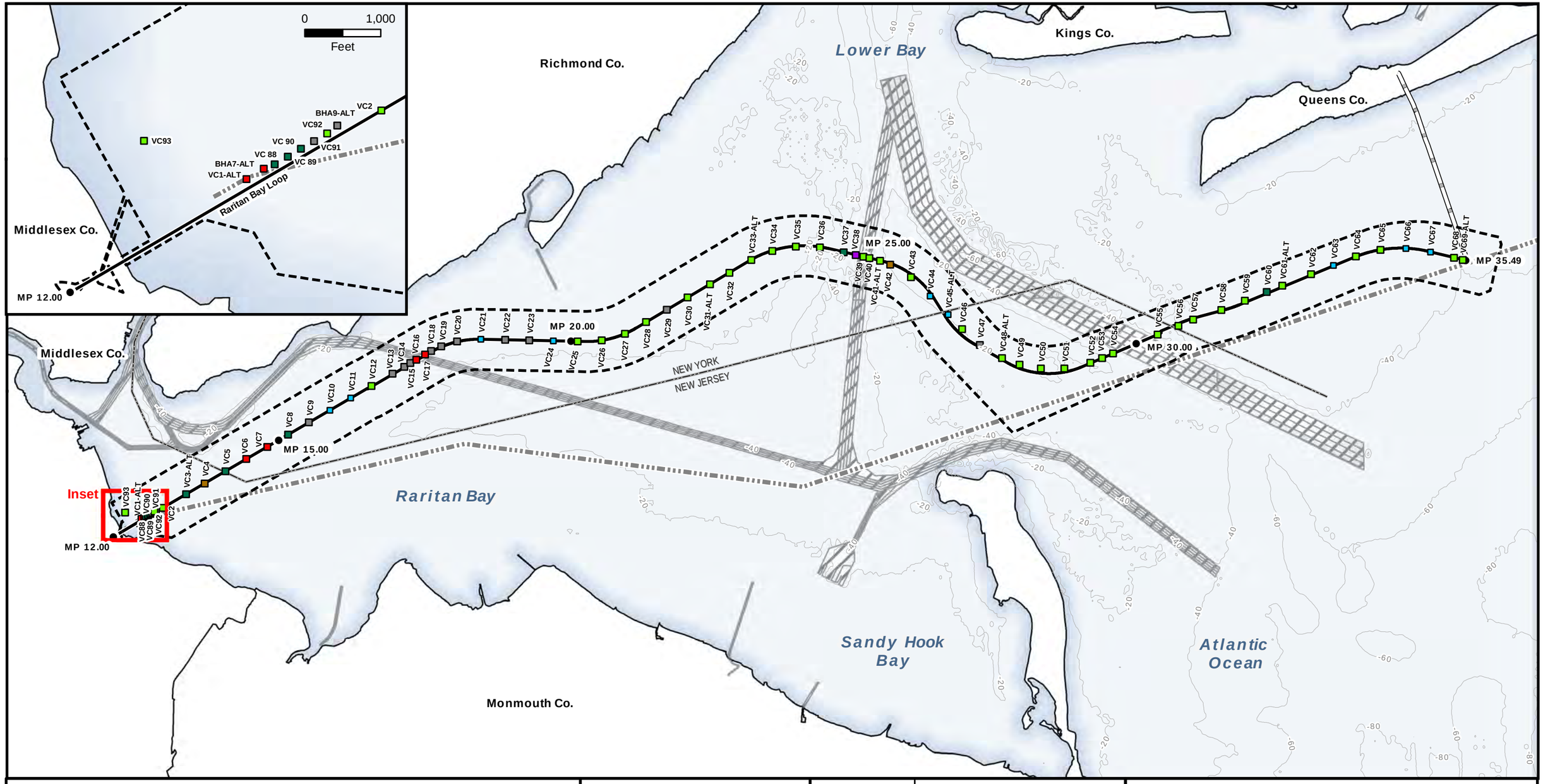
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE SITES
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

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CHECKED BY:	DATE:	ISSUE FOR CONSTRUCTION: N/A	Project features ver15
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Data Sources: Williams 2017; E&E 2017; ESRI 2012, 2017; NJDEP Digital Data Series Bathymetric Data 2006; NOAA ENC, Approach Scale, data downloaded 2016; USGS 2005; NYS Office of Information Technology Services GPO 2016.

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Legend

Metals Exceedances*

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- 1-2
- 3-4
- 5-6
- 7-8
- 11-12
- 13-15

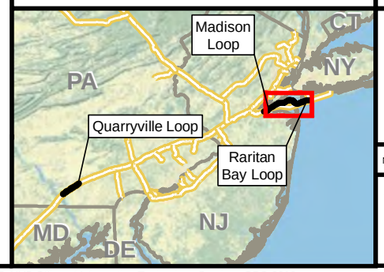
- Milepost
- Proposed Raritan Bay Loop
- Construction Workspace
- NY/NJ Boundary
- Lower NY Bay Lateral
- Rockaway Delivery Lateral
- Maintained Navigation Channel

*Number of analyte exceedances for lowest threshold of either the NJDEP Ecological Screening Criteria or NYSDEC Sediment Guidance Values.

N

0 1 Miles

SITE LOCATION



DRAWING NO.	REFERENCE TITLE

NO.	DATE	BY	REVISION DESCRIPTION	W.O. NO.	CHK.	APP.
A	8/22/2017	AL		1000891		

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
OFFSHORE SEDIMENT SAMPLE CONTAMINATION RESULTS
METALS EXCEEDANCES
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

DRAWN BY: AL	DATE: 8/22/2017	ISSUE FOR BID: N/A	SCALE: 1:95,000
CHECKED BY:	DATE:	ISSUE FOR CONSTRUCTION: N/A	Project features ver15
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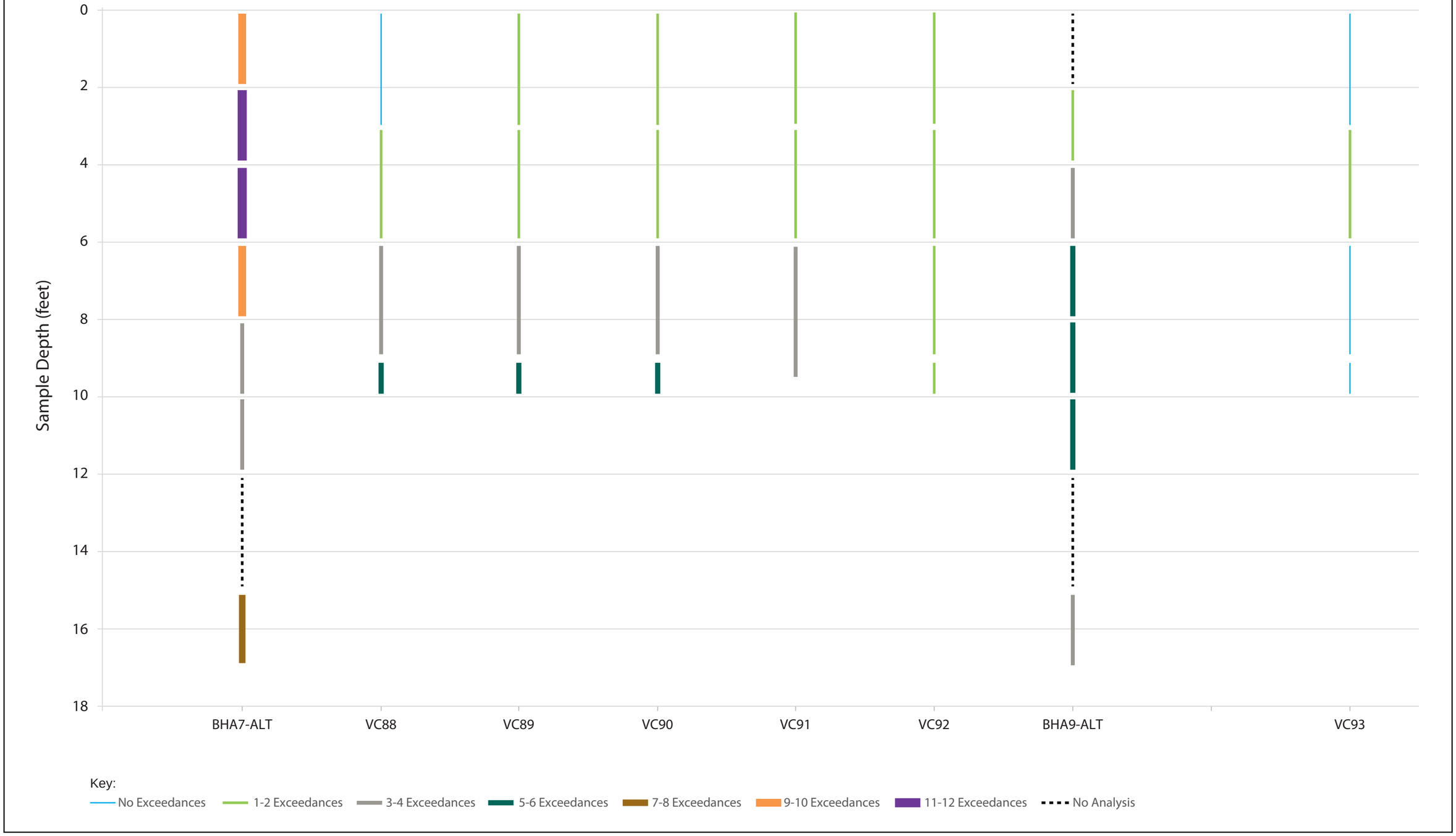


Figure A-3: Metals Exceedances at Depth

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B Complete Sediment Chemistry Data Tables

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Summary of Analytical Results for Additional New Jersey Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	Screening Criteria [1]	Location ID:	BHA 7-ALT	BHA 7-ALT	BHA 7-ALT	BHA 7-ALT	BHA 7-ALT	BHA 7-ALT	BHA 7-ALT	BHA 9-ALT	BHA 9-ALT	BHA 9-ALT	BHA 9-ALT	BHA 9-ALT
		Sample Name:	BHA-7 SS1 0-2	BHA-7 SS2 2-4	BHA-7 SS3 4-6	BHA-7 SS4 6-8	BHA-7 SS5 8-10	BHA-7 SS6 10-12	BHA-7 SS7 15-17	BHA-9 SS2 2-4	BHA-9 SS3 4-6	BHA-9 SS4 6-8	BHA-9 SS5 8-10	BHA-9 SS6 10-12
		Depth:	0 - 2 ft	2 - 4 ft	4 - 6 ft	6 - 8 ft	8 - 10 ft	10 - 12 ft	15 - 17 ft	2 - 4 ft	4 - 6 ft	6 - 8 ft	8 - 10 ft	10 - 12 ft
		Date:	10/08/16	10/08/16	10/08/16	10/08/16	10/08/16	10/08/16	10/08/16	10/08/16	10/08/16	10/08/16	10/08/16	10/08/16
Metals by Method SW-846 6020A (mg/kg)														
Aluminum	18000	**10200**	**12200**	**11200**	**10500**	**10200**	**4800**	**15700**	**4710**	**4460**	**1680**	**1950**	**1440**
Antimony	9.3	**6.80 J**	**8.32**	**7.15**	**6.19**	1.06 U	0.475 U	0.458 U	0.396 U	0.403 U	0.226 U	0.256 U	0.290 U
Arsenic	8.2	**39.1 J**	**118**	**94.6**	**60.6**	**12.6**	**7.46**	**15.1**	**6.05**	**6.27**	**1.13**	**1.89**	**1.64**
Barium	48	**50.9 J**	**84.2**	**58.6**	**49.6**	**22.2**	**11.5**	**28.5**	**10.6**	**9.50**	**18.8**	**16.8**	**8.82**
Beryllium	N/A	**0.870**	**1.09**	**0.784**	**0.767**	**0.722**	**0.373 J**	**1.09**	**0.298 J**	**0.318 J**	**0.730**	**0.781**	**0.983**
Cadmium	1.2	**1.00**	**1.55**	**0.676**	**0.94**	**0.248 J**	0.034 U	**0.426**	**0.109 J**	**0.099 J**	0.031 U	0.030 U	0.030 U
Calcium	N/A	**5040 J**	**5010**	**3000**	**3390**	**6100**	**4580**	**7750**	**4020**	**6760**	**272 J**	**253 J**	**397 J**
Chromium, Total	81	**69.3 J**	**136**	**87.3**	**78.4**	**28.9**	**12.8**	**37.9**	**15.2**	**13.4**	**5.16**	**5.81**	**5.72**
Cobalt	10	**9.22**	**9.17**	**8.15**	**8.34**	**7.00**	**3.56**	**11.1**	**3.72**	**3.46**	**11.8**	**19.7**	**34.8**
Copper	34	**140**	**294**	**145**	**138**	**19.0**	**7.65**	**18.8**	**9.88**	**7.42**	**11.1**	**8.38**	**11.0**
Iron	N/A	**32400**	**41900**	**42300**	**32000**	**29100**	**13900**	**42900**	**14600**	**13800**	**1660**	**887**	**2660**
Lead	47	**156**	**163**	**125**	**104**	**17.3**	**8.51**	**14.7**	**12.4**	**11.0**	**8.05**	**10.6**	**14.1**
Magnesium	N/A	**5490**	**6430**	**5680**	**5110**	**5650**	**2510**	**7550**	**2850**	**2860**	**415**	**482**	**424**
Manganese	260	**274 J**	**249**	**285**	**231**	**265**	**125**	**348**	**139**	**132**	**4.98**	**3.99**	**13.1**
Molybdenum	N/A	0.209 U	0.202 U	0.192 U	0.184 U	**2.10**	**0.663 J**	**4.95**	1.04 U	0.132 U	0.191 U	0.317 U	0.181 U
Nickel	21	**23.9**	**35.4**	**26.3**	**25.3**	**19.3**	**8.47**	**27.8**	**9.27**	**7.96**	**13.7**	**22.2**	**15.9**
Potassium	N/A	**2370**	**2810**	**2350**	**2240**	**2570**	**1100**	**3570**	**1290**	**1200**	**684**	**716**	**718**
Selenium	1	**3.36 J**	**7.22**	**4.24**	**4.44**	**1.88 J**	**1.19 J**	**2.27 J**	1.03 U	0.924 U	**3.01**	**3.78**	**2.61**
Silver	1	**2.44**	**3.87**	**1.86**	**1.93**	**0.152 J**	0.062 U	0.09 U	**0.122 J**	0.060 U	0.056 U	0.056 U	0.055 U
Sodium	N/A	**8960**	**11100**	**8510**	**7360**	**6430**	**3020**	**8180**	**3700**	**4070**	**1570**	**1870**	**1840**
Strontium	N/A	**51.5**	**66.2**	**41.9**	**38.2**	**39.8**	**30.5**	**50.1**	**24.4**	**37.8**	**6.58**	**7.18**	**6.47**
Thallium	N/A	**0.228 J**	**0.249 J**	**0.173 J**	**0.176 J**	**0.144 J**	0.066 U	**0.275 J**	0.070 U	0.063 U	**0.102 J**	**0.133 J**	**0.177 J**
Tin	N/A	**19.1**	**28.2**	**21.4**	**18.1**	0.183 U	**0.878 J**	1.55 U	1.01 U	0.876 U	0.624 U	0.478 U	0.583 U
Titanium	N/A	**362 J**	**472**	**503**	**455**	**203**	**104**	**245**	**113**	**116**	**18.1**	**21.6**	**31.2**
Vanadium	57	**46.9**	**66.2**	**62.8**	**57.1**	**30.6**	**18.5**	**44.2**	**17.2**	**17.1**	**13.1**	**13.6**	**17.2**
Zinc	150	**243**	**276**	**207**	**212**	**71.6**	**31.7**	**86.1**	**38.6**	**33.8**	**29.4**	**22.7**	**23.6**

Key:

Qualifiers

J = Estimated value

U = Not detected (method detection limit shown)

Other

mg/kg = milligrams per kilogram

N/A = No standard or guidance value available

Other:

Bold values denote a positive detection of the analyte.

Shaded values denote an exceedance of the screening criteria.

Notes:

1. NJDEP Ecological Screening Criteria (2009). Screening criteria are for sediment, saline water criteria, effects range low. If an effect range low value is not available, the effects range medium value was used. The effects range medium values are denoted with *.

Summary of Analytical Results for Additional New Jersey Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	Screening Criteria [1]	Location ID: BHA 9-ALT Sample Name: BHA-9 SS7 15-17 Depth: 15 - 17 ft Date: 10/08/16
Metals by Method SW-846 6020A (mg/kg)		
Aluminum	18000	**554**
Antimony	9.3	0.167 U
Arsenic	8.2	**1.40**
Barium	48	**3.40 J**
Beryllium	N/A	0.108 U
Cadmium	1.2	0.033 U
Calcium	N/A	**289 J**
Chromium, Total	81	**5.70**
Cobalt	10	**0.203 J**
Copper	34	**5.92**
Iron	N/A	**2010**
Lead	47	**3.95**
Magnesium	N/A	**202**
Manganese	260	**3.73**
Molybdenum	N/A	0.279 U
Nickel	21	**0.584 J**
Potassium	N/A	**321**
Selenium	1	0.936 U
Silver	1	0.060 U
Sodium	N/A	**986**
Strontium	N/A	**2.37**
Thallium	N/A	0.064 U
Tin	N/A	0.323 U
Titanium	N/A	**69.4**
Vanadium	57	**8.44**
Zinc	150	**4.33 J**

Key:

Qualifiers

J = Estimated value

U = Not detected (method detection limit shown)

Other

mg/kg = milligrams per kilogram

N/A = No standard or guidance value available

Other:

Bold values denote a positive detection of the analyte.

Shaded values denote an exceedance of the screening criteria.

Notes:

1. NJDEP Ecological Screening Criteria (2009). Screening criteria are for sediment, saline water criteria, effects range low. If an effect range low value is not available, the effects range medium value was used. The effects range medium values are denoted with *.

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range	NYSDEC No Appreciable Contamination Threshold Values	Location ID: / Sample Name: / Date: VC 88 DUP-D9-10E 5/19/17		VC 88 VC88-D0-3E 5/19/17		VC 88 VC88-D3-6E 5/19/17		VC 88 VC88-D6-9E 5/19/17		VC 88 VC88-D9-10E 5/19/17		VC 89 DUP-D0-3E 5/19/17		VC 89 VC89-D0-3E 5/19/17		VC 89 VC89-D3-6E 5/19/17		VC 89 VC89-D6-9E 5/19/17	
Metals by Method SW-846 6020A (mg/kg)																				
Aluminum	18000*	N/A	**15700**		**1150**		**3650**		**16100**		**16600**		**1500**		**1590**		**7700**		**15900**	
Antimony	9.3*	N/A	0.248	U	0.171	U	0.189	U	0.247	U	0.322	U	0.164	U	0.168	U	0.21	U	0.229	
Arsenic	8.2	8.2	**14**		**1.74**		**3.84**		**13.1**		**14.5**		**6.2**		**4.62**		**6.63**		**14.8**	
Barium	48*	N/A	**26.9**		2.12	U	**7.14**		**26.4**		**27.6**		1.75	U	2.4	U	**17.7**		**28.1**	
Beryllium	N/A	N/A	**0.974**		**0.112**	J	**0.26**	J	**1.05**		**1.05**		**0.216**	J	**0.187**	J	**0.575**		**0.997**	
Cadmium	1.2	1.2	**0.132**	J	0.033	U	**0.055**	J	**0.121**	J	**0.088**	J	0.032	U	0.033	U	**0.085**	J	**0.101**	
Calcium	N/A	N/A	**3070**		**4190**		**1660**		**2480**		**3000**		**773**		**747**		**1330**		**4000**	
Chromium, Total	81	81	**33.6**		**4.63**		**10.5**		**34.3**		**36.1**		**11.3**		**7.8**		**18.2**		**34.7**	
Cobalt	10*	N/A	**9.4**		**0.785**		**2.32**		**9.72**		**10.2**		**0.943**		**1.82**		**5.3**		**9.73**	
Copper	34	34	**13.3**		1.26	U	0.271	U	**13.5**		**14.3**		0.235	U	0.241	U	0.302	U	**14.2**	
Iron	N/A	N/A	**36100**		**6530**		**10600**		**37400**		**39100**		**13600**		**13000**		**21600**		**41500**	
Lead	47	47	**13.1**		**1.76**		**4.12**		**13.2**		**13.6**		**2.15**		**2.57**		**9.83**		**13.4**	
Magnesium	N/A	N/A	**7950**		**882**		**1970**		**8160**		**8390**		**776**		**880**		**3980**		**7830**	
Manganese	260*	N/A	**277**		**25.4**		**74.3**		**269**		**308**		**22.4**		**24.9**		**139**		**276**	
Molybdenum	N/A	N/A	**2.01**		**0.9**	J	**1.66**		**2.55**		**1.88**		**0.983**		**1.06**		**1.34**		**1.46**	
Nickel	21	21	**24.4**		**1.74**		**5.65**		**24.4**		**25.3**		**2.76**		**2.24**		**12.4**		**24.6**	
Potassium	N/A	N/A	**3530**		**295**		**848**		**3520**		**3710**		**293**		**350**		**1730**		**3410**	
Selenium	1*	N/A	**11.2**		0.958	U	**3.08**		**10.4**		**12.3**		**1.58**	J	**1.86**	J	**9.02**		**11.8**	
Silver	1	1	0.089	U	0.062	U	0.068	U	0.089	U	0.087	U	0.059	U	0.061	U	0.076	U	0.083	
Sodium	N/A	N/A	**7660**		**2190**		**2950**		**7400**		**7530**		**2130**		**2180**		**3880**		**5460**	
Strontium	N/A	N/A	**29**		**22.5**		**12.3**		**26.4**		**29.7**		**6.01**		**5.97**		**16.1**		**32.2**	
Thallium	N/A	N/A	**0.142**	J	0.065	U	0.072	U	**0.155**	J	**0.157**	J	0.063	U	**0.188**	J	**0.082**	J	**0.141**	
Tin	N/A	N/A	**0.515**	J	0.157	U	0.173	U	**0.495**	J	**0.568**	J	0.15	U	0.154	U	**0.493**	J	**0.548**	
Titanium	N/A	N/A	**290**		**60.9**		**118**		**278**		**303**		**44**		**65.5**		**160**		**290**	
Vanadium	57*	N/A	**38.9**		**9.49**		**15**		**40.4**		**39.9**		**19**		**20.3**		**27.4**		**39.7**	
Zinc	150	150	**72.1**		**5.74**	J	**17.6**		**74.7**		**76.4**		**10.3**	J	**9.74**	J	**39.9**		**75.7**	
Mercury by Method SW-846 7474 (mg/kg)																				
Mercury	0.15	0.15	**0.012**	J	0.002	U	**0.005**	J	**0.015**	J	**0.014**	J	0.002	U	0.002	U	**0.01**	J	**0.013**	

Key at end of table.

Source: Ecology and Environment, Inc. 2017

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range	NYSDEC No Appreciable Contamination Threshold Values	Location ID:		VC 89		VC 90		VC 90		VC 90		VC 90		VC 90		VC 91		VC 91	
			Sample Name:		VC89-D9-10E		DUP-D6-9E		VC90-D0-3E		VC90-D3-6E		VC90-D6-9E		VC90-D9-10E		VC91-D0-3E		VC91-D3-6E	
			Date:		5/19/17		5/19/17		5/19/17		5/19/17		5/19/17		5/19/17		5/19/17		5/19/17	
Metals by Method SW-846 6020A (mg/kg)																				
Aluminum	18000*	N/A			**17000**		**15300**		**3730**		**8420**		**15800**		**17000**		**2050**		**12500**	
Antimony	9.3*	N/A		U	0.25	U	0.236	U	0.16	U	0.186	U	0.238	U	0.243	U	0.154	U	0.215	U
Arsenic	8.2	8.2			**14**		**12.9**		**3.66**		**9.33**		**14.6**		**16.2**		**2.06**		**11.2**	
Barium	48*	N/A			**33.4**		**29.1**		**7.69**		**16.5**		**26.6**		**30.5**		**6.15**		**25.1**	
Beryllium	N/A	N/A			**1.02**		**0.99**		**0.289**	J	**0.636**		**1**		**1.04**		**0.201**	J	**0.84**	
Cadmium	1.2	1.2		J	**0.174**	J	**0.149**	J	**0.041**	J	**0.071**	J	**0.079**	J	**0.26**	J	**0.146**	J	**0.114**	J
Calcium	N/A	N/A			**3980**		**34100**		**11200**		**2950**		**2740**		**16500**		**260**	J	**11000**	
Chromium, Total	81	81			**37.4**		**34.2**		**9.58**		**21.2**		**34.1**		**38.1**		**8.18**		**27.7**	
Cobalt	10*	N/A			**10.1**		**8.94**		**2.82**		**5.59**		**9.42**		**10.1**		**1.41**		**7.6**	
Copper	34	34			**15.4**		**13.6**		0.23	U	0.266	U	**14.5**		**15.3**		**10.7**		**11.8**	
Iron	N/A	N/A			**52500**		**37200**		**11200**		**27300**		**38500**		**44200**		**7760**		**31700**	
Lead	47	47			**13.2**		**12.3**		**6.21**		**8.87**		**13.5**		**13.7**		**5.77**		**11.4**	
Magnesium	N/A	N/A			**7620**		**7320**		**1830**		**4880**		**7860**		**7880**		**797**		**6310**	
Manganese	260*	N/A			**291**		**282**		**102**		**158**		**275**		**375**		**25.7**		**227**	
Molybdenum	N/A	N/A			**1.91**		**2.29**		**0.587**	J	**2.63**		**1.3**	J	**3.81**		**0.243**	J	**1.56**	
Nickel	21	21			**27.2**		**23.8**		**5.73**		**13.7**		**24.7**		**27.4**		**3.5**		**18.9**	
Potassium	N/A	N/A			**3700**		**3490**		**771**		**1960**		**3500**		**3760**		**372**		**2800**	
Selenium	1*	N/A			**11.3**		**10.8**		**2.65**		**8.94**		**11.4**		**11.3**		**2.54**		**9.21**	
Silver	1	1		U	0.09	U	0.085	U	0.058	U	0.067	U	0.086	U	0.088	U	0.056	U	0.078	U
Sodium	N/A	N/A			**5570**		**6410**		**2610**		**4560**		**6100**		**6650**		**1760**		**5440**	
Strontium	N/A	N/A			**31.2**		**213**		**53.2**		**24**		**26.5**		**79.6**		**4.77**		**62.8**	
Thallium	N/A	N/A		J	**0.165**	J	**0.171**	J	0.061	U	**0.086**	J	**0.138**	J	**0.185**	J	**0.177**	J	**0.17**	J
Tin	N/A	N/A		J	**0.493**	J	**0.477**	J	**0.202**	J	**0.317**	J	**0.47**	J	**0.518**	J	0.141	U	**0.433**	J
Titanium	N/A	N/A			**328**		**343**		**101**		**183**		**272**		**335**		**43.9**		**233**	
Vanadium	57*	N/A			**41.6**		**38.3**		**14.3**		**34.8**		**38.2**		**41**		**17.1**		**31.4**	
Zinc	150	150			**82**		**69.8**		**18.5**		**47.1**		**76**		**77.1**		**13.3**		**58.7**	
Mercury by Method SW-846 7474 (mg/kg)																				
Mercury	0.15	0.15		J	**0.013**	J	**0.013**	J	**0.002**	J	**0.007**	J	**0.013**	J	**0.015**	J	0.002	U	**0.011**	J

Key at end of table.

Source: Ecology and Environment, Inc. 2017

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range	NYSDEC No Appreciable Contamination Threshold Values	Location ID: VC 91 Sample Name: VC91-D6-9.6E Date: 5/19/17		Location ID: VC 92 Sample Name: DUP-D3-6E Date: 5/19/17		Location ID: VC 92 Sample Name: VC92-D0-3E Date: 5/19/17		Location ID: VC 92 Sample Name: VC92-D3-6E Date: 5/19/17		Location ID: VC 92 Sample Name: VC92-D6-9E Date: 5/19/17		Location ID: VC 92 Sample Name: VC92-D9-10E Date: 5/19/17		Location ID: VC 93 Sample Name: VC93-D0-3E Date: 5/20/17	
Metals by Method SW-846 6020A (mg/kg)																
Aluminum	18000*	N/A	**16000**		**2800**		**1290**		**3950**		**4620**		**2430**		**928**	
Antimony	9.3*	N/A	0.235	U	0.17	U	0.177	U	0.169	U	0.264	U	0.176	U	0.404	U
Arsenic	8.2	8.2	**12.8**		**4.51**		**1.57**		**7.81**		**5.22**		**3.05**		**3.71**	
Barium	48*	N/A	**28.9**		**4.5**		3.17	U	**6.38**		**9.34**		**6.22**		**2.36**	J
Beryllium	N/A	N/A	**1.04**		**0.236**	J	0.114	U	**0.374**	J	**0.343**	J	**0.214**	J	**0.148**	J
Cadmium	1.2	1.2	**0.152**	J	**0.055**	J	0.035	U	**0.039**	J	**0.075**	J	**0.053**	J	**0.108**	J
Calcium	N/A	N/A	**3410**		**345**	J	**1510**		**916**		**794**		**324**	J	**252**	J
Chromium, Total	81	81	**35.1**		**11.1**		**4.36**		**12**		**12.9**		**7.27**		**7.7**	
Cobalt	10*	N/A	**9.79**		**1.57**		**1.13**		**2.78**		**2.61**		**1.34**		**1.92**	
Copper	34	34	**15**		0.244	U	1.44	U	0.243	U	0.261	U	0.252	U	**12.9**	
Iron	N/A	N/A	**39900**		**12000**		**4890**		**15900**		**14400**		**9800**		**6890**	
Lead	47	47	**13.2**		**2.78**		**3.22**		**4.57**		**5.2**		**3.66**		**11.9**	
Magnesium	N/A	N/A	**7540**		**1190**		**878**		**1970**		**1800**		**965**		**485**	
Manganese	260*	N/A	**260**		**38.4**		**44.3**		**72.9**		**95.1**		**85.7**		**31.3**	
Molybdenum	N/A	N/A	**1.88**		**0.796**	J	**0.198**	J	**1.01**		**1.28**		**1.02**	J	**0.701**	J
Nickel	21	21	**26.5**		**3.78**		**1.97**		**6.39**		**6.65**		**3.34**		**4.94**	
Potassium	N/A	N/A	**3530**		**624**		**367**		**835**		**964**		**548**		**219**	
Selenium	1*	N/A	**12.7**		**2.3**	J	**1.67**	J	**3.29**		**4.14**		**2.55**	J	**0.948**	J
Silver	1	1	0.085	U	0.061	U	0.064	U	0.061	U	0.066	U	0.063	U	**0.138**	J
Sodium	N/A	N/A	**6370**		**2200**		**2430**		**2740**		**2520**		**2360**		**1840**	
Strontium	N/A	N/A	**29.8**		**5.6**		**11.5**		**9.64**		**9.76**		**5.92**		**4.11**	
Thallium	N/A	N/A	**0.182**	J	0.065	U	0.068	U	0.065	U	**0.102**	J	0.067	U	0.063	U
Tin	N/A	N/A	**0.546**	J	0.156	U	0.162	U	0.155	U	**0.265**	J	0.161	U	**1.29**	J
Titanium	N/A	N/A	**290**		**71.2**		**65.6**		**101**		**109**		**75**		**54.9**	
Vanadium	57*	N/A	**39.8**		**15**		**5.99**		**20.3**		**17.6**		**10.2**		**12.3**	
Zinc	150	150	**74.7**		**14.6**		**7.8**	J	**25.2**		**20.5**		**11.5**	J	**42.9**	
Mercury by Method SW-846 7474 (mg/kg)																
Mercury	0.15	0.15	**0.013**	J	0.002	U	0.002	U	0.002	U	0.002	U	0.002	U	0.002	U

Key at end of table.

Source: Ecology and Environment, Inc. 2017

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte

Key:

Qualifiers

J = Estimated value

U = Not detected (method detection limit shown)

Other

mg/kg = milligrams per kilogram

N/A = No standard or guidance value available.

Notes:

1. Bold value denotes a detection of the analyte. Shaded values denote an exceedance of the NYSDEC screening criteria, underlined values denote an exceedance of the NJDEP screening criteria and an underlined and shaded value denotes an exceedance of both criteria.

[1] Reporting limits from USACE/EPA Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal (2014). Present reporting limits from The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters (1997). If both documents provide reporting limits, only the lowest is shown here.

[2] NJDEP Ecological Screening Criteria (2009). Values denoted with * are Effects Range Medium.

[3] Primary threshold value from NYS TOGS 5.1.9 (2004). Value presented from NYS Screening and Assessment of Contaminated Sediment (2014).

Key at end of table.

C Sediment Chemistry Laboratory Reports

If requested, electronic copies of the Sediment Chemistry Laboratory Reports will be provided.

D Data Usability Summary Reports

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Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: 10 April 2017	Completed by: Lynne Parker

The analytical data provided by the laboratory were reviewed for precision, accuracy, and completeness based on applicable sections of the following guidance:

- EPA Region 2 Data Validation Standard Operating Procedures.
  - *SOP NO. HW-3B, Rev. 0, ICP-MS Data Validation*

Specific criteria for QC limits were obtained from the site specific QAPP: *Offshore Sampling and Analysis Plan / Quality Assurance Project Plan, Version 1.1, Northeast Supply Enhancement Project*. Compliance with the project QA program is indicated in the checklist and tables below. Any major or minor concerns affecting data usability are listed below. The checklist and tables also indicate whether data qualification is required and/or the type of qualifier assigned.

Reference:

Project ID	Lab Work Order	Laboratory
1000891.0020.05	L1710301	Alpha Analytical

Table 1: Sample List

SDG	Matrix	Sample Name	Lab ID	Sample Date	Sample Type	ID Correction
L1710301	SE	BHA-7 SS1 0-2	L1710301-01	10/8/2016	N	
L1710301	SE	BHA-7 SS2 2-4	L1710301-02	10/8/2016	N	
L1710301	SE	BHA-7 SS3 4-6	L1710301-03	10/8/2016	N	
L1710301	SE	BHA-7 SS4 6-8	L1710301-04	10/8/2016	N	
L1710301	SE	BHA-7 SS5 8-10	L1710301-05	10/8/2016	N	
L1710301	SE	BHA-7 SS6 10-12	L1710301-06	10/8/2016	N	
L1710301	SE	BHA-7 SS7 15-17	L1710301-07	10/8/2016	N	
L1710301	SE	BHA-9 SS2 2-4	L1710301-08	10/8/2016	N	
L1710301	SE	BHA-9 SS3 4-6	L1710301-09	10/8/2016	N	
L1710301	SE	BHA-9 SS4 6-8	L1710301-10	10/8/2016	N	
L1710301	SE	BHA-9 SS5 8-10	L1710301-11	10/8/2016	N	
L1710301	SE	BHA-9 SS6 10-12	L1710301-12	10/8/2016	N	
L1710301	SE	BHA-9 SS7 15-17	L1710301-13	10/8/2016	N	

Table 1A: Sample Test Summary

SDG	Matrix	Test Method	Prep Method	Number of Samples	Sample Type
L1710301	SE	SW6020A	EPA 3050B	13	N
L1710301	SE	SW7474	EPA 7474	13	N

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: 10 April 2017	Completed by: Lynne Parker

General Sample Information	
Do Samples and Analyses on COC check against Lab Sample Tracking Form?	No. The samples were marked as collected on 10/9/16 on the COC; however, the case narrative noted that Alpha contacted Alpine and they confirmed via email that the collection date was 10/8/16. The sample collection forms indicate that Boring Number BHA-7 was started on 10/9/16 and completed 10/9/16, and Boring Number BHA-9 was started on 10/8/16 and completed 10/9/16. The COC did not request mercury analysis; however, the laboratory analyzed for the analyte.
Did coolers arrive at lab between 2 and 6°C and in good condition as indicated on COC and Cooler Receipt Form?	No. The samples arrived at the laboratory at 14.9°C and were stored at ambient conditions prior to shipment to the laboratory. Thermal preservation is not required for solid samples for metals analysis.
Frequency of Field QC Samples Correct? Field Duplicate - 1/20 samples MS/MSD – 1/20 samples Trip Blank - Every cooler with VOCs waters only Equipment Blank - 1/ set of samples per day?	Field QC samples were not required. These samples were analyzed for confirmation of the chemical vibracore results. The laboratory used sample BHA-7-SS1 0-2 for laboratory QC – MS and DUP for both Methods SW-846 6020 and 7474.
Case narrative present and complete?	Yes.
Any holding time violations?	Yes. All of the results for mercury were outside of holding time and R/UR qualified as rejected.

The following tables are presented at the end of this DUSR and provided summaries of results outside QC criteria:

- Method Blanks Results (Table 2 and 2A)
- Surrogates Outside Limits (Table 3)
- MS/MSD/LR Outside Limits (Table 4 and 4A)
- LCS Outside Limits (Table 5 and 5A)
- Re-analysis Results (Table 6)
- Field Duplicate Results (Table 7)

Go to Tables List

Metals & Mercury by ICP/MS and CVAA	
Description	**Notes and Qualifiers**
Are any compounds present in method blank as noted on Table 2?	Antimony was detected below the PQL in method blank WG991289-1. There were several detections for antimony, molybdenum, tin, sodium, arsenic, and/or silver in the initial calibration blank (ICB) and continuing calibration blank (CCB).
For samples, if results are <5 times the blank then "U" flag data (See Table 2A)	Eight sample results for antimony were less than 5X the blank detection and were U qualified as non-detect due to method blank contamination. Eleven sample results for molybdenum and nine sample results for tin were U qualified as non-detect due to ICB and/or CCB detections.
Is laboratory QC frequency one blank and LCS with each batch and one set of MS/MSD per 20 samples?	Yes.
Are MS/MSD within QC criteria (see Table 4)? If not, was a post digestion spike analyzed? QC limits are not applicable to sample results greater than 4 times spike amount.	No. Arsenic, calcium, manganese, selenium, and titanium were recovered outside of laboratory control limits. The case narrative noted that the recoveries in the post digestion spike were acceptable; therefore, the results in the parent sample were J qualified as estimated. The native concentration of aluminum and iron was greater than 4 times the spiking concentration; therefore, no qualification of the data was required.
Were elements recovered ≤30%? If so, "R" flag associated NDs.	Yes. Antimony was recovered at 20% in LCS WG991289-2. The laboratory limits for the reference material are 1-200%. No qualification of the data was made.
Is LCS within QC criteria (see Table 5 and 5A)? If out, and the recovery is high with no positive values, then no data qualification is required.	Yes.
Is there one serial dilution per 20 samples? Flag all data reported with an "E" as "J".	Yes.
Spot check ICS recoveries 80-120%. Contact lab if unacceptable.	The ICS recoveries were acceptable
Spot check ICV 90-110%. Contact lab if unacceptable.	The ICV recoveries were acceptable.
Spot check CCV 90-110%. Contact lab if unacceptable.	The CCV recoveries were acceptable.
Spot check ICB/CCB +/- RL. Contact lab if unacceptable.	The ICB/CCB detections were all less than the PQL.
Are laboratory replicates within limits (see Table 4A)?	No. Antimony, arsenic, barium, calcium, chromium, and titanium were outside of control limits in the laboratory duplicate of sample BHA-7 SS1 0-2. The results in the parent sample were J qualified as estimated.
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and dilutions is only one reportable result by flagged?	Samples BHA-7 SS1 0-2 and BHA-7 SS1 2-4 were analyzed at 10X to bring the mercury concentration within the calibration curve. No data usability issues.
Do field duplicate results show good precision for all compounds (see Table 7)?	Not applicable.

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: 10 April 2017	Completed by: Lynne Parker

Summary of Findings
• Arsenic, calcium, manganese, selenium, and titanium were recovered outside of laboratory control limits for the MS/MSD. The case narrative noted that the recoveries in the post digestion spike were acceptable; therefore, the results in sample BHA-7 SS1 0-2 were J qualified as estimated. • Antimony, arsenic, barium, calcium, chromium, and titanium were outside of control limits in the laboratory duplicate of sample BHA-7 SS1 0-2. The results in the parent sample were J qualified as estimated. • Eight sample results for antimony were less than 5X the blank detection and were U qualified as non-detect. • Eleven sample results for molybdenum and nine sample results for tin were U qualified as non-detect due to ICB and/or CCB detections.

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: 10 April 2017	Completed by: Lynne Parker

Table 2 – List of Positive Results for Blank Samples

Method	Sample ID	Sample Type	Analyte	Result	Qualifier	Units	MDL	PQL
SW6020	WG991289-1	MB	ANTIMONY	0.645	J	mg/kg	0.169	2.00
SW6020	R955527-2	ICB	ANTIMONY	2.33	J	µg/L	0.338	4.0
SW6020	R955527-2	ICB	MOLYBDENUM	0.327	J	µg/L	0.27	2.0
SW6020	R955527-2	ICB	TIN	0.382	J	µg/L	0.31	3.0
SW6020	R955527-23	CCB	ANTIMONY	3.22	J	µg/L	0.338	4.0
SW6020	R955527-23	CCB	MOLYBDENUM	0.647	J	µg/L	0.27	2.0
SW6020	R955527-23	CCB	TIN	0.784	J	µg/L	0.31	3.0
SW6020	R955527-25	CCB	ANTIMONY	3.13	J	µg/L	0.338	4.0
SW6020	R955527-25	CCB	MOLYBDENUM	0.582	J	µg/L	0.27	2.0
SW6020	R955527-25	CCB	TIN	0.810	J	µg/L	0.31	3.0
SW6020	R955527-27	CCB	ANTIMONY	3.67	J	µg/L	0.338	4.0
SW6020	R955527-27	CCB	MOLYBDENUM	0.567	J	µg/L	0.27	2.0
SW6020	R955527-27	CCB	TIN	0.793	J	µg/L	0.31	3.0
SW6020	R955527-29	CCB	ANTIMONY	3.52	J	µg/L	0.338	4.0
SW6020	R955527-29	CCB	MOLYBDENUM	0.618	J	µg/L	0.27	2.0
SW6020	R955527-29	CCB	SODIUM	51.5	J	µg/L	29.3	375
SW6020	R955527-29	CCB	TIN	0.790	J	µg/L	0.31	3.0
SW6020	R955527-31	CCB	ANTIMONY	3.55	J	µg/L	0.338	4.0
SW6020	R955527-31	CCB	ARSENIC	0.203	J	µg/L	0.165	1.25
SW6020	R955527-31	CCB	MOLYBDENUM	0.601	J	µg/L	0.27	2.0
SW6020	R955527-31	CCB	SILVER	0.132	J	µg/L	0.122	1.25
SW6020	R955527-31	CCB	SODIUM	31.2	J	µg/L	29.3	375
SW6020	R955527-31	CCB	TIN	0.753	J	µg/L	0.31	3.0

Table 2A – List of Samples Qualified for Blank Contamination

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	PQL	Affected Samples	Sample Flag
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	6.8		3.1	BHA-7 SS1 0-2	None
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	8.32		2.99	BHA-7 SS2 2-4	None
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	7.15		2.85	BHA-7 SS3 4-6	None
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	6.19		2.72	BHA-7 SS4 6-8	None

Data Usability Summary Report	Project: NESE Offshore Sampling
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Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	PQL	Affected Samples	Sample Flag
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	1.06	J	2.36	BHA-7 SS5 8-10	U Flag
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	0.475	J	2.03	BHA-7 SS6 10-12	U Flag
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	0.458	J	2.96	BHA-7 SS7 15-17	U Flag
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	0.396	J	2.18	BHA-9 SS2 2-4	U Flag
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	0.403	J	1.96	BHA-9 SS3 4-6	U Flag
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	0.226	J	1.85	BHA-9 SS4 6-8	U Flag
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	0.256	J	1.82	BHA-9 SS5 8-10	U Flag
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	0.29	J	1.79	BHA-9 SS6 10-12	U Flag
SW6020A	WG991289-1	SQ	Antimony, Total	0.645	ND	U	1.98	BHA-9 SS7 15-17	None
SW6020A	R955527-23	SQ	MOLYBDENUM, TOTAL	0.647	2.683		2.0	BHA-7 SS1 0-2	U Flag
SW6020A	R955527-23	SQ	MOLYBDENUM, TOTAL	0.647	2.838		2.0	BHA-7 SS2 2-4	U Flag
SW6020A	R955527-23	SQ	MOLYBDENUM, TOTAL	0.647	2.557		2.0	BHA-7 SS3 4-6	U Flag
SW6020A	R955527-23	SQ	MOLYBDENUM, TOTAL	0.647	2.927		2.0	BHA-7 SS4 6-8	U Flag
SW6020A	R955527-25	SQ	TIN, TOTAL	0.810	3.874		3.0	BHA-7 SS5 8-10	U Flag
SW6020A	R955527-25	SQ	MOLYBDENUM, TOTAL	0.582	1.306		2.0	BHA-7 SS6 10-12	U Flag
SW6020A	R955527-25	SQ	TIN, TOTAL	0.810	1.731		3.0	BHA-7 SS6 10-12	U Flag
SW6020A	R955527-25	SQ	TIN, TOTAL	0.810	2.099	J	3.0	BHA-7 SS7 15-17	U Flag
SW6020A	R955527-25	SQ	MOLYBDENUM, TOTAL	0.582	1.912	J	2.0	BHA-9 SS2 2-4	U Flag
SW6020A	R955527-25	SQ	TIN, TOTAL	0.810	1.860	J	3.0	BHA-9 SS2 2-4	U Flag
SW6020A	R955527-25	SQ	TIN, TOTAL	0.810	1.792	J	3.0	BHA-9 SS3 4-6	U Flag
SW6020A	R955527-25	SQ	MOLYBDENUM, TOTAL	0.582	2.407		2.0	BHA-9 SS3 4-6	U Flag
SW6020A	R955527-25	SQ	TIN, TOTAL	0.810	1.350	J	3.0	BHA-9 SS4 6-8	U Flag
SW6020A	R955527-25	SQ	MOLYBDENUM, TOTAL	0.582	0.413	J	2.0	BHA-9 SS4 6-8	U Flag
SW6020A	R955527-25	SQ	TIN, TOTAL	0.810	0.653	J	3.0	BHA-9 SS7 15-17	U Flag
SW6020A	R955527-25	SQ	MOLYBDENUM, TOTAL	0.582	0.564	J	2.0	BHA-9 SS7 15-17	U Flag
SW6020A	R955527-29	SQ	TIN, TOTAL	0.790	1.051	J	3.0	BHA-9 SS5 8-10	U Flag
SW6020A	R955527-29	SQ	MOLYBDENUM, TOTAL	0.618	0.696	J	2.0	BHA-9 SS5 8-10	U Flag
SW6020A	R955527-29	SQ	TIN, TOTAL	0.790	1.301	J	3.0	BHA-9 SS6 10-12	U Flag
SW6020A	R955527-29	SQ	MOLYBDENUM, TOTAL	0.618	0.404	J	2.0	BHA-9 SS6 10-12	U Flag

Table 3 – List of Samples with Surrogates outside Control Limits

N/A

Table 4A - List MS/MSD Recoveries outside Control Limits

Method	Sample ID	Sample Type	Analyte	Orig. Result	Spike Amount	MS	Dil Fac	Low Limit	High Limit	Sample Qualifier
SW6020A	WG991289-3-MS	SQ	Aluminum, Total	10200	303	594	10	75	125	4X
SW6020A	WG991289-3-MS	SQ	Arsenic, Total	39.1	18.2	134	10	75	125	J Flag
SW6020A	WG991289-3-MS	SQ	Calcium, Total	5040	1510	0	10	75	125	J Flag
SW6020A	WG991289-3-MS	SQ	Iron, Total	32400	151	0	10	75	125	4X
SW6020A	WG991289-3-MS	SQ	Manganese, Total	274	75.7	13	10	75	125	J Flag
SW6020A	WG991289-3-MS	SQ	Selenium, Total	3.36	18.2	126	10	75	125	J Flag
SW6020A	WG991289-3-MS	SQ	Titanium, Total	362	151	167	10	75	125	J Flag

Table 4B – List RPDs outside Control Limits

Method	Sample ID	Analyte	RPD	RPD Limit	Sample Qualifier
SW6020A	WG991289-4-DUP	Antimony, Total	28	20	J Flag
SW6020A	WG991289-4-DUP	Arsenic, Total	40	20	J Flag
SW6020A	WG991289-4-DUP	Barium, Total	25	20	J Flag
SW6020A	WG991289-4-DUP	Calcium, Total	22	20	J Flag
SW6020A	WG991289-4-DUP	Chromium, Total	21	20	J Flag
SW6020A	WG991289-4-DUP	Titanium, Total	32	20	J Flag

Table 5A - List LCS Recoveries outside Control Limits

None

Table 5B – List RPDs outside Control Limits

None

Table 6 –Samples that were Reanalyzed/Diluted

Sample ID	Lab ID	Method	Sample Type	Action
BHA-7 SS1 0-2	L1710301-01	SW7474	N	10X – Sample was diluted to bring the concentration within the calibration curve.
BHA-7 SS2 2-4	L1710301-02	SW7474	N	10X – Sample was diluted to bring the concentration within the calibration curve.

Table 7 – Summary of Field Duplicate Results
N/A

Acronym List and Table Key:

BD	=	blank spike duplicate
BS	=	blank spike
COC	=	chain of custody
CVAA	=	cold vapor atomic absorption
DUSR	=	data usability summary report
FD	=	field duplicate sample
ICP/MS	=	Inductively coupled / mass spectrometry
LB	=	laboratory blank
LCS	=	laboratory control sample
LCSD	=	laboratory control sample duplicate
LR	=	laboratory replicate
MB	=	method blank
MS	=	matrix spike
MSD	=	matrix spike duplicate
N	=	normal field sample
NC	=	not calculated
ND	=	not detected
NJDEP	=	New Jersey Department of Environmental Protection
NYSDEC	=	New York State Department of Environmental Conservation
PQL	=	practical quantitation limit
QA	=	quality assurance
QAPP	=	quality assurance project plan
QC	=	quality control
RB	=	rinsate blank sample
RPD	=	relative percent difference
SDG	=	sample delivery group

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: June 20, 2017	Completed by: Eridania Marte; Lynne Parker

The analytical data provided by the laboratory were reviewed for precision, accuracy, and completeness based on applicable sections of the following guidance:

- EPA Region 2 Data Validation Standard Operating Procedures.
  - *SOP NO. HW-3B, Rev. 0, ICP-MS Data Validation*
  - *SOP NO. HW-3C, Rev. 0, Mercury and Cyanide Data Validation*

Specific criteria for QC limits were obtained from the site specific QAPP: *Offshore Sampling and Analysis Plan / Quality Assurance Project Plan, Version 1.1, Northeast Supply Enhancement Project*. Compliance with the project QA program is indicated in the checklist and tables below. Any major or minor concerns affecting data usability are listed below. The checklist and tables also indicate whether data qualification is required and/or the type of qualifier assigned.

Reference:

Project ID	Lab Work Order	Laboratory
100891.0020.05	L1717026	Alpha Analytical

Table 1: Sample List

SDG	Sample Name	Matrix	Lab ID	Sample Date	Sample Type	ID Correction
L1717026	DUP-D0-3E	SE	L1717026-10	5/19/2017 4:15:00 PM	FD	
L1717026	DUP-D3-6E	SE	L1717026-27	5/19/2017 2:38:00 PM	FD	
L1717026	DUP-D6-9E	SE	L1717026-18	5/19/2017 3:42:00 PM	FD	
L1717026	DUP-D9-10E	SE	L1717026-09	5/19/2017 5:07:00 PM	FD	
L1717026	VC88-D0-3E	SE	L1717026-01	5/19/2017 5:07:00 PM	N	
L1717026	VC88-D3-6E	SE	L1717026-02	5/19/2017 5:07:00 PM	N	
L1717026	VC88-D6-9E	SE	L1717026-03	5/19/2017 5:07:00 PM	N	
L1717026	VC88-D9-10E	SE	L1717026-04	5/19/2017 5:07:00 PM	N	
L1717026	VC89-D0-3E	SE	L1717026-05	5/19/2017 4:15:00 PM	N	
L1717026	VC89-D3-6E	SE	L1717026-06	5/19/2017 4:15:00 PM	N	
L1717026	VC89-D6-9E	SE	L1717026-07	5/19/2017 4:15:00 PM	N	
L1717026	VC89-D9-10E	SE	L1717026-08	5/19/2017 4:15:00 PM	N	
L1717026	VC90-D0-3E	SE	L1717026-11	5/19/2017 3:42:00 PM	N	
L1717026	VC90-D3-6E	SE	L1717026-12	5/19/2017 3:42:00 PM	N	
L1717026	VC90-D6-9E	SE	L1717026-13	5/19/2017 3:42:00 PM	N	
L1717026	VC90-D9-10E	SE	L1717026-14	5/19/2017 3:42:00 PM	N	
L1717026	VC91-D0-3E	SE	L1717026-15	5/19/2017 3:10:00 PM	N	
L1717026	VC91-D3-6E	SE	L1717026-16	5/19/2017 3:10:00 PM	N	
L1717026	VC91-D6-9.6E	SE	L1717026-17	5/19/2017 3:10:00 PM	N	
L1717026	VC92-D0-3E	SE	L1717026-19	5/19/2017 2:38:00 PM	N	
L1717026	VC92-D3-6E	SE	L1717026-20	5/19/2017 2:38:00 PM	N	
L1717026	VC92-D6-9E	SE	L1717026-21	5/19/2017 2:38:00 PM	N	
L1717026	VC92-D9-10E	SE	L1717026-22	5/19/2017 2:38:00 PM	N	
L1717026	VC93-D0-3E	SE	L1717026-23	5/20/2017 1:58:00 PM	N	

SDG	Sample Name	Matrix	Lab ID	Sample Date	Sample Type	ID Correction
L1717026	VC93-D3-6E	SE	L1717026-24	5/20/2017 1:58:00 PM	N	
L1717026	VC93-D6-9E	SE	L1717026-25	5/20/2017 1:58:00 PM	N	
L1717026	VC93-D9-10E	SE	L1717026-26	5/20/2017 1:58:00 PM	N	
L1717026	VC89-D0-3E-MS	SQ	WG1007570-3	5/19/2017 4:15:00 PM	MS	
L1717026	VC89-D0-3E-MS	SQ	WG1008096-3	5/19/2017 4:15:00 PM	MS	
L1717026	VC89-D0-3E-MSD	SQ	WG1007570-4	5/19/2017 4:15:00 PM	MSD	
L1717026	VC89-D0-3E-MSD	SQ	WG1008096-4	5/19/2017 4:15:00 PM	MSD	
L1717026	VC93-D0-3E-MS	SQ	WG1007571-3	5/20/2017 1:58:00 PM	MS	
L1717026	VC93-D0-3E-MS	SQ	WG1009290-3	5/20/2017 1:58:00 PM	MS	
L1717026	VC93-D0-3E-MSD	SQ	WG1007571-4	5/20/2017 1:58:00 PM	MSD	
L1717026	VC93-D0-3E-MSD	SQ	WG1009290-4	5/20/2017 1:58:00 PM	MSD	
L1717026	RINSE1	WH	L1717026-29	5/19/2017 6:04:00 PM	RB	RINSE1_20171905
L1717026	RINSE2	WH	L1717026-28	5/24/2017 12:00:00 PM	RB	RINSE2_20172405

Table 1A: Sample Test Summary

SDG	Matrix	Test Method	Prep Method	Number of Samples	Sample Type
L1717026	SE	6020A	3050B	23	N
L1717026	SE	6020A	3050B	4	FD
L1717026	WH	6020A	SW3020A	2	RB
L1717026	SE	E7474/SW846	METHOD	23	N
L1717026	SE	E7474/SW846	METHOD	4	FD
L1717026	WH	E7474/SW846	METHOD	2	RB

Table 1B: Holding Time Exceedances

None.

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: June 20, 2017	Completed by: Eridania Marte; Lynne Parker

General Sample Information	
Do Samples and Analyses on COC check against Lab Sample Tracking Form?	Yes. The rinsate sample names were updated to include the collection date as noted in Table 1.
Did coolers arrive at lab between 2 and 6°C and in good condition as indicated on COC and Cooler Receipt Form?	Yes.
Frequency of Field QC Samples Correct? Field Duplicate - 1/20 samples MS/MSD – 1/20 samples Trip Blank - Every cooler with VOCs waters only Equipment Blank - 1/ set of samples per day?	Yes. 4 FD per 27 samples. 1 MS/MSD per 27 samples. VOCs not collected, trip blank not required. 2 equipment blanks, one per day.
Case narrative present and complete?	Yes.
Any holding time violations?	No.

The following tables are presented at the end of this DUSR and provided summaries of results outside QC criteria:

- Method Blanks Results (Table 2)
- Surrogates Outside Limits (Table 3)
- MS/MSD/LR Outside Limits (Table 4)
- LCS/SRM Outside Limits (Table 5)
- Re-analysis Results (Table 6)
- Field Duplicate Results (Table 7)

Go to Tables List

Metals & Mercury by ICP/MS and CVAA	
Description	**Notes and Qualifiers**
Are any compounds present in method and field blanks as noted on Table 2?	Yes.
For samples, if results are <5 times the blank then "U" flag data.	Sodium was detected in method blank WG1007571-1. Associated sample results were greater than 5X the blank detection; therefore, no qualifications were made. Antimony and sodium were detected in method blank WG1008707-1. Associated sample results in RINSE2 were less than 5X the blank detection; therefore, the results were U qualified as non-detect and the MDL were elevated to the sample result. Mercury was detected in method blank WG1011473-1. Associated sample results in RINSE1 and RINSE2 were less than 5X the blank detection; therefore, the results were U qualified as non-detect and the MDL were elevated to the sample result. Barium, chromium, copper, manganese, sodium, strontium, and titanium were detected in RINSE1. All associated results for chromium, manganese, sodium, strontium, and titanium were greater than

	5X the blank detection; therefore, no qualifications were made. Copper was detected less than 5X the rinsate blank detection in twelve samples and barium was detected less than 5X the rinsate blank detection in four samples. In instances where the blank detection was greater than the PQL, the PQL was elevated to the sample result. In instances where the blank detection was less than the PQL, the MDL was elevated to the sample result. Aluminum, barium, copper, strontium, and titanium were detected in RINSE2. All associated samples for aluminum, strontium, and titanium were greater than 5X the blank detection; therefore, no qualifications were made. Copper and barium were detected less than 5X the rinsate blank detection in three samples. The results were U qualified as non-detect and the MDL was elevated to sample result.
Is laboratory QC frequency one blank and LCS with each batch and one set of MS/MSD per 20 samples?	Yes.
Are MS/MSD within QC criteria (see Table 3)? If not, was a post digestion spike analyzed? QC limits are not applicable to sample results greater than 4 times spike amount.	No. The MS/MSD for sample VC89-D0-3E was recovered outside of acceptance limits for aluminum, iron, and zinc. The native concentration of aluminum and iron were greater than 4X the spike amount; therefore, no qualification was made. Zinc was recovered high in the MS and exceeded the acceptance criteria in the post digestion spike. The results in the parent sample were J qualified as estimated. The MS/MSD for sample VC93-D0-3E was recovered outside of acceptance limits for aluminum, iron, and calcium. The native concentration of aluminum and iron were greater than 4X the spike amount; therefore, no qualification was made. Calcium was recovered high in the MS and MSD; however, the post digestion spike recovery was acceptable. The MS and MSD exhibited poor precision for calcium. The results in the parent sample were J qualified as estimated.
Were elements recovered ≤30%? If so, “R” flag associated NDs.	The MS/MSD for sample VC89-D0-3E exhibited 0% recovery for iron, as noted above. No qualification was made.
Is LCS within QC criteria (see Table 4)? If out, and the recovery is high with no positive values, then no data qualification is required.	Yes.
Is there one serial dilution per 20 samples? Flag all data reported with an “E” as “J”.	Yes.
Spot check ICS recoveries 80-120%. Contact lab if unacceptable.	Yes. The ICS exhibited recovery at 122% for titanium. All sediment sample results for titanium were J qualified as estimated.
Spot check ICV 90-110%. Contact lab if unacceptable.	Yes.

Spot check CCV 90-110%. Contact lab if unacceptable.	Yes.
Spot check ICB/CCB +/- RL. Contact lab if unacceptable.	Yes. ICB R974427-2 exhibited detection for antimony at 0.759 µg/L. Associated samples VC92-D6-9E (0.492 µg/L), VC88-D9-10E (0.451 µg/L), and VC93-D0-3E (0.823 µ/L) were less than 5X the blank detection. The results were U qualified as non-detect and the MDL was elevated to the sample result. CCBs throughout the analytical batch also exhibited detections for antimony, which indicates instrument contamination. CCB's R974427-25, R974427-27, and R974427-29 exhibited detections for antimony at 0.814, 0.758, and 0.799 µg/L respectively. Thirteen samples associated with CCB R974427-25, fifteen samples with CCB R974427-27, and eleven samples with CCB R974427-29 were greater than 5X the blank detection. No qualification was made. CCB R974427-23 and CCB R974427-31 exhibited detection for antimony at 0.758 and 0.719 µg/L respectively. Four samples associated with CCB R974427-23 and six with CCB R974427-31 were greater than 5X the blank detection. No qualification was made. CCB's R974427-27, R974427-29, and R974427-31 exhibited detections for sodium at 43.4, 55.8, and 38.7 µg/L respectively. Seventeen samples associated with CCB R974427-27, thirteen with CCB R974427-29, and six with CCB R974427-31 were greater than 5X the blank detection. No qualification was made. CCB's R973606-15 and R973606-17 exhibited detections for sodium at 90.8 and 72.1 µg/L respectively. Associated sample RINSE1 (283 µg/L) was less than 5X the blank detection. The result was U qualified as non-detect and the PQL was elevated to the sample result. ICB R973606-2 and CCB's R973606-8, R973606-11, and R973606-17 exhibited detections for antimony at 2.36, 2.90, 2.73, and 2.47 µg/L respectively. The sample results were U qualified due to instrument contamination. No further qualification made. CCB R973606-11 exhibited detection for silver at 0.242 µg/L. Associated samples were non-detected; therefore, no qualification was made.
Are laboratory replicates within limits?	Yes.
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and	All sediment samples were analyzed at a 5X dilution for mercury analysis.

dilutions is only one reportable result by flagged?	All sediment samples were analyzed at a 10X dilution for metals analysis. All of the detection limits were less than the screening criteria; therefore, there is not impact to data usability.
Do field duplicate results show good precision for all compounds (see Table 6)?	Overall, the field duplicate results show good precision. Only few metals were qualified as follows: Sample VC92-D3-6E and field duplicate DUP-D3-6E exhibited poor precision for calcium. The sample results were J qualified as estimated. Sample VC90-D6-9E and field duplicate DUP-D6-9E exhibited poor precision for calcium and strontium. The sample results were J qualified as estimated.

Summary of Findings

- 6020A: Antimony and sodium were detected in method blank WG1008707-1. Associated sample was U qualified as non-detect and the MDL was elevated to the sample result.
- 6020A: Barium and copper were detected in rinse blank RINSE1. Sample results were less than 5X the blank detection and were U qualified as non-detect. Sample results in which were less than the PQL, the MDL was elevated to the sample result and sample results in which were greater than the PQL, the PQL was elevated to the sample result.
- 6020A: Barium and copper were detected in rinse blank RINSE2. Sample results were less than 5X the blank detection and were U qualified as non-detect. The MDL was elevated to sample result.
- E7474: Mercury was detected in method blank WG1011473-1. Sample results were less than 5X the blank detection and were U qualified as non-detect. The MDL was elevated to the sample result.
- 6020A: ICB R974427-2 exhibited detection for antimony. Associated sample results were less than 5X the blank detection and were U qualified as non-detect. The MDL was elevated to the sample result.
- 6020A: CCB's R973606-15 and R973606-17 exhibited detections for sodium. Associated sample results were less than 5X the blank detection and were U qualified as non-detect. The PQL was elevated to the sample result.
- 6020A: Sample VC92-D3-6E and field duplicate DUP-D3-6E exhibited poor precision for calcium. Sample VC90-D6-9E and field duplicate DUP-D6-9E exhibited poor precision for calcium and strontium. The sample results were J qualified as estimated in the field duplicate pair.

Table 2 List of Positive Results for Blank Samples

Method	Sample Name	Matrix	Analyte	Result	Qualifier	Units	MDL	QL
6020A	WG1007571-1	LB	SODIUM, TOTAL	21.6	J	mg/kg	11.7	150
6020A	WG1008707-1	LB	ANTIMONY, TOTAL	0.00118	J	mg/l	0.00042	0.004
6020A	WG1008707-1	LB	SODIUM, TOTAL	0.0419	J	mg/l	0.0293	0.2
E7474/SW846	WG1011473-1	LB	MERCURY, TOTAL	0.00003	J	mg/l	0.00001	0.00005
6020A	RINSE1	RB	BARIUM, TOTAL	0.00164		mg/l	0.00017	0.0005
6020A	RINSE1	RB	CHROMIUM, TOTAL	0.00022	J	mg/l	0.00017	0.0005
6020A	RINSE1	RB	COPPER, TOTAL	0.00339		mg/l	0.00038	0.001
6020A	RINSE1	RB	MANGANESE, TOTAL	0.00047	J	mg/l	0.00044	0.001
6020A	RINSE1	RB	SODIUM, TOTAL	0.283		mg/l	0.0293	0.1
6020A	RINSE1	RB	STRONTIUM, TOTAL	0.00065		mg/l	0.00003	0.0005
6020A	RINSE1	RB	TITANIUM, TOTAL	0.00021	J	mg/l	0.00007	0.0005
6020A	RINSE2	RB	ALUMINUM, TOTAL	0.00461	J	mg/l	0.00327	0.01
6020A	RINSE2	RB	BARIUM, TOTAL	0.00092		mg/l	0.00017	0.0005
6020A	RINSE2	RB	COPPER, TOTAL	0.00148		mg/l	0.00038	0.001
6020A	RINSE2	RB	STRONTIUM, TOTAL	0.00034	J	mg/l	0.00003	0.0005
6020A	RINSE2	RB	TITANIUM, TOTAL	0.00016	J	mg/l	0.00007	0.0005

Table 2A List of Samples Qualified for Blank Contamination

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	Units	MDL	QL	Affected Sample	Sample Flag
6020A	WG1008707-1	WH	ANTIMONY, TOTAL	0.00118	0.00076	J	mg/l	0.00042	0.004	RINSE2	U Flag
6020A	WG1008707-1	WH	SODIUM, TOTAL	0.0419	0.0787	J	mg/l	0.0293	0.2	RINSE2	U Flag
E7474/SW846	WG1011473-1	WH	MERCURY, TOTAL	0.00003	0.00002	J	mg/l	0.00001	0.00005	RINSE1	U Flag
E7474/SW846	WG1011473-1	WH	MERCURY, TOTAL	0.00003	0.00002	J	mg/l	0.00001	0.00005	RINSE2	U Flag
6020A	RINSE1	RB	Copper	3.39	2.49	J	ug/L	0.246	2.53	VC88-D0-3E	U Flag

Table 2A List of Samples Qualified for Blank Contamination

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	Units	MDL	QL	Affected Sample	Sample Flag
6020A	RINSE1	RB	Copper	3.39	6.15		ug/L	0.271	2.79	VC88-D3-6E	U Flag
6020A	RINSE1	RB	Copper	3.39	6.0		ug/L	0.241	2.49	VC89-D0-3E	U Flag
6020A	RINSE1	RB	Copper	3.39	14.6		ug/L	0.302	3.12	VC89-D3-6E	U Flag
6020A	RINSE1	RB	Copper	3.39	6.98		ug/L	0.235	2.42	DUP-D0-3E	U Flag
6020A	RINSE1	RB	Copper	3.39	9.132		ug/L	0.23	2.37	VC90-D0-3E	U Flag
6020A	RINSE1	RB	Copper	3.39	16.6		ug/L	0.266	2.74	VC90-D3-6E	U Flag
6020A	RINSE1	RB	Copper	3.39	2.8	J	ug/L	0.254	2.62	VC92-D0-3E	U Flag
6020A	RINSE1	RB	Copper	3.39	10.0		ug/L	0.243	2.5	VC92-D3-6E	U Flag
6020A	RINSE1	RB	Copper	3.39	9.47		ug/L	0.261	2.69	VC92-D6-9E	U Flag
6020A	RINSE1	RB	Copper	3.39	6.25		ug/L	0.252	2.6	VC92-D9-10E	U Flag
6020A	RINSE1	RB	Copper	3.39	5.9		ug/L	0.244	2.51	DUP-D3-6E	U Flag
6020A	RINSE1	RB	Barium	1.64	4.17	J	ug/L	0.268	3.8	VC88-D0-3E	U Flag
6020A	RINSE1	RB	Barium	1.64	4.813	J	ug/L	0.263	3.73	VC89-D0-3E	U Flag
6020A	RINSE1	RB	Barium	1.64	3.6	J	ug/L	0.256	3.63	DUP-D0-3E	U Flag
6020A	RINSE1	RB	Barium	1.64	6.047	J	ug/L	0.277	3.93	VC92-D0-3E	U Flag
6020A	RINSE2	RB	Copper	1.48	2.137	J	ug/L	0.226	2.33	VC93-D3-6E	U Flag
6020A	RINSE2	RB	Copper	1.48	1.34	J	ug/L	0.24	2.47	VC93-D6-9E	U Flag
6020A	RINSE2	RB	Copper	1.48	1.429	J	ug/L	0.239	2.47	VC93-D9-10E	U Flag
6020A	RINSE2	RB	Barium	0.92	4.01	J	ug/L	0.246	3.49	VC93-D3-6E	U Flag
6020A	RINSE2	RB	Barium	0.92	1.95	J	ug/L	0.261	3.71	VC93-D6-9E	U Flag
6020A	RINSE2	RB	Barium	0.92	1.903	J	ug/L	0.26	3.7	VC93-D9-10E	U Flag

Table 3 – List of Samples with Surrogates outside Control Limits

None.

Table 4 List MS/MSD Recoveries outside Control Limits

Method	Parent Sample	Analyte	Dil Fac	Unit	MS	MSD	Low Limit	High Limit	Qualifier
6020A	VC89-D0-3E	ALUMINUM, TOTAL	10	mg/kg	501	295	75	125	None- 4X
6020A	VC89-D0-3E	IRON, TOTAL	10	mg/kg	2430	0	75	125	None - 4X
6020A	VC89-D0-3E	ZINC, TOTAL	10	mg/kg	127		75	125	Diluted Out
6020A	VC93-D0-3E	ALUMINUM, TOTAL	10	mg/kg	371	430	75	125	None - 4X
6020A	VC93-D0-3E	CALCIUM, TOTAL	10	mg/kg	174	310	75	125	Diluted Out
6020A	VC93-D0-3E	IRON, TOTAL	10	mg/kg	857	1670	75	125	None - 4X

Table 4A List RPDs outside Control Limits

Method	Parent Sample	Analyte	Dil Fac	Unit	RPD	RPD Limit	Qualifier	Sample Type
6020A	VC93-D0-3E	CALCIUM, TOTAL	10	mg/kg	57	20	Diluted Out	MSD

Table 4B – List of Laboratory Replicate outside Control Limits

None.

Table 5 – List LCS Recoveries outside Control Limits

None.

Table 5A – List RPDs outside Control Limits

None.

Table 5B – List SRM Recoveries outside Control Limits

N/A

Table 6 Samples that were Reanalyzed/Diluted

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
DUP-D0-3E	SE	6020A	FD	N	5	10	INITIAL	Sample initially diluted at 10X for antimony, cadmium, silver, thallium, and tin

Table 6 Samples that were Reanalyzed/Diluted

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
DUP-D0-3E	SE	6020A	FD	Y	21	10	INITIAL	Sample initially diluted at 10X
DUP-D0-3E	SE	E7474/SW846	FD	N	1	5	INITIAL	Sample initially diluted at 5X
DUP-D3-6E	SE	6020A	FD	N	4	10	INITIAL	Sample initially diluted at 10X for antimony, silver, thallium, and tin
DUP-D3-6E	SE	6020A	FD	Y	22	10	INITIAL	Sample initially diluted at 10X
DUP-D3-6E	SE	E7474/SW846	FD	N	1	5	INITIAL	Sample initially diluted at 5X
DUP-D6-9E	SE	6020A	FD	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
DUP-D6-9E	SE	6020A	FD	Y	24	10	INITIAL	Sample initially diluted at 10X
DUP-D6-9E	SE	E7474/SW846	FD	Y	1	5	INITIAL	Sample initially diluted at 5X
DUP-D9-10E	SE	6020A	FD	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
DUP-D9-10E	SE	6020A	FD	Y	24	10	INITIAL	Sample initially diluted at 10X
DUP-D9-10E	SE	E7474/SW846	FD	Y	1	5	INITIAL	Sample initially diluted at 5X
VC88-D0-3E	SE	6020A	N	N	6	10	INITIAL	Sample initially diluted at 10X for antimony, cadmium, selenium, silver, thallium, and tin
VC88-D0-3E	SE	6020A	N	Y	20	10	INITIAL	Sample initially diluted at 10X
VC88-D0-3E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X
VC88-D3-6E	SE	6020A	N	N	4	10	INITIAL	Sample initially diluted at 10X for antimony, silver, thallium, and tin
VC88-D3-6E	SE	6020A	N	Y	22	10	INITIAL	Sample initially diluted at 10X
VC88-D3-6E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC88-D6-9E	SE	6020A	N	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
VC88-D6-9E	SE	6020A	N	Y	24	10	INITIAL	Sample initially diluted at 10X
VC88-D6-9E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC88-D9-10E	SE	6020A	N	N	1	10	INITIAL	Sample initially diluted at 10X for silver
VC88-D9-10E	SE	6020A	N	Y	25	10	INITIAL	Sample initially diluted at 10X

Table 6 Samples that were Reanalyzed/Diluted

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
VC88-D9-10E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC89-D0-3E	SE	6020A	N	N	4	10	INITIAL	Sample initially diluted at 10X for antimony, silver, cadmium, and tin
VC89-D0-3E	SE	6020A	N	Y	22	10	INITIAL	Sample initially diluted at 10X
VC89-D0-3E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X
VC89-D3-6E	SE	6020A	N	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
VC89-D3-6E	SE	6020A	N	Y	24	10	INITIAL	Sample initially diluted at 10X
VC89-D3-6E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC89-D6-9E	SE	6020A	N	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
VC89-D6-9E	SE	6020A	N	Y	24	10	INITIAL	Sample initially diluted at 10X
VC89-D6-9E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC89-D9-10E	SE	6020A	N	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
VC89-D9-10E	SE	6020A	N	Y	24	10	INITIAL	Sample initially diluted at 10X
VC89-D9-10E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC90-D0-3E	SE	6020A	N	N	3	10	INITIAL	Sample initially diluted at 10X for antimony, silver, and thallium
VC90-D0-3E	SE	6020A	N	Y	23	10	INITIAL	Sample initially diluted at 10X
VC90-D0-3E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC90-D3-6E	SE	6020A	N	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
VC90-D3-6E	SE	6020A	N	Y	24	10	INITIAL	Sample initially diluted at 10X
VC90-D3-6E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC90-D6-9E	SE	6020A	N	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
VC90-D6-9E	SE	6020A	N	Y	24	10	INITIAL	Sample initially diluted at 10X
VC90-D6-9E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X

Table 6 Samples that were Reanalyzed/Diluted

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
VC90-D9-10E	SE	6020A	N	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
VC90-D9-10E	SE	6020A	N	Y	24	10	INITIAL	Sample initially diluted at 10X
VC90-D9-10E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC91-D0-3E	SE	6020A	N	N	3	10	INITIAL	Sample initially diluted at 10X for antimony, silver, and tin
VC91-D0-3E	SE	6020A	N	Y	23	10	INITIAL	Sample initially diluted at 10X
VC91-D0-3E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X
VC91-D3-6E	SE	6020A	N	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
VC91-D3-6E	SE	6020A	N	Y	24	10	INITIAL	Sample initially diluted at 10X
VC91-D3-6E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC91-D6-9.6E	SE	6020A	N	N	2	10	INITIAL	Sample initially diluted at 10X for antimony and silver
VC91-D6-9.6E	SE	6020A	N	Y	24	10	INITIAL	Sample initially diluted at 10X
VC91-D6-9.6E	SE	E7474/SW846	N	Y	1	5	INITIAL	Sample initially diluted at 5X
VC92-D0-3E	SE	6020A	N	N	6	10	INITIAL	Sample initially diluted at 10X for antimony, cadmium, beryllium, silver, thallium, and tin
VC92-D0-3E	SE	6020A	N	Y	20	10	INITIAL	Sample initially diluted at 10X
VC92-D0-3E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X
VC92-D3-6E	SE	6020A	N	N	4	10	INITIAL	Sample initially diluted at 10X for antimony, silver, thallium, and tin
VC92-D3-6E	SE	6020A	N	Y	22	10	INITIAL	Sample initially diluted at 10X
VC92-D3-6E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X
VC92-D6-9E	SE	6020A	N	N	1	10	INITIAL	Sample initially diluted at 10X for silver
VC92-D6-9E	SE	6020A	N	Y	25	10	INITIAL	Sample initially diluted at 10X
VC92-D6-9E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X
VC92-D9-10E	SE	6020A	N	N	4	10	INITIAL	Sample initially diluted at 10X for antimony, silver, thallium, and tin

Table 6 Samples that were Reanalyzed/Diluted

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
VC92-D9-10E	SE	6020A	N	Y	22	10	INITIAL	Sample initially diluted at 10X
VC92-D9-10E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X
VC93-D0-3E	SE	6020A	N	N	1	10	INITIAL	Sample initially diluted at 10X for thallium
VC93-D0-3E	SE	6020A	N	Y	25	10	INITIAL	Sample initially diluted at 10X
VC93-D0-3E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X
VC93-D3-6E	SE	6020A	N	N	4	10	INITIAL	Sample initially diluted at 10X for antimony, silver, thallium, and cadmium
VC93-D3-6E	SE	6020A	N	Y	22	10	INITIAL	Sample initially diluted at 10X
VC93-D3-6E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X
VC93-D6-9E	SE	6020A	N	N	11	10	INITIAL	Sample initially diluted at 10X for antimony, cadmium, selenium, silver, thallium, tin, beryllium, cobalt, molybdenum, nickel, and zinc
VC93-D6-9E	SE	6020A	N	Y	15	10	INITIAL	Sample initially diluted at 10X
VC93-D6-9E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X
VC93-D9-10E	SE	6020A	N	N	10	10	INITIAL	Sample initially diluted at 10X for antimony, selenium, silver, thallium, tin, beryllium, cobalt, molybdenum, nickel, and zinc
VC93-D9-10E	SE	6020A	N	Y	16	10	INITIAL	Sample initially diluted at 10X
VC93-D9-10E	SE	E7474/SW846	N	N	1	5	INITIAL	Sample initially diluted at 5X

Table 7 – Summary of Field Duplicate Results

Method	Analyte	Unit	Matrix	PQL	VC89-D0-3E	DUP-D0-3E	RPD	RPD Rating	Sample Qual
6020A	ALUMINUM, TOTAL	mg/kg	SE	124	1590	1500	5.8%	Good	None
6020A	ARSENIC, TOTAL	mg/kg	SE	0.622	4.62	6.2	29.2%	Good	None
6020A	BERYLLIUM, TOTAL	mg/kg	SE	0.373	0.187	0.216	14.4%	Good	None

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: June 20, 2017	Completed by: Eridania Marte; Lynne Parker

Method	Analyte	Unit	Matrix	PQL	VC89-D0-3E	DUP-D0-3E	RPD	RPD Rating	Sample Qual
6020A	CALCIUM, TOTAL	mg/kg	SE	622	747	773	3.4%	Good	None
6020A	CHROMIUM, TOTAL	mg/kg	SE	2.49	7.8	11.3	36.6%	Good	None
6020A	COBALT, TOTAL	mg/kg	SE	0.622	1.82	0.943	63.5%	Good	None
6020A	IRON, TOTAL	mg/kg	SE	249	13000	13600	4.5%	Good	None
6020A	LEAD, TOTAL	mg/kg	SE	0.747	2.57	2.15	17.8%	Good	None
6020A	MAGNESIUM, TOTAL	mg/kg	SE	124	880	776	12.6%	Good	None
6020A	MANGANESE, TOTAL	mg/kg	SE	2.49	24.9	22.4	10.6%	Good	None
6020A	MOLYBDENUM, TOTAL	mg/kg	SE	0.996	1.06	0.983	7.5%	Good	None
6020A	NICKEL, TOTAL	mg/kg	SE	1.24	2.24	2.76	20.8%	Good	None
6020A	POTASSIUM, TOTAL	mg/kg	SE	124	350	293	17.7%	Good	None
6020A	SELENIUM, TOTAL	mg/kg	SE	2.49	1.86	1.58	16.3%	Good	None
6020A	SODIUM, TOTAL	mg/kg	SE	187	2180	2130	2.3%	Good	None
6020A	STRONTIUM, TOTAL	mg/kg	SE	1.24	5.97	6.01	0.7%	Good	None
6020A	THALLIUM, TOTAL	mg/kg	SE	0.249	0.188	ND	NC	--	--
6020A	TITANIUM, TOTAL	mg/kg	SE	0.622	65.5	44	39.3%	Good	None
6020A	VANADIUM, TOTAL	mg/kg	SE	1.24	20.3	19	6.6%	Good	None
6020A	ZINC, TOTAL	mg/kg	SE	12.4	9.74	10.3	5.6%	Good	None

Method	Analyte	Unit	Matrix	PQL	VC92-D3-6E	DUP-D3-6E	RPD	RPD Rating	Samp Qual
6020A	ALUMINUM, TOTAL	mg/kg	SE	125	3950	2800	34.1%	Good	None
6020A	ARSENIC, TOTAL	mg/kg	SE	0.626	7.81	4.51	53.6%	Good	None
6020A	BARIUM, TOTAL	mg/kg	SE	3.76	6.38	4.5	34.6%	Good	None
6020A	BERYLLIUM, TOTAL	mg/kg	SE	0.376	0.374	0.236	45.2%	Good	None
6020A	CADMIUM, TOTAL	mg/kg	SE	0.25	0.039	0.055	34.0%	Good	None
6020A	CALCIUM, TOTAL	mg/kg	SE	626	916	345	90.6%	Poor	J Flag
6020A	CHROMIUM, TOTAL	mg/kg	SE	2.5	12	11.1	7.8%	Good	None
6020A	COBALT, TOTAL	mg/kg	SE	0.626	2.78	1.57	55.6%	Good	None
6020A	IRON, TOTAL	mg/kg	SE	250	15900	12000	28.0%	Good	None
6020A	LEAD, TOTAL	mg/kg	SE	0.751	4.57	2.78	48.7%	Good	None
6020A	MAGNESIUM, TOTAL	mg/kg	SE	125	1970	1190	49.4%	Good	None

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: June 20, 2017	Completed by: Eridania Marte; Lynne Parker

Method	Analyte	Unit	Matrix	PQL	VC92-D3-6E	DUP-D3-6E	RPD	RPD Rating	Samp Qual
6020A	MANGANESE, TOTAL	mg/kg	SE	2.5	72.9	38.4	62.0%	Good	None
6020A	MOLYBDENUM, TOTAL	mg/kg	SE	1	1.01	0.796	23.7%	Good	None
6020A	NICKEL, TOTAL	mg/kg	SE	1.25	6.39	3.78	51.3%	Good	None
6020A	POTASSIUM, TOTAL	mg/kg	SE	125	835	624	28.9%	Good	None
6020A	SELENIUM, TOTAL	mg/kg	SE	2.5	3.29	2.3	35.4%	Good	None
6020A	SODIUM, TOTAL	mg/kg	SE	188	2740	2200	21.9%	Good	None
6020A	STRONTIUM, TOTAL	mg/kg	SE	1.25	9.64	5.6	53.0%	Good	None
6020A	TITANIUM, TOTAL	mg/kg	SE	0.626	101	71.2	34.6%	Good	None
6020A	VANADIUM, TOTAL	mg/kg	SE	1.25	20.3	15	30.0%	Good	None
6020A	ZINC, TOTAL	mg/kg	SE	12.5	25.2	14.6	53.3%	Good	None

Method	Analyte	Unit	Matrix	PQL	VC90-D6-9E	DUP-D6-9E	RPD	RPD Rating	Samp Qual
6020A	ALUMINUM, TOTAL	mg/kg	SE	176	15800	15300	3.2%	Good	None
6020A	ARSENIC, TOTAL	mg/kg	SE	0.881	14.6	12.9	12.4%	Good	None
6020A	BARIUM, TOTAL	mg/kg	SE	5.28	26.6	29.1	9.0%	Good	None
6020A	BERYLLIUM, TOTAL	mg/kg	SE	0.528	1	0.99	1.0%	Good	None
6020A	CADMIUM, TOTAL	mg/kg	SE	0.352	0.079	0.149	61.4%	Good	None
6020A	CALCIUM, TOTAL	mg/kg	SE	881	2740	34100	170.2%	Poor	J Flag
6020A	CHROMIUM, TOTAL	mg/kg	SE	3.52	34.1	34.2	0.3%	Good	None
6020A	COBALT, TOTAL	mg/kg	SE	0.881	9.42	8.94	5.2%	Good	None
6020A	COPPER, TOTAL	mg/kg	SE	3.52	14.5	13.6	6.4%	Good	None
6020A	IRON, TOTAL	mg/kg	SE	352	38500	37200	3.4%	Good	None
6020A	LEAD, TOTAL	mg/kg	SE	1.06	13.5	12.3	9.3%	Good	None
6020A	MAGNESIUM, TOTAL	mg/kg	SE	176	7860	7320	7.1%	Good	None
6020A	MANGANESE, TOTAL	mg/kg	SE	3.52	275	282	2.5%	Good	None
E7474	MERCURY, TOTAL	mg/kg	SE	0.023	0.013	0.013	0.0%	Good	None
6020A	MOLYBDENUM, TOTAL	mg/kg	SE	1.41	1.3	2.29	55.2%	Good	None
6020A	NICKEL, TOTAL	mg/kg	SE	1.76	24.7	23.8	3.7%	Good	None
6020A	POTASSIUM, TOTAL	mg/kg	SE	176	3500	3490	0.3%	Good	None
6020A	SELENIUM, TOTAL	mg/kg	SE	3.52	11.4	10.8	5.4%	Good	None

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: June 20, 2017	Completed by: Eridania Marte; Lynne Parker

Method	Analyte	Unit	Matrix	PQL	VC90-D6-9E	DUP-D6-9E	RPD	RPD Rating	Samp Qual
6020A	SODIUM, TOTAL	mg/kg	SE	264	6100	6410	5.0%	Good	None
6020A	STRONTIUM, TOTAL	mg/kg	SE	1.76	26.5	213	155.7%	Poor	J Flag
6020A	THALLIUM, TOTAL	mg/kg	SE	0.352	0.138	0.171	21.4%	Good	None
6020A	TIN, TOTAL	mg/kg	SE	2.11	0.47	0.477	1.5%	Good	None
6020A	TITANIUM, TOTAL	mg/kg	SE	0.881	272	343	23.1%	Good	None
6020A	VANADIUM, TOTAL	mg/kg	SE	1.76	38.2	38.3	0.3%	Good	None
6020A	ZINC, TOTAL	mg/kg	SE	17.6	76	69.8	8.5%	Good	None

Method	Analyte	Unit	Matrix	PQL	VC88-D9-10E	DUP-D9-10E	RPD	RPD Rating	Samp Qual
6020A	ALUMINUM, TOTAL	mg/kg	SE	179	16600	15700	5.6%	Good	None
6020A	ARSENIC, TOTAL	mg/kg	SE	0.894	14.5	14	3.5%	Good	None
6020A	BARIUM, TOTAL	mg/kg	SE	5.36	27.6	26.9	2.6%	Good	None
6020A	BERYLLIUM, TOTAL	mg/kg	SE	0.536	1.05	0.974	7.5%	Good	None
6020A	CADMIUM, TOTAL	mg/kg	SE	0.358	0.088	0.132	40.0%	Good	None
6020A	CALCIUM, TOTAL	mg/kg	SE	894	3000	3070	2.3%	Good	None
6020A	CHROMIUM, TOTAL	mg/kg	SE	3.58	36.1	33.6	7.2%	Good	None
6020A	COBALT, TOTAL	mg/kg	SE	0.894	10.2	9.4	8.2%	Good	None
6020A	COPPER, TOTAL	mg/kg	SE	3.58	14.3	13.3	7.2%	Good	None
6020A	IRON, TOTAL	mg/kg	SE	358	39100	36100	8.0%	Good	None
6020A	LEAD, TOTAL	mg/kg	SE	1.07	13.6	13.1	3.7%	Good	None
6020A	MAGNESIUM, TOTAL	mg/kg	SE	179	8390	7950	5.4%	Good	None
6020A	MANGANESE, TOTAL	mg/kg	SE	3.58	308	277	10.6%	Good	None
E7474	MERCURY, TOTAL	mg/kg	SE	0.026	0.014	0.012	15.4%	Good	None
6020A	MOLYBDENUM, TOTAL	mg/kg	SE	1.43	1.88	2.01	6.7%	Good	None
6020A	NICKEL, TOTAL	mg/kg	SE	1.79	25.3	24.4	3.6%	Good	None
6020A	POTASSIUM, TOTAL	mg/kg	SE	179	3710	3530	5.0%	Good	None
6020A	SELENIUM, TOTAL	mg/kg	SE	3.58	12.3	11.2	9.4%	Good	None
6020A	SODIUM, TOTAL	mg/kg	SE	268	7530	7660	1.7%	Good	None
6020A	STRONTIUM, TOTAL	mg/kg	SE	1.79	29.7	29	2.4%	Good	None
6020A	THALLIUM, TOTAL	mg/kg	SE	0.358	0.157	0.142	10.0%	Good	None
6020A	TIN, TOTAL	mg/kg	SE	2.14	0.568	0.515	9.8%	Good	None

Method	Analyte	Unit	Matrix	PQL	VC88-D9-10E	DUP-D9-10E	RPD	RPD Rating	Samp Qual
6020A	TITANIUM, TOTAL	mg/kg	SE	0.894	303	290	4.4%	Good	None
6020A	VANADIUM, TOTAL	mg/kg	SE	1.79	39.9	38.9	2.5%	Good	None
6020A	ZINC, TOTAL	mg/kg	SE	17.9	76.4	72.1	5.8%	Good	None

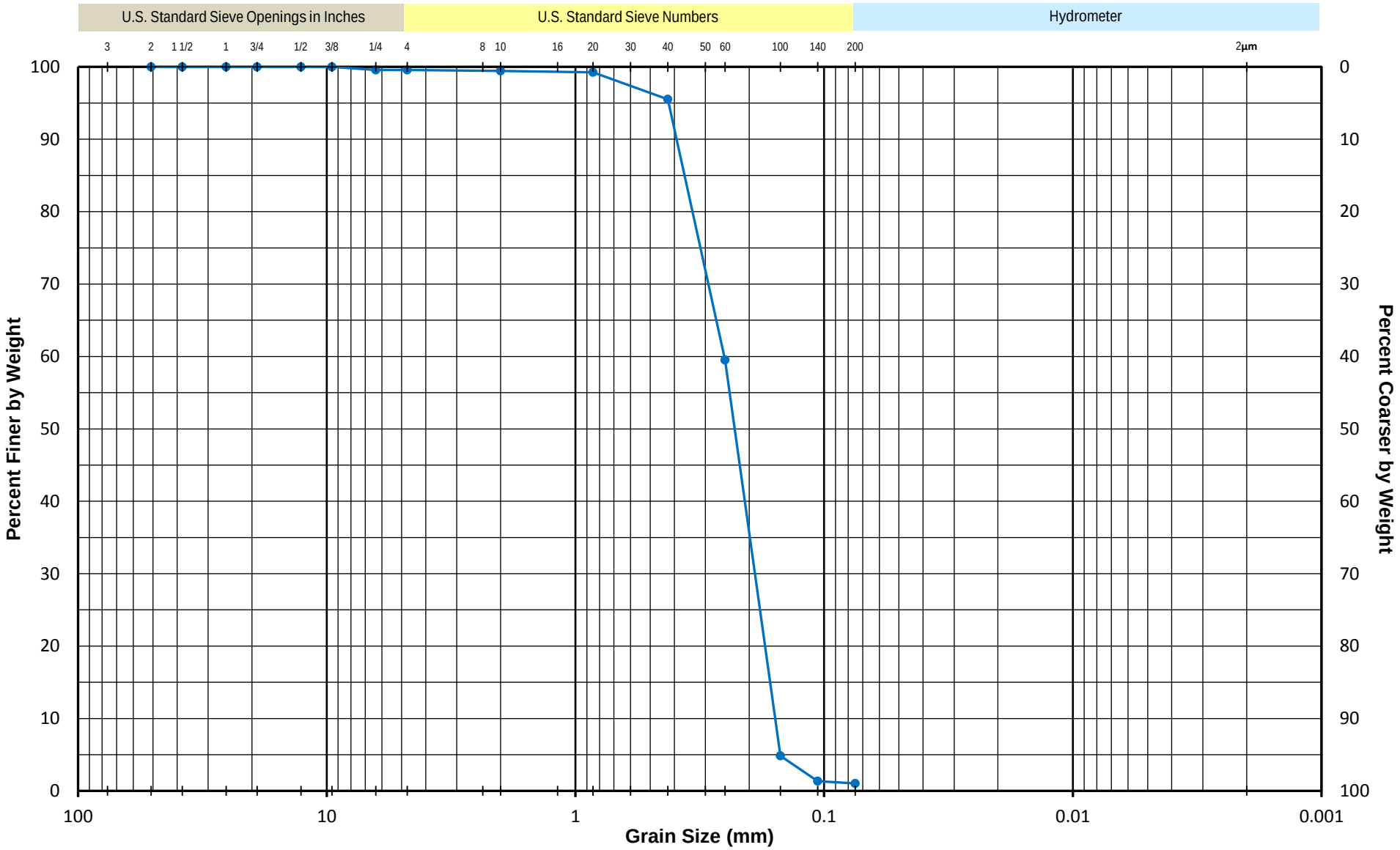
Acronym List and Table Key:

BD = blank spike duplicate
BS = blank spike
COC = chain of custody
CVAA = cold vapor atomic absorption
DUSR = data usability summary report
FD = field duplicate sample
ICP/MS = Inductively coupled / mass spectrometry
LB = laboratory blank
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
LR = laboratory replicate
MB = method blank
MS = matrix spike
MSD = matrix spike duplicate
N = normal field sample
NC = not calculated
ND = not detected
NJDEP = New Jersey Department of Environmental Protection
NYSDEC = New York State Department of Environmental Conservation
PQL = practical quantitation limit
QA = quality assurance
QAPP = quality assurance project plan
QC = quality control
RB = rinsate blank sample
RPD = relative percent difference
SDG = sample delivery group

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E Grain Size Curves

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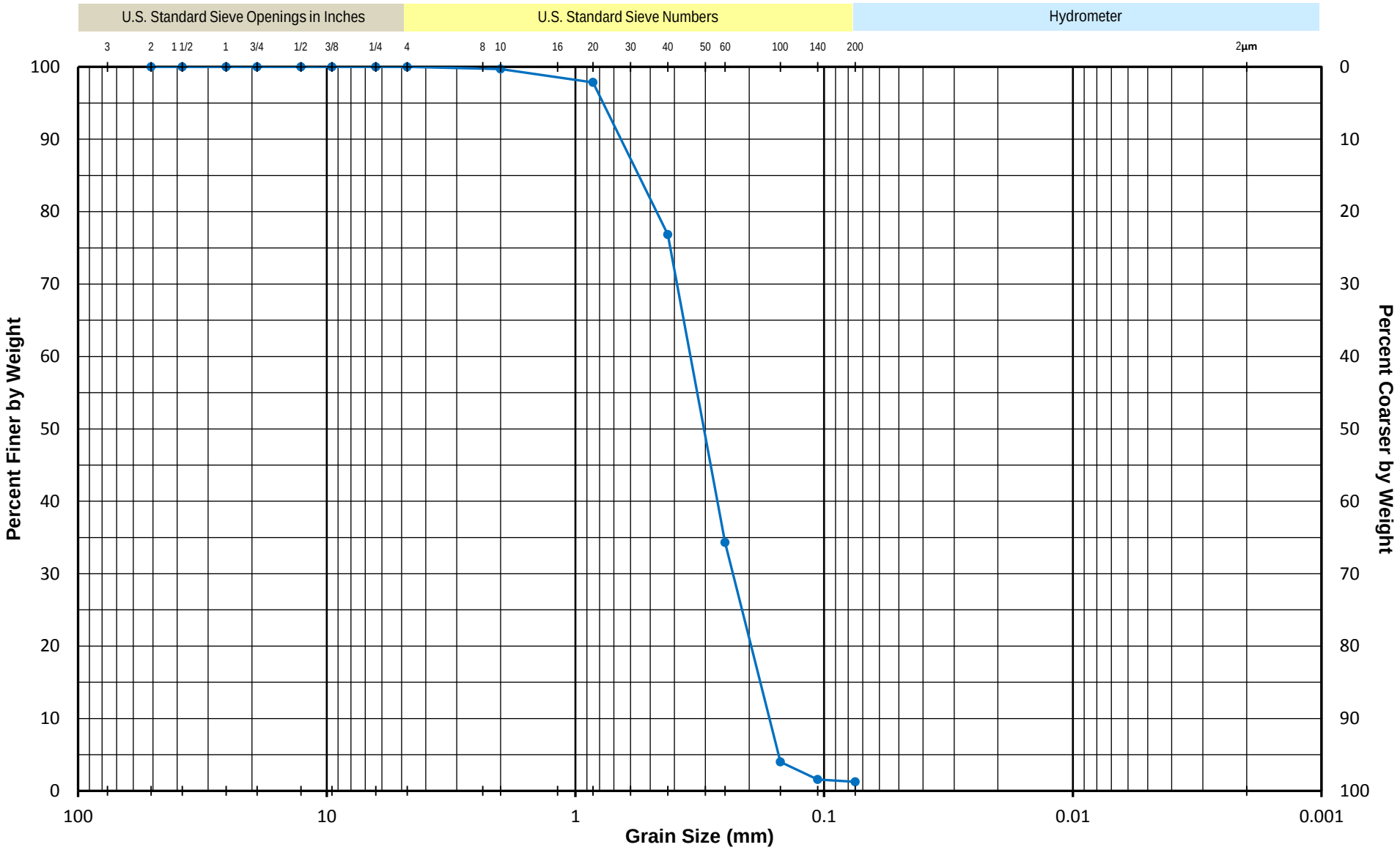


Test Depth	Core ID Section/Sample	Material Description
0.08 ft	VC93 0-2	Dark Grayish Brown and Very Dark Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

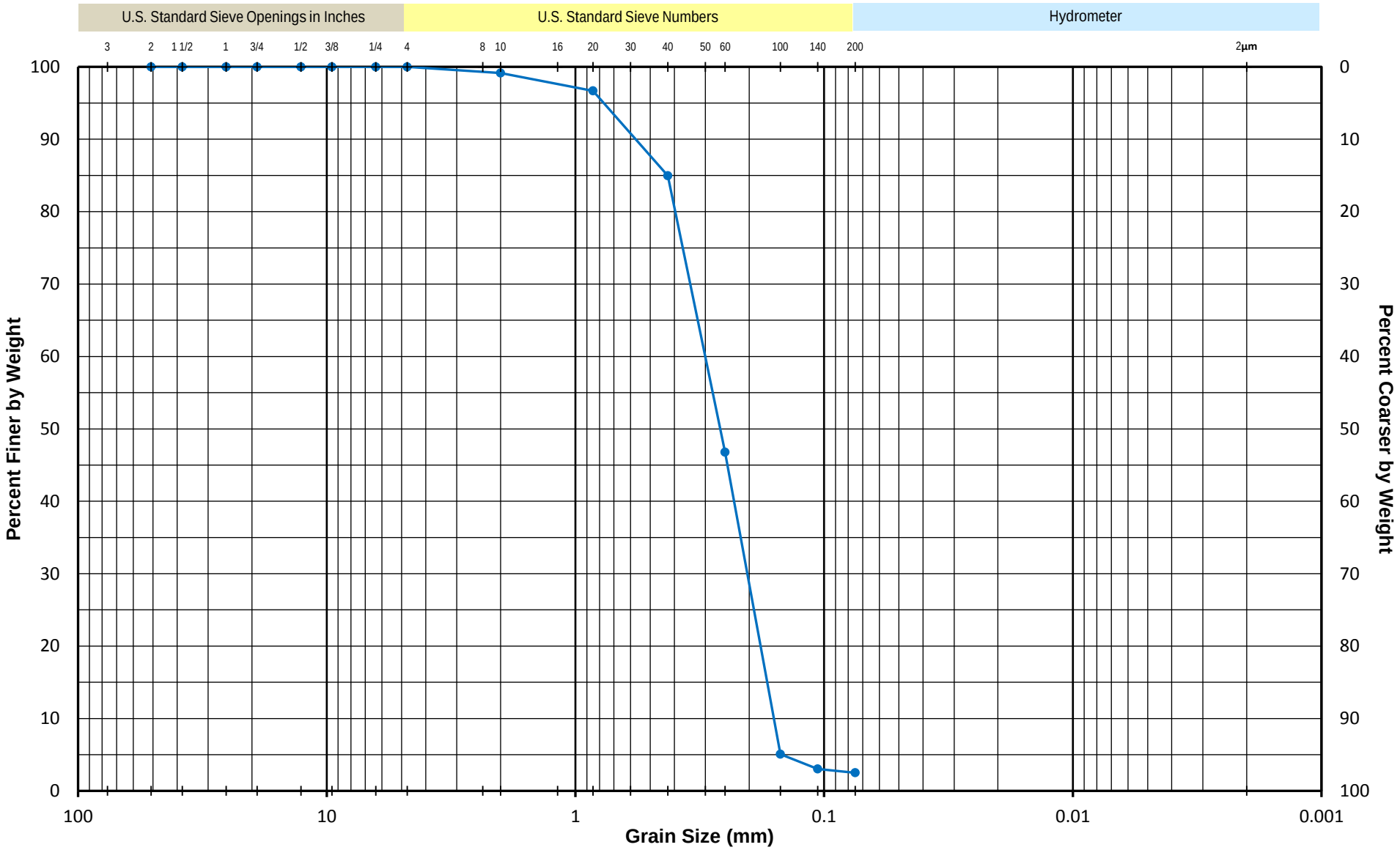


Test Depth	Core ID Section/Sample	Material Description
1.0 ft	VC93 0-2	Dark Olive Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

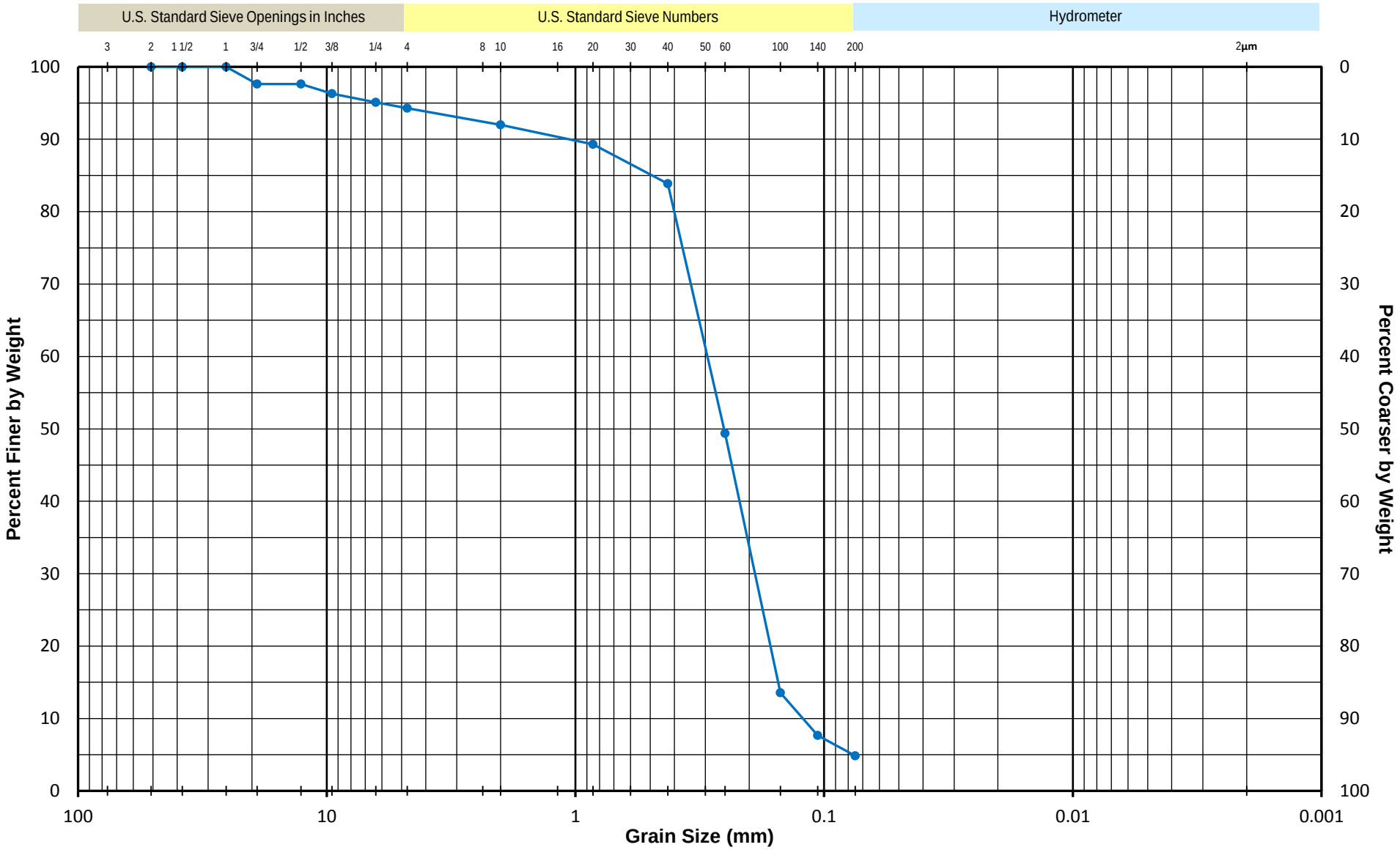


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC93 0-2	Dark Olive Gray and Gray Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

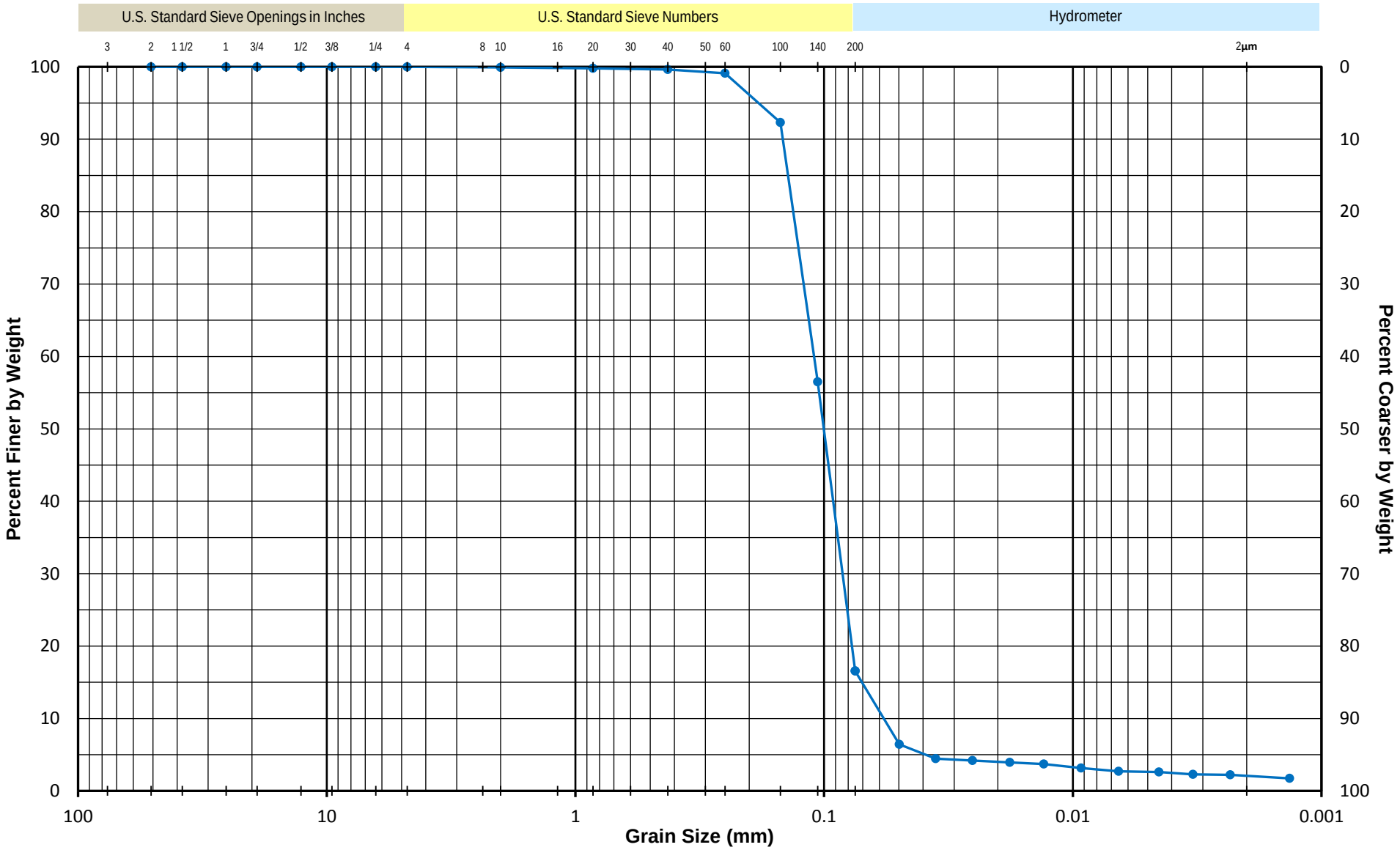


Test Depth	Core ID Section/Sample	Material Description
3.0 ft	VC93 2-4	Dark Grayish Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

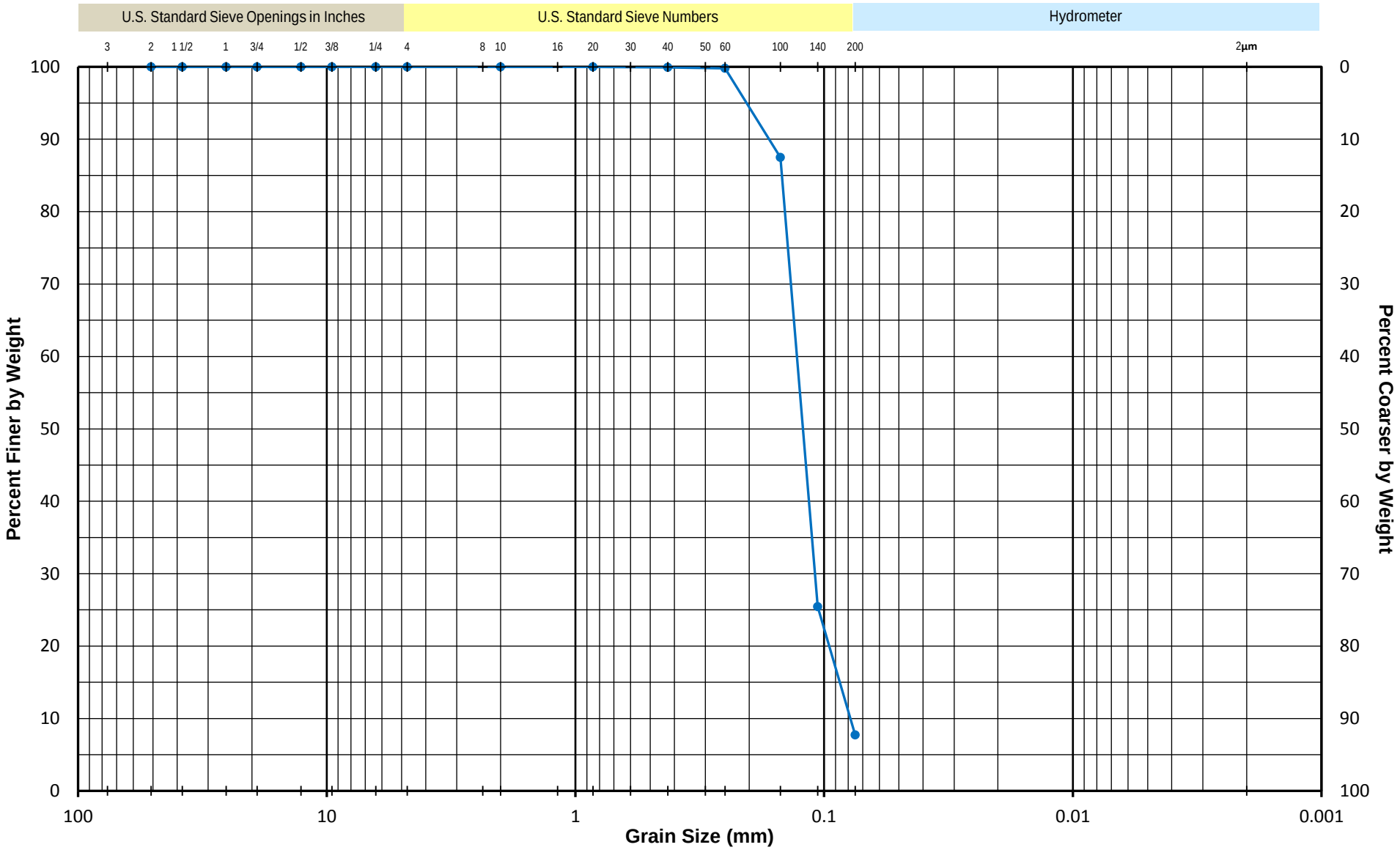


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC93 2-4	Light Brownish Gray Silty Fine Sand with dark yellowish brown seams

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

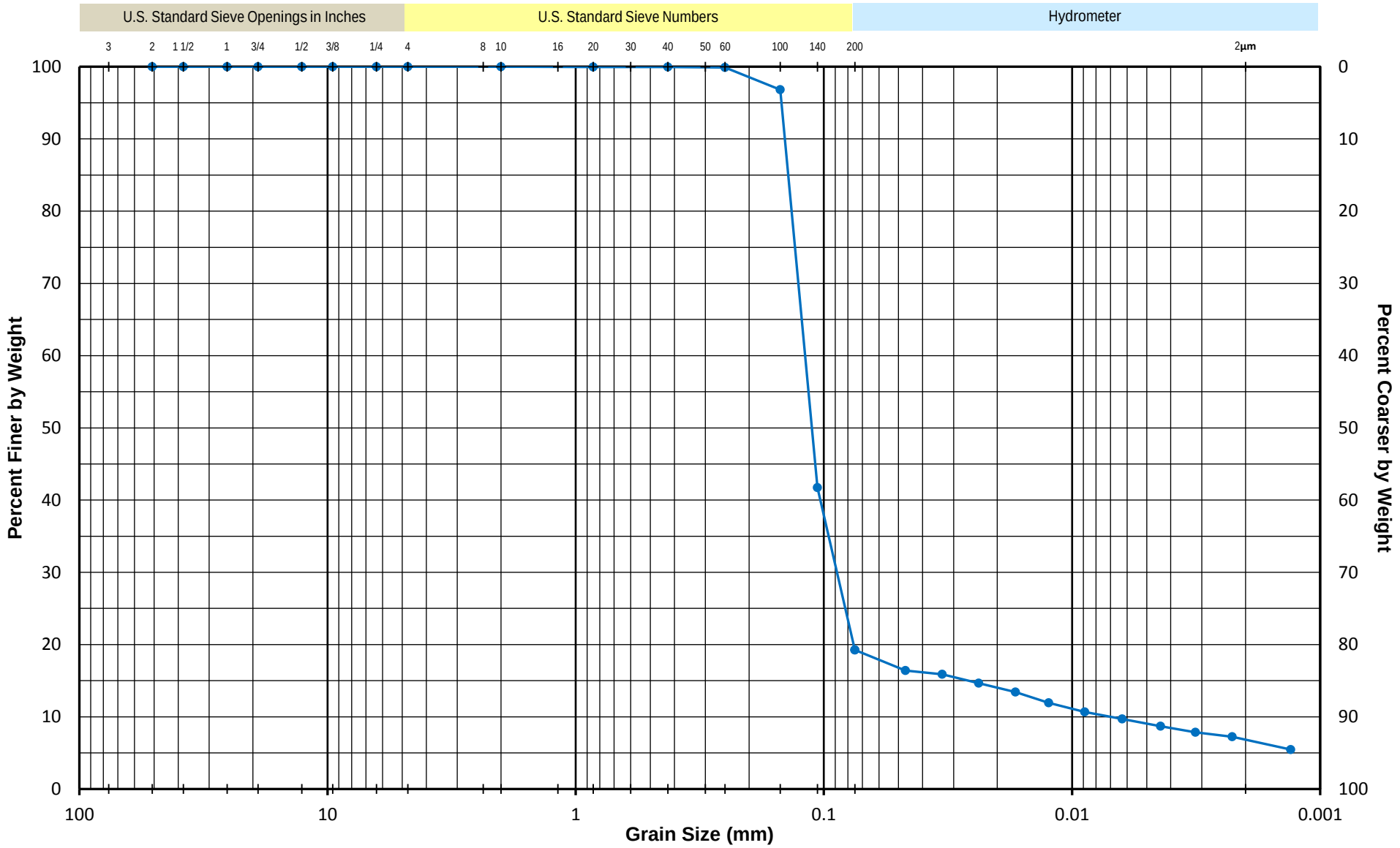


Test Depth	Core ID Section/Sample	Material Description
5.0 ft	VC93 4-6	Light Brownish Gray Fine Sand with silt

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

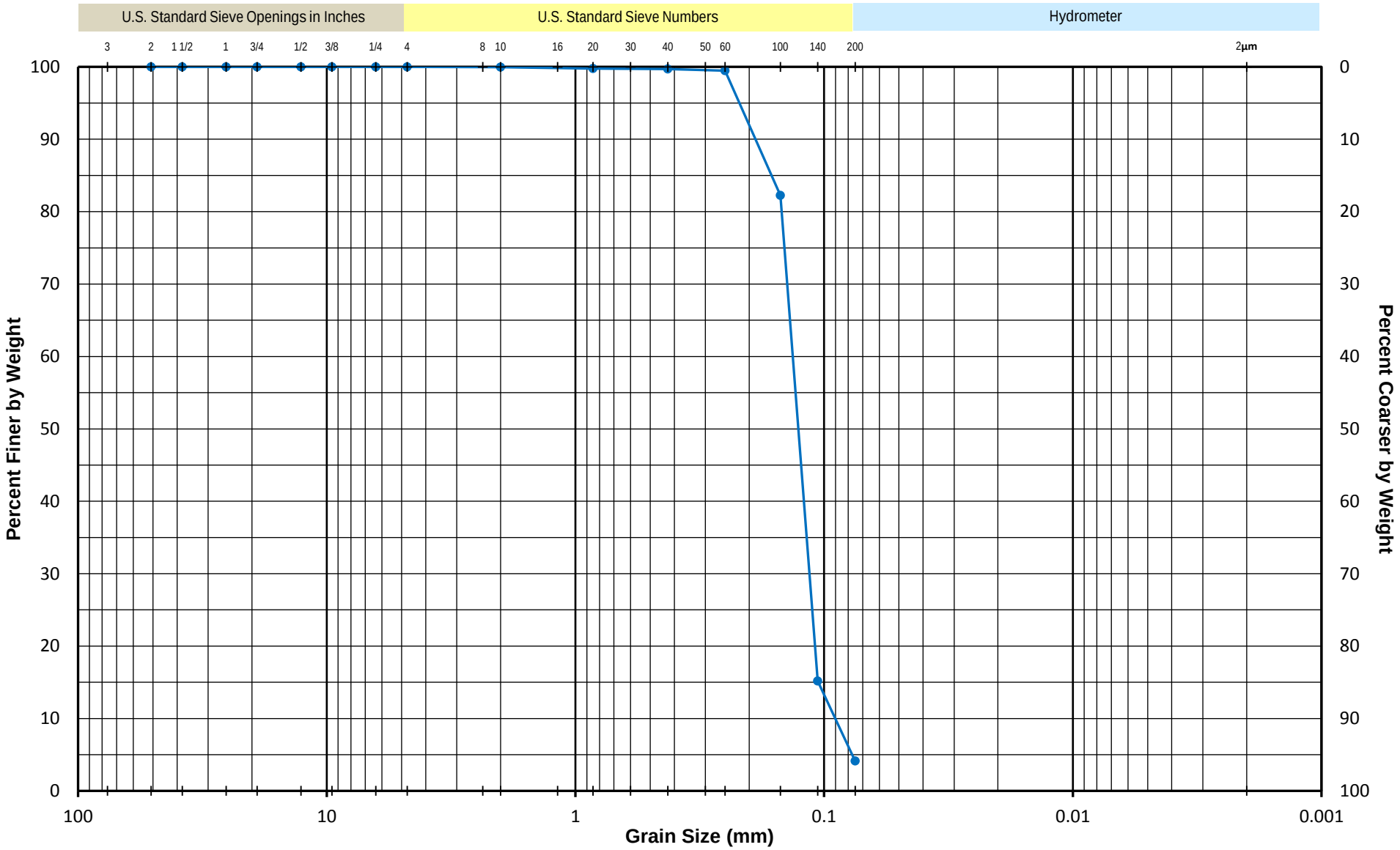


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC93 4-6	Light Brownish Gray Silty Fine Sand with clay seam and dark yellowish brown seams

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

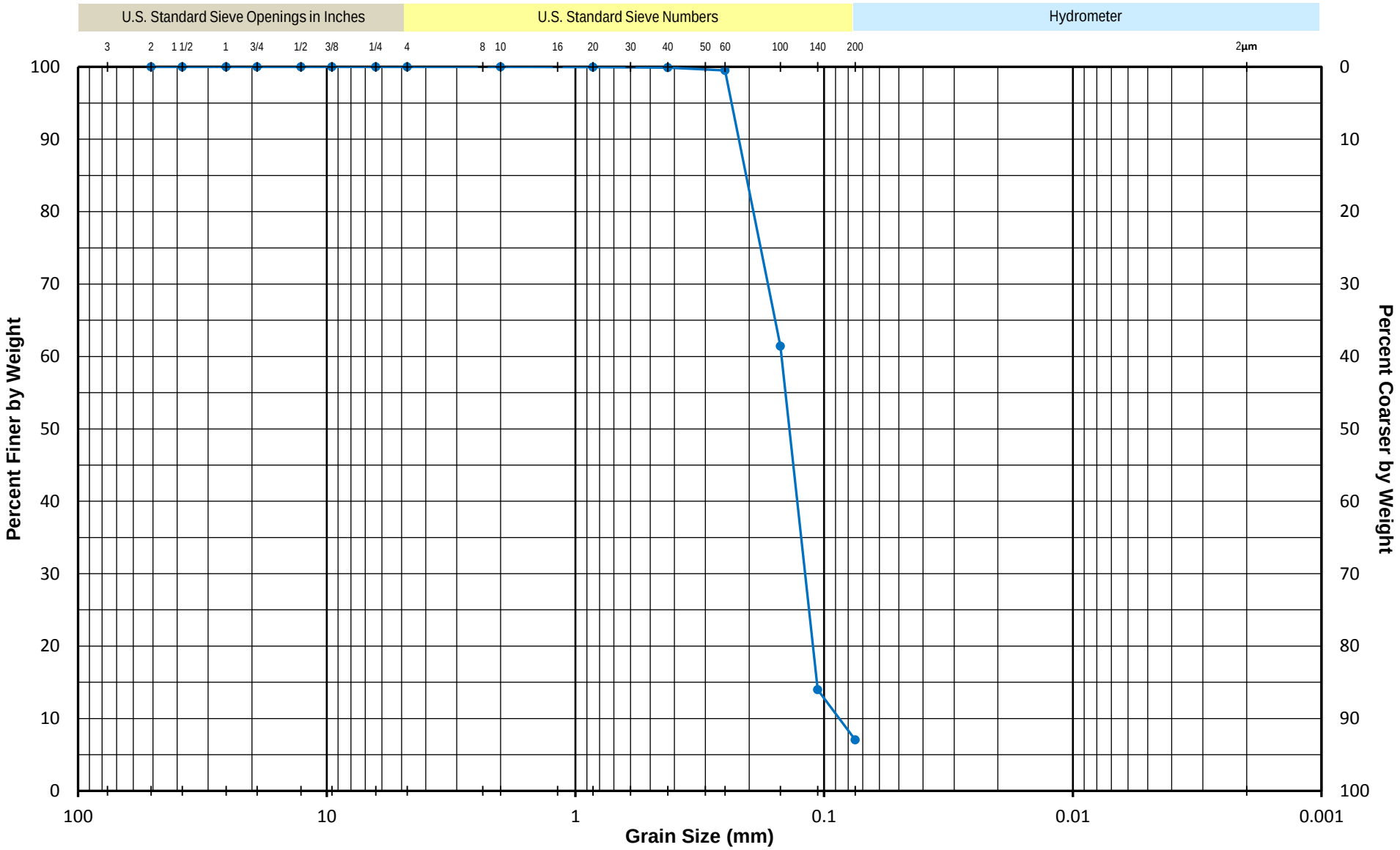


Test Depth	Core ID Section/Sample	Material Description
7.0 ft	VC93 6-8	Light Brownish Gray Fine Sand with gray seam with mica

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

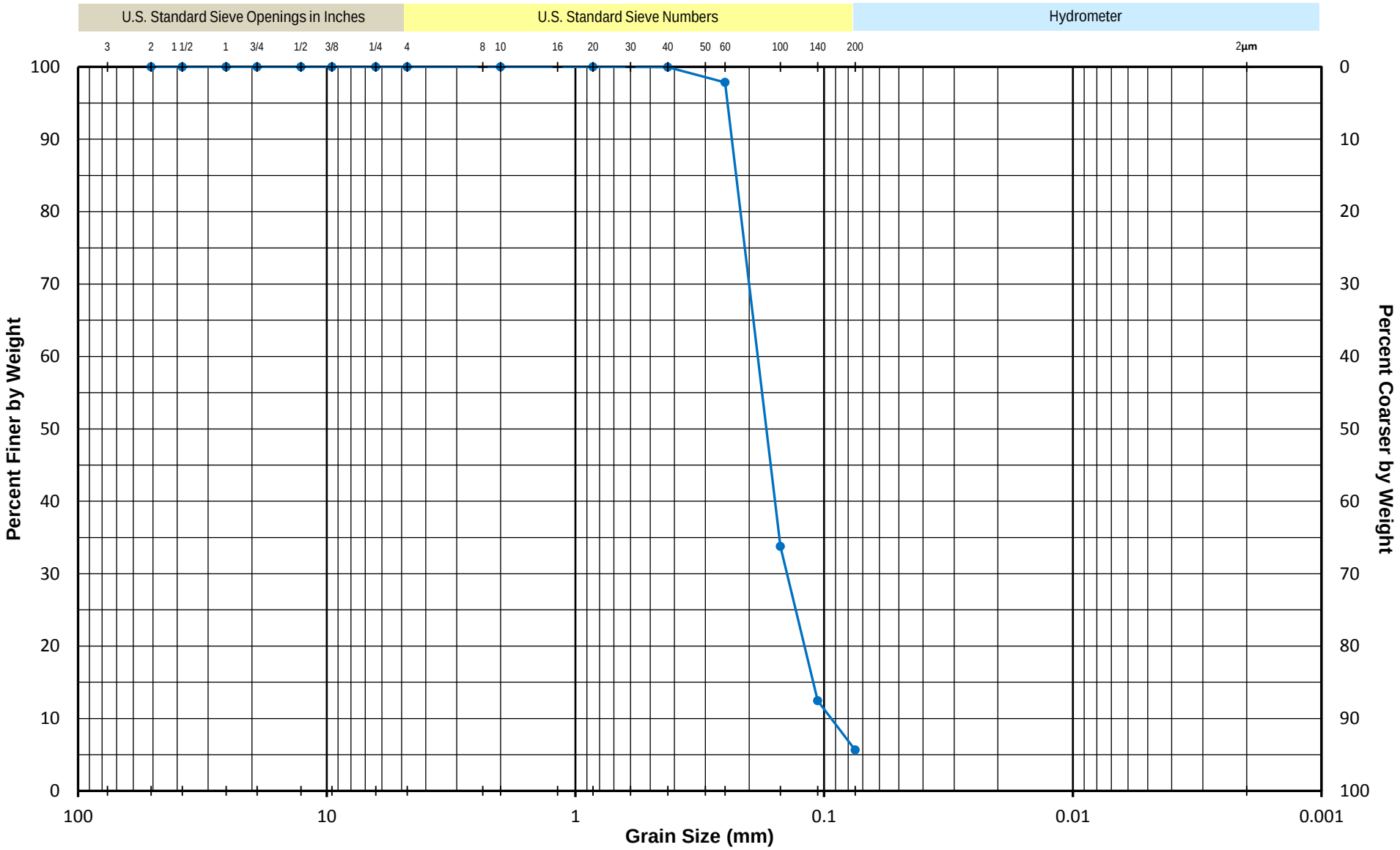


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC93 6-8	Light Brownish Gray Fine Sand with silt and gray seams with mica

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

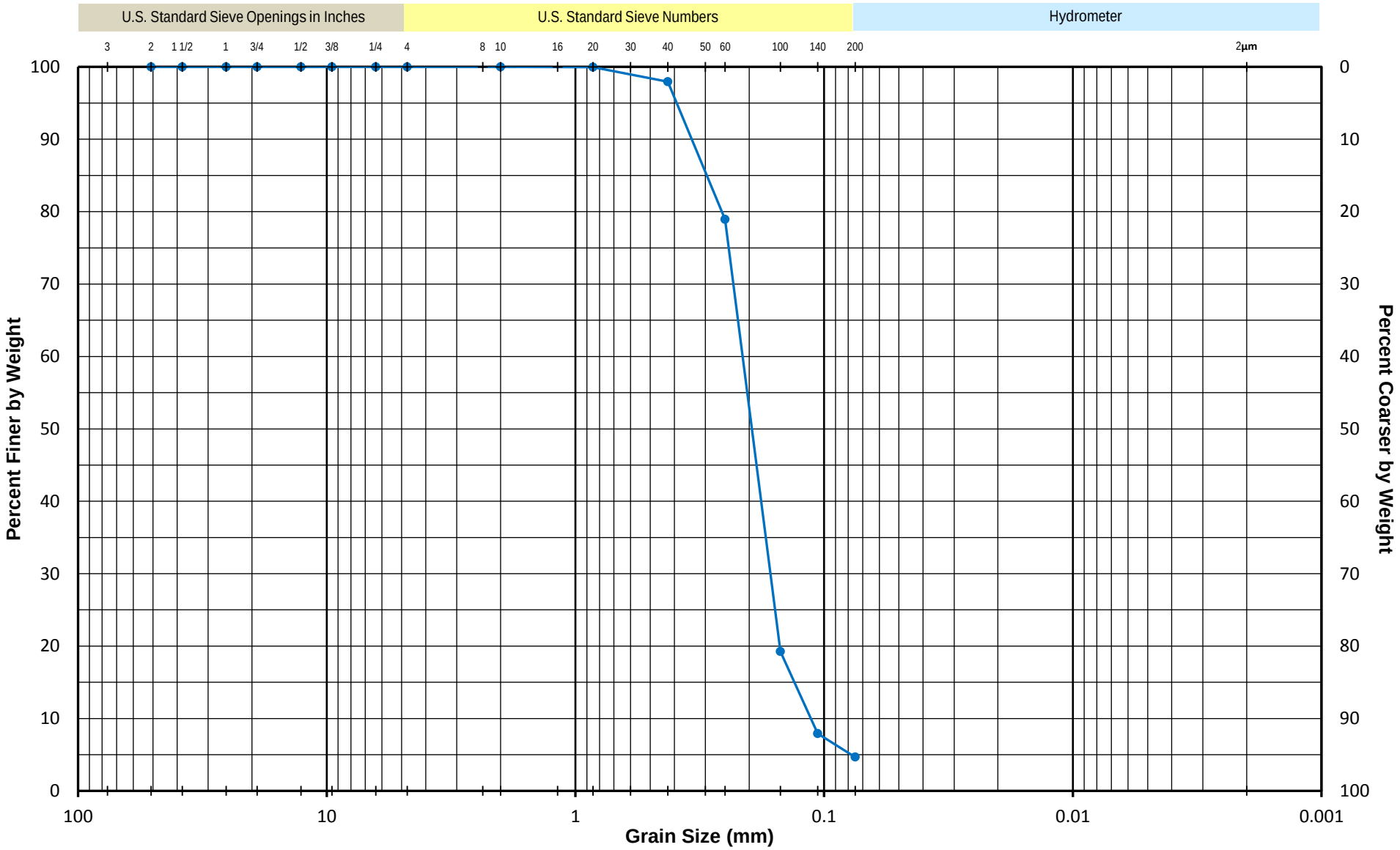


Test Depth	Core ID Section/Sample	Material Description
9.0 ft	VC93 8-10	Light Brownish Gray Fine Sand with gray silt seams with mica and dark yellowish brown seams

GRADATION CURVES

ASTM D-422, D-6913

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Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC93 8-10	Light Brownish Gray Fine Sand with gray seams with mica

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

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F Core Logs

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Alpine Ocean Seismic Survey, Inc
155 Hudson Avenue
Norwood, NJ 07648
Telephone: 201-768-8000

BORING NUMBER VC 01 Alt

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/14/16 **COMPLETED** 12/14/16
WATER DEPTH -8.71 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.48
DRILLING METHOD Vibracore
CORE RECOVERY: 15.92
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:912889.51 Y:110112.09
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	1.2			115
		(CH) Very soft black Clay;non plastic						
		(CH) Black Clay with odor and organics	VC 2	100	1.2			
5		(CH) Soft brown silty Clay; thinly bedded with black silty Clay; non plastic				0	0	
		(CH) Unsplit	VC 3	100	1.7			20
		(CH) Soft gray Clay; non plastic				0.1	0	
		(CH) Unsplit	VC 4	100	1.1			28
10		(CH) Soft gray Clay with trace shell fragments; non plastic				0.1	0	
		(CH) Unsplit	VC 5	100	1.1			26
		(OLSH) Soft gray-brown Clay with trace shell fragments; non plastic				0	0	
		(OLSH) Soft grayish brown Clay with few whole shells; non plastic				0.1	0	
15						0.1	0	
						0.25	0	

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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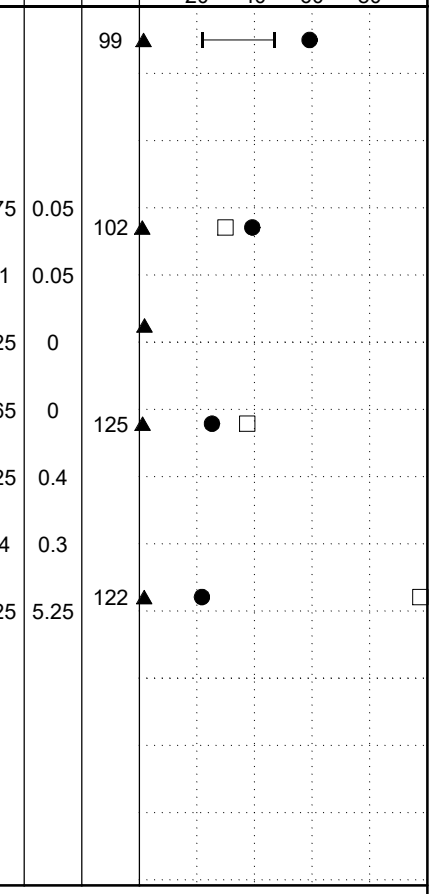
Alpine Ocean Seismic Survey, Inc
155 Hudson Avenue
Norwood, NJ 07648
Telephone: 201-768-8000

BORING NUMBER VC 02

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/5/16 **COMPLETED** 12/5/16
WATER DEPTH -8.23 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.08
DRILLING METHOD Vibracore
CORE RECOVERY: 9.29
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:914612.32 Y:110957.65
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CL) Very soft, gray Clay, nonplastic and noncohesive	VC 1	100	1.4			99
		(SC) Very soft, light brown silty Clay with trace fine sand	VC 2	100	1.0	0.75	0.05	102
5		(SW) Very loose, light brown medium Sand and cobbles, well graded	VC 3	100	1.8	0.1	0.05	
		(SC) Loose, dark gray fine Sand with trace silt				0.25	0	
		(CH) Very stiff, dry, very light gray Clay; Cohesive and high plasticity.	VC 4	100	1.1	0.65	0	125
						1.25	0.4	
						1.4	0.3	
			VC 5	100	1.7	2.25	5.25	122
10								

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
□ FINES CONTENT (%) □ 20 40 60 80



Bottom of borehole at 13.1 feet.

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Alpine Ocean Seismic Survey, Inc
155 Hudson Avenue
Norwood, NJ 07648
Telephone: 201-768-8000

BORING NUMBER VC 03 Alt

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/5/16 **COMPLETED** 12/5/16
WATER DEPTH -8.86 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.49
DRILLING METHOD Vibracore
CORE RECOVERY: 11.83
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:916536.59 Y:112053.47
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Soft, gray Clay; noncohesive, nonplastic	VC 1	100	1.9			91
		(CH) Dark greenish gray Clay	VC 2	100	1.5			91
5		(CL) Soft, gray Clay; noncohesive, nonplastic						
		(CL) Dark greenish gray Clay, trace shell fragments	VC 3	100	1.2			92
		(CL) Soft, gray Clay; noncohesive, nonplastic						
		(CH) Dark greenish gray Clay, trace shell fragments	VC 4	100	1.5			92
10		(CL) Soft, gray Clay; noncohesive, nonplastic						
		(CL) Dark greenish gray Clay, trace shell fragments	VC 5	100	1.0			92
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 19.5 feet.

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Alpine Ocean Seismic Survey, Inc
155 Hudson Avenue
Norwood, NJ 07648
Telephone: 201-768-8000

BORING NUMBER VC 04

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/4/16 **COMPLETED** 12/4/16
WATER DEPTH -7.62 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 17.95
DRILLING METHOD Vibracore
CORE RECOVERY: 11.83
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:918089.31 Y:112938.49
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	2.1			88
		(CH) Unsplit	VC 2	100	0.9			91
		(CH) Unsplit	VC 3	100	0.9			91
		(CH) Black Clay with trace shell fragments and odor	VC 4	100	1.7			93
		(CH) Black Clay with trace shell fragments and odor	VC 5	100	1.4			92
5		(CH) Greenish black Clay with trace shell fragments	VC 6	100	0.9			91
		(CH) Greenish black Clay with trace shell fragments	VC 7	100	1.5			93
		(CL) Soft, medium gray Clay, noncohesive, nonplastic				0	0.05	
		(CH) Dark greenish gray Clay with trace shell fragments	VC 8	100	1.2			89
10		(CL) Soft, medium gray Clay, noncohesive, nonplastic				0	0.1	
		(CH) Dark greenish gray Clay with trace shell fragments	VC 9	100	1.3			92
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 18.0 feet.

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Alpine Ocean Seismic Survey, Inc
155 Hudson Avenue
Norwood, NJ 07648
Telephone: 201-768-8000

BORING NUMBER VC 05

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/2/16 **COMPLETED** 12/2/16
WATER DEPTH -11.3 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 17.15
DRILLING METHOD Vibracore
CORE RECOVERY: 11.17
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:919825.29 Y:113928.17
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	2.3			91
		(CH) Unsplit	VC 2	100	2.2			89
		(CL) very soft, dark gray Clay; noncohesive, nonplastic				0	0	
		(CH) Unsplit	VC 3	100	1.6			89
		(CH) Unsplit	VC 4	100	1.6			89
5		(CH) Unsplit	VC 5	100	1.6			93
		(CH) Unsplit	VC 6	100	1.4	0	0	90
		(CL) very soft, dark gray Clay; noncohesive, nonplastic				0	0.05	
						0	0	
		(CH) Unsplit	VC 7	100	1.6			93
10		(CH) Unsplit	VC 8	100	1.6			91
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 17.2 feet.

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BORING NUMBER VC 06

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/28/16 **COMPLETED** 11/28/16
WATER DEPTH -13.93 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.49
DRILLING METHOD Vibracore
CORE RECOVERY: 11.08
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:921564.31 Y:114918.1
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	2.1			82
		(CL) Very soft, black Clay; noncohesive, nonplastic						
		(CH) Unsplit	VC 2	100	1.6			85
5		(CL) Very soft, light gray Clay with few shells; noncohesive, nonplastic						
		(CH) Unsplit	VC 3	100	1.4			89
		(CL) Very soft, light gray Clay with few shells; noncohesive, nonplastic						
10		(CH) Unsplit	VC 4	100	1.4			90
		(CH) Unsplit	VC 5	100	1.4			
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 07

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/28/16 **COMPLETED** 11/28/16
WATER DEPTH -13.93 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.48
DRILLING METHOD Vibracore
CORE RECOVERY: 10.75
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:923299.9 Y:115910.19
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	1.6			83
		(CL) Very soft, black Clay; noncohesive, nonplastic						
		(CH) Unsplit	VC 2	100	1.2			91
5		(CL) Very soft, light gray Clay with trace shells; noncohesive, nonplastic						
		(CH) Unsplit	VC 3	100	1.4			90
		(CL) Very soft, light gray Clay; noncohesive, nonplastic						
10		(CH) Unsplit	VC 4	100	1.3			92
		(CH) Unsplit	VC 5	100	1.4			92
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 08

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/2/16 **COMPLETED** 12/2/16
WATER DEPTH -15.44 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.5
DRILLING METHOD Vibracore
CORE RECOVERY: 10.67
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:925038.28 Y:116897.95
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Very soft, greenish gray Clay with trace shell fragments; noncohesive, nonplastic	VC 1	100	1.4			87
		(CH) Very soft, greenish gray Clay; noncohesive, nonplastic				0	0.05	
						0	0	
						0	0	
5			VC 2	100	1.2	0	0	90
		(CH) Soft, greenish gray Clay; noncohesive, nonplastic				0	0	
						0	0	
			VC 3	100	1.1	0	0.03	86
						0	0.04	
10			VC 4	100	1.2	0	0.05	94
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 09

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/1/16 **COMPLETED** 12/1/16
WATER DEPTH -12.43 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 17
DRILLING METHOD Vibracore
CORE RECOVERY: 11.79
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:926774.97 Y:117889.99
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	1.6			86
		(CL) Very soft, greenish gray Clay; noncohesive, nonplastic						
		(CH) Very soft, greenish gray Clay; noncohesive, nonplastic	VC 2	100	1.7			86
5		(CH) Very soft, greenish black Clay; noncohesive, nonplastic						
		(CH) Very soft, greenish gray Clay; noncohesive, nonplastic	VC 3	100	1.5			89
		(CH) Soft, greenish gray Clay; noncohesive, nonplastic						
10		(CH) Soft, greenish gray Clay; noncohesive, nonplastic	VC 4	100	1.6			90
		(CH) Soft, greenish gray Clay; noncohesive, nonplastic						
		(CH) Soft, greenish gray Clay; noncohesive, nonplastic	VC 5	100	1.3			90
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 17.0 feet.

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BORING NUMBER VC 10

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/1/16 **COMPLETED** 12/1/16
WATER DEPTH -8.81 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.3
DRILLING METHOD Vibracore
CORE RECOVERY: 11.63
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:928514.39 Y:118877.43
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Loose, dark gray medium Sand with trace pebbles; poorly sorted	VC 1	100	2.1			113
		(SP) Loose, reddish brown medium Sand with trace pebbles up to 1"						
		Rock						
		(SP) Loose, brownish orange coarse Sand with large cobbles >1"; poorly sorted	VC 2	100	2.1			118
		(SP) Loose, brownish orange coarse Sand						
5		(SW) Loose, brownish orange coarse Sand with some large cobbles >1"; poorly sorted						
			VC 3	100	2.4			114
			VC 4	100	3.2			126
10		(GW-GC) Medium dense, large cobbles in a red coarse sandy Clay matrix						
		(SP) Loose, brown coarse Sand	VC 5	100	4.1			116
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.3 feet.

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BORING NUMBER VC 11

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/25/16 **COMPLETED** 11/25/16
WATER DEPTH -11.53 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.58
DRILLING METHOD Vibracore
CORE RECOVERY: 10.58
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:930250.82 Y:119866.2
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Very loose, dark brown medium Sand with trace shell fragments and pebbles	VC 1	100	2.1			106
		(SP-SM) Very loose, dark gray clayey Sand with trace pebbles; noncohesive, nonplastic						
		(SPG) Loose, brown medium Sand with trace pebbles						
		(SP) Medium dense, light gray, medium to coarse Sand with trace cobbles	VC 2	100	1.7			113
5		(SW) Loose, very coarse Sand and gravel in a red silty matrix; poorly sorted						
		(SW) Loose coarse Sand and gravel in a red sandy silt matrix	VC 3	100	3.2			120
		(SP) Loose, light grayish brown medium Sand	VC 4	100	4.7			104
10								
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.6 feet.

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BORING NUMBER VC 12

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/25/16 **COMPLETED** 11/25/16
WATER DEPTH -14.43 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.9
DRILLING METHOD Vibracore
CORE RECOVERY: 12.33
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:931990.72 Y:120858.99
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Very soft, greenish gray Clay with trace shell fragments and coarse sand; nonplastic, noncohesive	VC 1	100	1.4			98
		(SP-SM) Loose, dark gray Silt with some gravel; noncohesive, nonplastic	VC 2	100	1.4			119
		(GW-GM) Loose, dark gray Gravel in a black silty sand matrix; poorly sorted						
5		(SP) Medium dense, yellowish red coarse Sand with some gravel; poorly sorted	VC 3	100	4.5			118
		Large Rock						
		(SP) Medium dense, reddish brown medium Sand; well sorted	VC 4	100	9.9			111
10		(SW-SC) Medium dense, fine brown Sand; few light brown clay laminations	VC 5	100	4.3			119
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 15.9 feet.

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BORING NUMBER VC 13

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/25/16 **COMPLETED** 11/25/16
WATER DEPTH -15.76 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.21
DRILLING METHOD Vibracore
CORE RECOVERY: 12.33
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:933727.16 Y:121847.92
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	1.6			91
		(CH) Very soft, greenish gray Clay with trace shell fragments; nonplastic, noncohesive						
		(CH) Unsplit	VC 2	100	1.6			92
5		(CL) Very soft, greenish gray Clay; noncohesive, nonplastic						
		(CH) Unsplit	VC 3	100	1.7			94
		(CL) Soft, light gray Clay; cohesive, nonplastic						
		(CH) Unsplit	VC 4	100	1.7			91
10		(CL) Soft, light gray Clay; cohesive, nonplastic						
		(CH) Unsplit	VC 5	100	1.4			91
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.2 feet.

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BORING NUMBER VC 14

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/25/16 **COMPLETED** 11/25/16
WATER DEPTH -16.69 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.5
DRILLING METHOD Vibracore
CORE RECOVERY: 14.38
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:934710.63 Y:122409.57
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	2.8			91
		(CH) Very soft, greenish gray Clay with trace shell fragments; nonplastic, noncohesive						
			VC 2	100	2.0			89
5								
			VC 3	100	2.4			86
			VC 4	100	3.6			
		(CH) Very soft, greenish gray Clay with trace shell fragments; nonplastic, noncohesive	VC 5	100	2.3			
10		(CH) Very soft, greenish gray Clay with trace shell fragments; nonplastic, noncohesive						
		(CH) Very soft, greenish gray Clay with trace shell fragments; nonplastic, noncohesive	VC 6	100	1.9			
		(CH) Very soft, greenish gray Clay with trace shell fragments; nonplastic, noncohesive						
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 15

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/26/16 **COMPLETED** 11/26/16
WATER DEPTH -18.01 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.5
DRILLING METHOD Vibracore
CORE RECOVERY: 15.96
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:935230.75 Y:122705.16
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(OL) Organic Shell hash	VC 1	100	0.2			
		(CH) Unsplit		100	1.8			92
		(CL) Very soft, greenish gray Clay with trace shell fragments; nonplastic, noncohesive	VC 2					
		(CL) Very soft dark gray Clay; noncohesive, nonplastic						
		(CH) Dark greenish gray Clay, with shell fragments	VC 3	100	1.4			93
5		(CH) Very soft dark gray Clay; noncohesive, nonplastic						
		(CH) Dark greenish gray Clay, with shell fragments	VC 4	100	1.2			91
		(CH) Very soft dark gray Clay; noncohesive, nonplastic						
		(CL) Dark greenish gray Clay	VC 5	100	1.2			92
10		(CL) Dark greenish gray Clay						
		(CH) Dark greenish gray Clay	VC 6	100	1.4			93
		(CL) Dark greenish gray Clay	VC 7	100	1.2			95
		(CL) Very soft dark gray Clay; noncohesive, nonplastic				0	0.075	
15						0	0.05	

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
☐ FINES CONTENT (%) ☐ 20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 16

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/26/16 **COMPLETED** 11/26/16
WATER DEPTH -29.81 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.5
DRILLING METHOD Vibracore
CORE RECOVERY: 16.42
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:935719.46 Y:122985.05
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	2.1			76
		(CL) Very soft black Clay; nonplastic, noncohesive						
		(CL) Black Clay with odor	VC 2	100	1.8			84
5		(CH) Very soft black Clay thinly bedded with grayish green clay; nonplastic, noncohesive						
		(CL) Black Clay with odor	VC 3	100	1.4			86
		(CH) Very soft black Clay thinly bedded with grayish green clay; nonplastic, noncohesive						
		(CH) Black Clay with odor	VC 4	100	2.0			81
10		(CH) Very soft black Clay thinly bedded with grayish green clay; nonplastic, noncohesive						
		(CH) Black Clay	VC 5	100	2.2			83
		(CH) Very soft black Clay thinly bedded with grayish green clay; nonplastic, noncohesive						
		(CH) Black Clay with odor	VC 6	100	1.6			80
15		(CH) Very soft black Clay thinly bedded with grayish green clay; nonplastic, noncohesive						

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 17

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/26/16 **COMPLETED** 11/26/16
WATER DEPTH -32.74 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.47
DRILLING METHOD Vibracore
CORE RECOVERY: 13.04
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:936446.7 Y:123397.59
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(MH) Unsplit	VC 1	100	2.1			79
		(CL) Very soft black clay mud; noncohesive, nonplastic				0	0	
		(MH) Unsplit	VC 2	100	1.4			82
5		(CL) Very soft, greenish gray clay mud bedded with black clay; noncohesive, nonplastic				0	0.05	
						0	0.08	
		(CH) Unsplit	VC 3	100	1.4			86
		(CL) Very soft, greenish gray clay mud bedded with black clay; noncohesive, nonplastic				0	0	
						0	0.05	
10		(CH) Unsplit	VC 4	100	1.4			92
		(CL) Very soft, greenish gray clay mud bedded with black clay; noncohesive, nonplastic				0	0.05	
						0	0.09	
		(CH) Unsplit	VC 5	100	1.4			93
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 18

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/26/16 **COMPLETED** 11/26/16
WATER DEPTH -17.28 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.5
DRILLING METHOD Vibracore
CORE RECOVERY: 16.58
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:936944.92 Y:123677.54
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	1.9			90
		(CH) Very soft, greenish gray Clay with trace shell fragments; cohesive, low plasticity						
		(CH) Very soft, greenish gray Clay with trace shell fragments; cohesive, low plasticity	VC 2	100	2.0			90
5		(CH) Very soft, dark gray clay laminated with brown clay; noncohesive, nonplastic				0	0.03	
						0	0.09	
		(CH) Very soft, dark gray clay with trace peat; noncohesive, nonplastic	VC 3	100	1.5			
		(CH) Very soft, dark gray clay with trace peat; noncohesive, nonplastic				0	0.07	
						0	0.10	
10		(CH) Very soft, dark gray clay with trace peat; noncohesive, nonplastic	VC 4	100	2.3			
		(CH) Very soft, dark gray clay with trace peat; noncohesive, nonplastic				0	0.06	
						0	0.12	
		(CH) Very soft, dark gray clay with trace peat; noncohesive, nonplastic	VC 5	100	2.6			
		(CH) Very soft, dark gray clay with trace peat; noncohesive, nonplastic				0	0.10	
		(CH) Very soft, dark gray clay with trace peat; noncohesive, nonplastic	VC 6	100	1.5			
15		(CH) Very soft, dark gray clay with trace peat; noncohesive, nonplastic				0	0.13	
						0	0.13	

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 19

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/26/16 **COMPLETED** 11/26/16
WATER DEPTH -18.94 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.48
DRILLING METHOD Vibracore
CORE RECOVERY: 11.67
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:937794.14 Y:124069.86
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Dark greenish gray Clay wih trace shell fragments	VC 1	100	1.6			95
		(CH) Very soft greenish gray Clay with trace shell fragments; noncohesive, nonplastic						
		(CH) Very soft greenish gray Clay with trace shell fragments; noncohesive, nonplastic	VC 2	100	2.4			88
5		(CH) Very soft greenish gray Clay with trace shell fragments; noncohesive, nonplastic						
		(CH) Dark greenish gray Clay with trace shell fragments	VC 3	100	2.2			96
		(CH) Dark greenish gray Clay with trace shell fragments						
		(CH) Dark greenish gray Clay with trace fine sand partings	VC 4	100	2.4			97
10		(CH) Dark greenish gray Clay with trace shell fragments						
		(CH) Dark greenish gray Clay with trace shell fragments	VC 5	100	1.5			93
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 20

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/27/16 **COMPLETED** 11/27/16
WATER DEPTH -17.8 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.49
DRILLING METHOD Vibracore
CORE RECOVERY: 14.50
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:939104.16 Y:124461.33
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0	(OL) Shell hash	VC 1	100	0.2			106
	(CL) Lean Clay with Sand, shell content and 2.8% organic material	VC 2	100	2.2			
	(CL-ML) Very soft, dark gray silty clay with trace shell hash; cohesive, low plasticity				0	0	
	(CH) Dark greenish gray Clay	VC 3	100	1.7			100
5	(CH) Very soft, dark gray clay with trace shell hash; cohesive, nonplastic				0	0.05	
					0	0.08	
	(CH) Dark greenish gray Clay	VC 4	100	2.3			96
	(CH) Very soft, dark gray clay with trace shell hash; cohesive, nonplastic				0	0.12	
					0	0.10	
	(CH) Dark greenish gray Clay with shell fragments	VC 5	100	2.0			97
10	(CH) Very soft, dark gray clay with trace shell hash; cohesive, nonplastic				0	0.06	
	(CH) Dark greenish gray Clay	VC 6	100	1.6			91
	(CH) Very soft, dark gray clay with trace shell hash; cohesive, nonplastic				0	0.11	
					0	0.13	
15					0	0.02	

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
□ FINES CONTENT (%) □ 20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 21

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/27/16 **COMPLETED** 11/27/16
WATER DEPTH -16.31 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.09
DRILLING METHOD Vibracore
CORE RECOVERY: 11.58
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:941097.33 Y:124619.57
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0	(OL) Shell hash layer	VC 1	100	0.5			
	(SM) Dark gray silty Sand with large shells and shell hash; noncohesive	VC 2	100	2.2	0.1	0.09	121
	(SM) Dark gray silty Sand with trace shell hash; very soft; noncohesive				0	0.09	
	(CL-ML) Dark gray silty Clay, trace shell hash; very soft; very low plasticity	VC 3	100	2.6	0.25	0.09	106
	(SC) Dark gray silty clay, trace shell hash; soft, cohesive; nonplastic				0	0.06	
5	(CL) Dark gray silty clay, trace shell hash and trace peat; soft; cohesive; nonplastic				0	0.05	
		VC 4	100	2.2	0	0.075	92
					0	0.1	
	(SC) Dark gray silty clay with trace fine sand and shells and few lenses of dense shell hash; soft; cohesive; nonplastic				0.15	0.08	
		VC 5	100	2.1	0	0.06	99
10					0.15	0.1	
		VC 6	100	1.5	0.35	0.11	109
15							

▲ SECONDS PER FOOT ▲ (20 40 60 80)
PL MC LL (20 40 60 80)
☐ FINES CONTENT (%) ☐ (20 40 60 80)

Bottom of borehole at 19.1 feet.

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BORING NUMBER VC 22

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/27/16 **COMPLETED** 11/27/16
WATER DEPTH -17.66 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.87
DRILLING METHOD Vibracore
CORE RECOVERY: 14.00
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:943095.96 Y:124553.81
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SC) Dark gray Clayey Sand with little shell hash; soft, cohesive, non-plastic	VC 1	100	1.4			111
		(SC) Dark gray Clayey Sand with trace shell fragments; one large shell at 1.25 ft. Very soft, slightly cohesive, non plastic; rare pebbles; pp- 0.1 kg/cm2 at 1.5 ft				0.1	0	
		(SM) Soft, dark gray clayey Silt with trace pebbles; noncohesive, nonplastic	VC 2	100	0.9			127
		(SM) Medium dense, dark gray Silt with some pebbles; noncohesive, nonplastic						
5		(SP) Medium dense, reddish brown medium Sand	VC 3	100	3.0			
		(SP) Medium dense, light brown medium Sand with trace pebbles	VC 4	100	4.2			118
		(SP) Medium dense, reddish brown medium to coarse Sand with little pebbles						
10			VC 5	100	9.5			125
			VC 6	100	10.1			115
15								

Bottom of borehole at 15.9 feet.

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
□ FINES CONTENT (%) □ 20 40 60 80

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BORING NUMBER VC 23

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/27/16 **COMPLETED** 11/27/16
WATER DEPTH -18.05 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.91
DRILLING METHOD Vibracore
CORE RECOVERY: 11.50
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:945096.62 Y:124485.31
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CL) Very soft, dark gray Clayey Sand with trace shell hash; cohesive, nonplastic	VC 1	100	1.6			108
						0	0.03	
			VC 2	100	2.6	0	0.04	99
						0	0.02	
5						0	0.04	
			VC 3	100	1.6	0	0	97
						0	0	
						0	0.06	
			VC 4	100	1.4	0	0.07	102
10						0.1	0.05	
			VC 5	100	1.7	0.1	0.08	104
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 16.9 feet.

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BORING NUMBER VC 24

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/27/16 **COMPLETED** 11/27/16
WATER DEPTH -18.18 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.59
DRILLING METHOD Vibracore
CORE RECOVERY: 11.46
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:947094.03 Y:124418.2
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / □ FINES CONTENT (%) □ 20 40 60 80
0		(SC) Very soft, dark gray clayey Sand with trace shell hash; cohesive, nonplastic	VC 1	100	2.1	0.1	0	112	▲ ● □
		(SC) Soft, dark gray clayey Silt with trace shell fragments; noncohesive, nonplastic	VC 2	100	3.1			105	▲ ● □
5			VC 3	100	1.9			125	▲ □ ●
		(SM) Medium dense, dark gray silty Sand with some pebbles							
		(SPG) Medium dense, brown coarse Sand with little pebbles							
		(SP) Dense, brown medium Sand	VC 4	100	3.3			114	▲ ●
10		(SP) Medium dense, reddish brown coarse Sand with some pebbles	VC 5	100	11.7			133	□ ▲
15									

Bottom of borehole at 15.6 feet.

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BORING NUMBER VC 25

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/27/16 **COMPLETED** 11/27/16
WATER DEPTH -17.84 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 14.35
DRILLING METHOD Vibracore
CORE RECOVERY: 11.38
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:949091.06 Y:124351.15
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Dark gray to light gray poorly gradedSand withsilt, little shell hash, trace pebbles; noncohesive, nonplastic	VC 1	100	2.1			109
		(SP) Light gray-brown fine to medium Sand, trace coarse sand and pebbles						
		(SP) Light brown fine to medium Sand, trace coarse sand and few pebbles						
		(SP) Medium dense, dark brown medium Sand with trace pebbles	VC 2	100	2.3			121
5		(SP-SM) Medium dense, brownish red medium Sand with trace pebbles	VC 3	100	19.2			123
		(SP) Medium dense, grayish brown coarse Sand with trace pebbles	VC 4	100	29.7			
		(SP) Dense, reddish brown coarse Sand with trace pebbles	VC 5	100	14.2			133
10		(SP) Dense, reddish gray coarse Sand with trace pebbles	VC 6	100	28.4			137

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 14.4 feet.

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BORING NUMBER VC 26

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/4/16 **COMPLETED** 12/4/16
WATER DEPTH -14.72 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.5
DRILLING METHOD Vibracore
CORE RECOVERY: 11.67
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:951089.14 Y:124409.84
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Light brown poorly graded Sand; trace shell hash	VC 1	100	0.2			106
		(SP-SM) Dark gray fine to medium Sand with silt and trace shell hash and pebbles; very soft	VC 2	100	1.6			
		(SW) Medium dense, dark reddish gray medium Sand with trace gravel and coarse sand	VC 3	100	0.9			126
5		(CL) Stiff, light gray sandy Clay with trace gravel, thinly bedded with red sandy clay; lenses of highly plastic and cohesive light gray clay at 5.04 ft and 5.17 ft				2.75	0.48	
		(SW-SM) Stiff, dark red silty fine Sand with some gravel Unsplit	VC 4	100	2.3			139
		(SW) Medium dense, brown medium Sand with little gravel						
		(SW) Brown, fine to medium Sand						
		Unsplit	VC 5	100	15.6			113
10		(SP) Dense, gray fine Sand						
		(SW) Dense, gray fine to medium Sand with trace coarse sand	VC 6	100	16.2			125
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 27

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/5/16 **COMPLETED** 12/5/16
WATER DEPTH -17.8 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.31
DRILLING METHOD Vibracore
CORE RECOVERY: 11.50
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:953005.77 Y:124939.45
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Very loose, dark gray fine to medium Sand with silt, trace shell hash and pebbles; one pebble 1.5 diameter at 0.75 feet	VC 1	100	1.4			130
		(SP) Very loose, dark red-brown fine Sand, trace coarse Sand						
		(SP) Very loose, dark brown medium to coarse Sand						
		(SP-SM) Loose, dry, gray coarse Sand	VC 2	100	2.9			118
		(SP-SM) Medium dense, red fine silty Sand						
		(SC) Medium dense, red clayey fine Sand						
5		(SP) Loose, fine to medium brownish red Sand	VC 3	100	3.9			116
			VC 4	100	8.7			
			VC 5	100	6.5			111
10			VC 6	100	4.6			118
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 15.3 feet.

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BORING NUMBER VC 28

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/5/16 **COMPLETED** 12/5/16
WATER DEPTH -18.83 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.27
DRILLING METHOD Vibracore
CORE RECOVERY: 10.92
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:954766.91 Y:125875.8
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SM) Dark gray silty fine Sand; trace shell hash	VC 1	100	1.9			124
		(SP) Dark gray medium Sand, trace pebbles and shell hash						
		(SP) Light gray medium Sand						
		(SM) Silty, gray medium Sand	VC 2	100	1.9			128
5		(SP) Loose, coarse gray Sand with gravel; well graded						
			VC 3	100	2.3			126
		(SP) Very loose, gray and red Gravel with coarse sand; well graded	VC 4	100	6.3			
		(SP) Loose, fine to medium dark gray Sand	VC 5	100	3.5			130
10		(SP) Loose, Gravel and coarse Sand; well graded	VC 6	100	2.76			130
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.3 feet.

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BORING NUMBER VC 29

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/5/16 **COMPLETED** 12/5/16
WATER DEPTH -17.83 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.58
DRILLING METHOD Vibracore
CORE RECOVERY: 11.29
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:956498.35 Y:126873.51
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SM) Very loose, dark gray silty Sand with red silty clay lamination at surface; cohesive, nonplastic	VC 1	100	1.6			114
		(SW) Very loose, dark gray fine Sand with trace pebbles and shells; noncohesive, nonplastic						
		(SW) Loose, dark red medium Sand with trace pebbles; thin clay lamination at 2.17 ft						
		(SP) Loose, reddish brown fine to coarse Sand; well graded	VC 2	100	2.6			114
5			VC 3	100	3.0			116
			VC 4	100	6.1			
		(SP) Medium dense coarse Sand and gravel; well graded	VC 5	100	2.6			125
10			VC 6	100	4.4			134
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 15.6 feet.

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BORING NUMBER VC 30

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/5/16 **COMPLETED** 12/5/16
WATER DEPTH -17.92 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.86
DRILLING METHOD Vibracore
CORE RECOVERY: 11.04
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:958231.75 Y:127870.52
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SW-SM) Loose, dark gray silty fine Sand with trace shell fragments and coarse sand; noncohesive, nonplastic	VC 1	100	2.1			131
		(SW) Loose, red and black silty medium Sand; poorly graded						
		(SW) Loose, red coarse gravelly Sand; Well graded	VC 2	100	0.9			125
5		(SW) Medium dense, reddish gray medium Sand with trace gravel; well graded						
		(SP) Loose, yellow brown medium Sand; poorly graded	VC 3	100	4.1			116
			VC 4	100	6.7			123
10			VC 5	100	6.2			114
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 15.9 feet.

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BORING NUMBER VC 31 ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/6/16 **COMPLETED** 12/6/16
WATER DEPTH -16.85 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.7
DRILLING METHOD Vibracore
CORE RECOVERY: 11.63
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:960083.55 Y:128941.61
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Loose, brown to gray poorly Sand with silt; trace shell fragments	VC 1	100	1.6			108
			VC 2	100	2.1			
		(SP) Very loose, yellowish gray medium Sand; poorly graded	VC 3	100	1.9			105
5		(SM) Loose, light gray silty fine Sand with trace shell fragments; noncohesive, nonplastic						
		(CL) Soft, gray silty Clay; noncohesive, nonplastic	VC 4	100	3.8			101
			VC 5	100	2.1			
		(SP) Loose, gray very coarse Sand with some gravel; well graded	VC 6	100	0.5			124
10								
			VC 7	100	3.9			131
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.7 feet.

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BORING NUMBER VC 32

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/6/16 **COMPLETED** 12/6/16
WATER DEPTH -17.79 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.95
DRILLING METHOD Vibracore
CORE RECOVERY: 11.08
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:961695.3 Y:129870.51
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Loose, brown medium Sand with trace shells; poorly graded	VC 1	100	2.1			124
		(SP-SM) Loose, dark gray medium Sand with silt; poorly graded						
		(SW) Loose, brown fine to coarse Sand with some gravel; well graded						
		(SP) Medium dense, reddish brown fine to coarse Sand with trace gravel; well graded	VC 2	100	1.9			113
5			VC 3	100	4.4			
			VC 4	100	4.2			117
10			VC 5	100	2.3			116
			VC 6	100	2.5			122
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 17.0 feet.

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BORING NUMBER VC 33 ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/6/16 **COMPLETED** 12/6/16
WATER DEPTH -17.2 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 17.3
DRILLING METHOD Vibracore
CORE RECOVERY: 11.46
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:963516.61 Y:130916.02
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light brown medium Sand; poorly graded	VC 1	100	2.3			107
		(SP-SM) Medium dense, dark brown silty Sand with some shell fragments; poorly graded						
		(SW) Loose, dark brown medium Sand with trace gravel	VC 2	100	1.5			117
5		(SW) Loose, brown medium Sand with trace coarse sand and gravel						
		(SP) Medium dense, dark gray fine Sand thinly bedded with dark brown and yellow brown fine sand	VC 3	100	2.6			113
			VC 4	100	3.3			
10			VC 5	100	1.6			116
		(SP) Medium dense, dark reddish brown medium Sand; layer of coarse sand and gravel from 10.63 - 10.92ft	VC 6	100	2.8			125
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 17.3 feet.

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BORING NUMBER VC 34

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/6/16 **COMPLETED** 12/6/16
WATER DEPTH -17.6 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.7
DRILLING METHOD Vibracore
CORE RECOVERY: 10.83
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:965273.19 Y:131643.93
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, gray medium Sand with little shell fragments; poorly graded	VC 1	100	1.6			98
			VC 2	100	1.5			
		(SP) Very loose, light brown medium Sand						
		(SP) Medium dense, gray medium Sand thickly bedded with dark gray medium sand with some shell hash laminations	VC 3	100	1.9			112
5		(SP) Medium dense, brown medium Sand						
		(SP-SM) Medium dense, dark gray medium Sand with trace shell fragments	VC 4	100	2.3			129
		(SP-SM) Medium dense, brown medium Sand						
		(SW) Medium dense, dark gray coarse sand with trace gravel and shell fragments						
		(SW) Loose, dark brown medium to coarse Sand with some gravel						
		(SW) Medium dense, dark brown fine Sand with trace gravel	VC 5	100	3.7			128
10		(SP) Loose, brown coarse Sand	VC 6	100	3.2			123
15								

▲ SECONDS PER FOOT ▲ (20 40 60 80)
PL MC LL (20 40 60 80)
☐ FINES CONTENT (%) ☐ (20 40 60 80)

Bottom of borehole at 15.7 feet.

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BORING NUMBER VC 35

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/6/16 **COMPLETED** 12/6/16
WATER DEPTH -18.98 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.24
DRILLING METHOD Vibracore
CORE RECOVERY: 11.21
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:967242.9 Y:131994.9
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Loose, gray medium Sand with silt; trace gravel; poorly graded	VC 1	100	1.2			125
		(SP-SM) Loose, reddish brown fine to coarse Sand with some gravel						
		(SW-SM) Medium dense, reddish brown very fine Sand with silt; noncohesive, nonplastic						
		(SP) Dense, reddish brown fine Sand	VC 2	100	1.4			128
5		(SW) Very loose, brown medium Sand thinly bedded with coarse sand	VC 3	100	3.0			
		(SP) Loose, brown, medium Sand with trace thin beds of coarse sand						
			VC 4	100	2.6			118
10		(SW) Medium dense, gray medium Sand with trace gravel	VC 5	100	4.4			111
			VC 6	100	8.3			123
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.2 feet.

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BORING NUMBER VC 36

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/6/16 **COMPLETED** 12/6/16
WATER DEPTH -20.33 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.51
DRILLING METHOD Vibracore
CORE RECOVERY: 17.17
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:969241 Y:131903.44
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SC) Soft, black clayey Sand; trace shells; organic material noted	VC 1	100	1.9	0.1	0.025	105
		(SC) Loose, medium yellow Sand with trace shells						
		(ML) Soft, dark brown sandy Silt with little shell fragments; noncohesive, nonplastic						
		(SM) Medium dense, dark gray silty Sand with trace shell fragments						
		(SW-SM) Loose, dark grayish brown fine Sand with trace pebbles and gray silt	VC 2	100	1.4			110
		(SP) Loose, dark grayish brown medium Sand with trace pebbles	VC 3	100	4.2			
5		(SP) Loose, grayish brown fine Sand						
		(SP) Medium dense, reddish gray brown coarse Sand with trace pebbles	VC 4	100	1.6			118
		(SP) Medium dense, brown medium Sand	VC 5	100	4.2			111
10			VC 6	100	9.7			
		(SP) Loose, gray medium Sand						
		(SP) Medium dense, orangish brown medium to coarse Sand; lens of fine sand from 16.08 - 16.21	VC 7	100	5.3			118
15			VC 8	100	7.4			104
		(SP) Very loose, orangish brown coarse Sand with trace pebbles						

Bottom of borehole at 19.5 feet.

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

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BORING NUMBER VC 37

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/8/16 **COMPLETED** 12/8/16
WATER DEPTH -21.78 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.51
DRILLING METHOD Vibracore
CORE RECOVERY: 15.42
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:971194.11 Y:971194.11
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Dark gray Clay; high plasticity	VC 1	100	2.1			92
		(CL-ML) Soft, black silty Clay; noncohesive, nonplastic; cobble at 4.42ft						
		(CL-ML) Soft, black silty Clay with some fine sand and shell fragments; noncohesive, nonplastic						
		(MH) Very soft, dark gray clayey Silt with little shell fragments; noncohesive, nonplastic	VC 2	100	2.8			114
5		(SP-SM) Medium dense, gray fine Sand with trace shell fragments and thin beds of gray clay						
		(SP-SM) Medium dense, dark gray silty Sand with little shell fragments	VC 3	100	2.3			128
		(SP) Medium dense, dark orange medium Sand thinly bedded with coarse sand						
10			VC 4	100	2.3			122
		(SP) Medium dense, orange fine Sand with trace beds of dark brown fine sand, and a lens of coarse sand from 12.25 - 12.5 ft	VC 5	100	8.4			
		(SP) Medium dense, light reddish brown fine to medium Sand	VC 6	100	5.6			126
15		(SP-SM) Medium dense, dark reddish brown fine Sand	VC 7	100	12.1			121

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 38

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/8/16 **COMPLETED** 12/8/16
WATER DEPTH -25.4 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.51
DRILLING METHOD Vibracore
CORE RECOVERY: 16.38
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:972198.55 Y:131238.87
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(OH) Soft, wet, black sandy Organic Clay; noncohesive	VC 1	100	2.6			94
		(SM) Soft, moist, dark brown silty Clay; noncohesive, nonplastic						
		(SM) Soft, wet, dark gray silty Clay; noncohesive, nonplastic	VC 2	100	3.2			105
5		(SP-SM) Soft, wet, dark gray silty Sand with trace shells						
			VC 3	100	2.3			129
		(SP) Dense, moist, dark brown fine Sand						
		(SP) Medium dense, reddish gray medium Sand thinly bedded with coarse sand						
10			VC 4	100	3.7			108
		(SP) Medium dense, dark orangish brown medium to coarse Sand						
		(SP) Medium dense, orangish brown medium to coarse Sand with brown laminations						
			VC 5	100	9.3			115
			VC 6	100	9.5			
		(SP) Medium dense, dark brown medium Sand with dark brown laminations						
15		(SP) Loose, dark orange-brown coarse Sand with clay lens from 15.5 - 15.67 ft, and trace pebbles	VC 7	100	7.3			112

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 39

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/8/16 **COMPLETED** 12/8/16
WATER DEPTH -34.78 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.5
DRILLING METHOD Vibracore
CORE RECOVERY: 18.83
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:972811.56 Y:131099
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SM) Soft, black silty Clay; noncohesive, nonplastic						
		(SM) Loose, gray, wet silty fine Sand with trace shells; poorly graded	VC 1	100	2.3			109
		(SP-SM) Loose, gray fine Sand with silt and coarse shell hash						
		(CL) Very stiff, dark brown Clay thinly bedded with gray clay and trace peat; nonplastic, noncohesive	VC 2	100	4.7	1.5	0.14	104
5		(ML) Very soft, wet, black sandy Silt				0.9	0.15	
		(SM) Stiff, wet, dark gray fine sand with trace coarse Sand	VC 3	100	2.1			121
		(SP) Stiff, moist, reddish gray medium Sand with trace coarse sand	VC 4	100	2.1			
		(SP) Medium dense, moist, gray medium Sand with trace pebbles						
10		(SP) Stiff, moist, dark grayish red medium Sand with trace coarse sand	VC 5	100	1.6			131
		(SC) Stiff, moist, grayish green silty Clay; cohesive, low plasticity				2.1	0.35	
			VC 6	100	4.9			126
		(ML) Medium dense, moist, reddish green sandy Silt						
15		(SM) Medium dense, moist, orange fine Sand	VC 7	100	9.2			118
		(SM) Dense, moist, dark brownish-red fine Sand						
		(SP) Dense, moist, dark brown medium Sand with trace coarse sand						
		(SW) Medium dense, dry, yellow brown medium Sand with some coarse sand and gravel						
		(CH) Dense, dry, light blue Clay; cohesive, highly plastic				1.1	0.35	

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
□ FINES CONTENT (%) □ 20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 40

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/8/16 **COMPLETED** 12/8/16
WATER DEPTH -32.48 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.5
DRILLING METHOD Vibracore
CORE RECOVERY: 15.42
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:973309.06 Y:130978.87
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Very loose, yellow, fine to medium Sand	VC 1	100	2.6			111
		(SP-SM) Loose, black, fine Sand lamination						
		(SP-SM) Loose, yellowish brown medium Sand; 1 inch diameter shell at 2.33 feet						
		(SP-SM) Medium dense, brown fine Sand; trace shell hash	VC 2	100	2.8			120
5		(SP-SM) Medium dense, dark gray fine Sand with coarse sand thin beds at 5.08 and 5.33 feet						
		(SP-SM) Medium dense, dark brownish red fine Sand with some coarse sand from 7.25 - 7.75 ft	VC 3	100	1.6			128
			VC 4	100	4.0			
		(SP) Medium dense, light brown coarse Sand with trace pebbles	VC 5	100	1.4			118
10								
		(SC) Loose, wet, dark gray fine Sand	VC 6	100	22.1			122
		(SP-SM) Medium dense, dark reddish brown medium Sand with trace coarse sand	VC 7	100	7.0			118
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 41 ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/10/16 **COMPLETED** 12/10/16
WATER DEPTH -24.3 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.49
DRILLING METHOD Vibracore
CORE RECOVERY: 15.5
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:974209.67 Y:130733.23
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(OL) Shell hash sample	VC 1	100	0.3			
		(SP-SM) Very loose, yellow medium Sand with some shell fragments	VC 2	100	1.4			108
		(ML) Very soft, black sandy Silt with trace shell fragments						
		(SM) Loose, black very fine silty Sand with trace shell fragments						
		(SP-SM) Medium dense, dark brown fine Sand thinly bedded with dark gray silty clay; trace shell fragments	VC 3	100	1.2			108
		(SP) Dense, gray fine Sand						
5			VC 4	100	2.1			121
			VC 5	100	3.7			122
10			VC 6	100	11.1			115
		(SP-SM) Dense, dark gray fine Sand with trace shells						
15			VC 7	100	48.7			120

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 42

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/10/16 **COMPLETED** 12/10/16
WATER DEPTH -16.5 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.03
DRILLING METHOD Vibracore
CORE RECOVERY: 11.33
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:975045.49 Y:130398.95
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Shell hash in yellow medium Sand	VC 1	100	0.2			
		(SP-SM) Very loose, brown medium Sand with shells		100	1.3			101
		(SP-SM) Loose, light gray medium Sand with clay lens from 5" to 6.5"	VC 2					
		(SP) Very loose, dark gray medium Sand with trace shells						
		(SP) Very loose, black medium Sand with trace shells						
		(CH) Loose, brown fine Sand with some clay						
		(CH) Medium dense, black sandy Silt with trace shells	VC 3	100	1.0	0	0.12	98
		(CH) Very soft, moist, dark greenish gray Clay with trace shells; cohesive, nonplastic						
5		(SP-SM) Loose, dark gray fine Sand with trace shells and some silt						
		(SP-SC) Medium dense, light gray fine Sand with some shells thinly bedded with dark gray clayey sand						
		(SM) Very soft, wet, dark gray clayey Sand with little shells	VC 4	100	1.2			111
		(SP) Medium dense, gray fine Sand with trace shells	VC 5	100	2.5			
10			VC 6	100	7.4			124
			VC 7	100	35.6			123
15								

▲ SECONDS PER FOOT ▲ (20 40 60 80)
PL MC LL (20 40 60 80)
☐ FINES CONTENT (%) ☐ (20 40 60 80)

Bottom of borehole at 16.0 feet.

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BORING NUMBER VC 43

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/10/16 **COMPLETED** 12/10/16
WATER DEPTH -16.8 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.21
DRILLING METHOD Vibracore
CORE RECOVERY: 11.92
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:976737.25 Y:129339.01
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Loose, light grayish brown medium Sand, and large shells in upper 2 inches	VC 1	100	2.8			98
		(SP) Medium dense, dark gray medium Sand						
		(SP) Loose, dry, yellow fine to medium Sand	VC 2	100	6.5			110
5		(SP) Loose, moist, light gray medium Sand						
		(SP) Loose, yellow, medium Sand	VC 3	100	32.1			105
		(SP) Medium dense, light gray medium Sand with trace shell fragments						
10		(SP) Medium dense, dry, yellow fine Sand	VC 4	100	22.9			104
		(SP) Medium dense, moist, dark gray fine to medium Sand with trace shell fragments						
			VC 5	100	11.3			128
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 15.2 feet.

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BORING NUMBER VC 44

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/10/16 **COMPLETED** 12/10/16
WATER DEPTH -16.69 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 14.52
DRILLING METHOD Vibracore
CORE RECOVERY: 12.00
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:978307.36 Y:127774.37
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light grayish brown medium Sand with trace shells	VC 1	100	1.6			104
		(SP) Medium dense, moist, dark gray medium Sand with 1" cobble at 2 ft	VC 2	100	1.9			123
			VC 3	100	2.5			128
		(SP) Medium dense, moist, light to dark gray fine Sand	VC 4	100	8.2			109
5			VC 5	100	24.9			
			VC 6	100	40.3			106
10			VC 7	100	92.1			106
			VC 8	100	69.4			125

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 14.5 feet.

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BORING NUMBER VC 45 ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/11/16 **COMPLETED** 12/11/16
WATER DEPTH -14.23 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 14.27
DRILLING METHOD Vibracore
CORE RECOVERY: 11.83
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:979791.2 Y:126210
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light gray medium Sand with trace shells	VC 1	100	1.6			106
		(SP) Loose, light brown medium Sand with some large shells						
		(SP) Loose, light gray medium Sand with some large shells	VC 2	100	2.7			
		(SP) Medium dense, gray medium Sand with trace shell fragments						
		(SP) Loose, dry, yellow medium Sand	VC 3	100	6.7			109
5		(SP) Medium dense, moist, dark gray medium Sand						
		(SP) Loose, dry, yellow medium Sand	VC 4	100	26.4			109
		(SP) Medium dense, moist, dark gray medium Sand						
		(SP) Loose, dry, yellow medium Sand	VC 5	100	31.1			107
10		(SP) Loose, moist, dark gray medium Sand						
			VC 6	100	15.1			123

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 14.3 feet.

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BORING NUMBER VC 46

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/11/16 **COMPLETED** 12/11/16
WATER DEPTH -17.45 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.74
DRILLING METHOD Vibracore
CORE RECOVERY: 12.17
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:980931.07 Y:125005.02
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SM) Very loose, moist, dark brown medium Sand with trace clay and shells	VC 1	100	1.4			94
		(SP) Medium dense, light gray medium Sand	VC 2	100	0.9			110
			VC 3	100	2.4			134
		(SP) Loose, dry, yellow medium Sand with trace shells	VC 4	100	6.9			105
5		(SP) Loose, moist, dark gray medium Sand with trace shells	VC 5	100	27.0			
		(SP) Loose, dry, yellow medium Sand with trace shells	VC 6	100	18.8			104
		(SP) Loose, moist, dark gray medium Sand with trace shells						
10		(SP) Loose, dry, yellow medium Sand with trace shells	VC 7	100	22.3			103
		(SP) Loose, moist, dark gray medium Sand with trace shells	VC 8	100	20.5			124

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
□ FINES CONTENT (%) □ 20 40 60 80

Bottom of borehole at 13.7 feet.

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BORING NUMBER VC 47

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/11/16 **COMPLETED** 12/11/16
WATER DEPTH -17.74 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.18
DRILLING METHOD Vibracore
CORE RECOVERY: 11.71
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:982379.96 Y:123660.9
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, brown medium Sand	VC 1	100	1.4			112
		(SP) Very loose, black fine to medium Sand with trace shell fragments and large shells						
		(CL-ML) Very loose, wet, black silty Clay with some shells						
		Unsplit	VC 2	100	0.9			92
		(SM) Loose, moist, black fine silty Sand with few shells						
5		(SP) Loose, moist, dark gray medium Sand						
		(SP) Loose, dry, yellow medium Sand	VC 3	100	5.8			104
		(SP) Loose, moist, yellow medium Sand thinly bedded with moist, loose, gray medium sand						
		(SP) Medium dense, moist, light gray medium Sand						
10		(SP) Loose, dry, medium yellow Sand; clay lamination from 9.0 - 9.21 ft	VC 4	100	11.7			110
		(SP) Very loose, dry, coarse brown Sand						
		(SP) Loose, moist, black fine Sand with some shells	VC 5	100	7.9			130
		(SP) Loose, moist, gray coarse Sand						
15								

Bottom of borehole at 15.2 feet.

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

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BORING NUMBER VC 48 ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/19/16 **COMPLETED** 12/19/16
WATER DEPTH -14.12 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 14.92
DRILLING METHOD Vibracore
CORE RECOVERY: 11.58
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:984202.76 Y:122534.78
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light brown medium Sand with trace shell hash	VC 1	100	2.3			101
		(SP-SM) Loose, gray medium sand with trace black sandy Silt	VC 2	100	2.1			124
		(SP) Loose, dry, pale yellow fine Sand						
		(SP) Loose, moist, medium yellow Sand	VC 3	100	2.1			110
5		(SP) Medium dense, moist, black medium Sand with shells	VC 4	100	36.3			
		(SP) Loose, dry, pale yellow fine Sand with shells	VC 5	100	36.2			111
		(SP) Medium dense, moist, light gray medium Sand						
		(SP) Loose, dry, pale yellow medium Sand	VC 6	100	63.3			113
10		(SP) Medium dense, moist, dark gray medium Sand	VC 7	100	65.7			123

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
☐ FINES CONTENT (%) ☐ 20 40 60 80

Bottom of borehole at 14.9 feet.

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BORING NUMBER VC 49

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/13/16 **COMPLETED** 12/13/16
WATER DEPTH -11.7 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.78
DRILLING METHOD Vibracore
CORE RECOVERY: 12.08
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:985638.86 Y:122017.58
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Loose, brown fine Sand	VC 1	100	2.1			98
		(SP) Medium dense, gray fine to medium Sand						
		(SP) Loose, moist, light yellow medium Sand	VC 2	100	17.4			105
5		(SP) Loose, moist, gray medium Sand	VC 3	100	11.7			102
		(SP) Loose, dry, very light yellow medium Sand	VC 4	100	20.5			102
10		(SP) Medium dense, moist, very dark gray medium Sand	VC 5	100	28.7			
			VC 6	100	30.3			128

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 13.8 feet.

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BORING NUMBER VC 50

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/11/16 **COMPLETED** 12/11/16
WATER DEPTH -17.91 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.38
DRILLING METHOD Vibracore
CORE RECOVERY: 12.04
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:987403.33 Y:121670.34
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light brown medium Sand with trace shells	VC 1	100	1.2			117
		(SP) Loose, gray medium Sand with trace shells						
		(SP) Loose, dark gray medium Sand	VC 2	100	1.4			119
		(SP) Loose, dry, light brown fine Sand	VC 3	100	3.7			98
			VC 4	100	8.9			99
5		(SP) Loose, dry, light brown medium Sand						
		(SP) Medium dense, dry, black medium Sand						
		(SP) Medium dense, dry, light brown, medium Sand	VC 5	100	15.2			111
		(SP) Medium dense, dry, black medium Sand with few gravel						
10		(SP) Medium dense, dry, pale yellow fine Sand	VC 6	100	24.1			107
		(SP) Medium dense, dry, dark grayish black medium Sand	VC 7	100	24.8			125

Bottom of borehole at 13.4 feet.

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
☐ FINES CONTENT (%) ☐ 20 40 60 80

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BORING NUMBER VC 51

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/11/16 **COMPLETED** 12/11/16
WATER DEPTH -23.24 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.19
DRILLING METHOD Vibracore
CORE RECOVERY: 12.17
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:989335.67 Y:121657.44
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light brown medium Sand with trace shell fragments	VC 1	100	1.9			98
		(SP) Loose, dark gray medium Sand						
		(SP) Medium dense, dry, yellow fine to medium Sand	VC 2	100	10.7			120
5		(SP) Loose, dry, fine black Sand with trace shell fragments	VC 3	100	52.4			
		(SP) Medium dense, dry, medium light gray Sand with some shell fragments	VC 4	100	30.3			124
10		(SP) Loose, dry, fine yellow Sand with few shells	VC 5	100	31.7			115
		(SP) Medium dense, dry, light gray medium Sand	VC 6	100	36.4			128

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 13.2 feet.

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BORING NUMBER VC 52

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/11/16 **COMPLETED** 12/11/16
WATER DEPTH -19.04 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.82
DRILLING METHOD Vibracore
CORE RECOVERY: 11.92
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:991493.28 Y:122094.84
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light brown medium Sand	VC 1	100	2.3			106
		(SP) Very loose, light gray medium Sand						
		(SP) Loose, gray medium Sand	VC 2	100	1.4			130
			VC 3	100	4.4			130
		(SP) Medium dense, dry, fine to medium yellow Sand	VC 4	100	16.8			111
5								
			VC 5	100	109.4			112
			VC 6	100	130.4			
			VC 7	100	49.7			113
10								
		(SP) Medium dense, dry, fine to medium black Sand with some shell fragments	VC 8	100	17.2			131

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
☐ FINES CONTENT (%) ☐ 20 40 60 80

Bottom of borehole at 13.8 feet.

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Telephone: 201-768-8000

BORING NUMBER VC 53

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/19/16 **COMPLETED** 12/19/16
WATER DEPTH -22.13 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.57
DRILLING METHOD Vibracore
CORE RECOVERY: 12.08
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:992466.79 Y:122464.21
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light brown fine to medium Sand	VC 1	100	2.8			105
		(SP) Medium dense, dark brown medium Sand with trace shell hash	VC 2	100	9.5			126
		(SP) Dense, black fine Sand	VC 3	100	14.3			124
		(SP) Loose, dry, yellow medium Sand	VC 4	100	24.0			111
5		(SP) Medium dense, dry, yellow coarse Sand and some gravel ~1"	VC 5	100	133.2			
		(SP) Very loose, dry, black coarse Sand and some gravel ~1"						
		(SP) Loose, moist, yellow medium to coarse Sand with few small gravel ~0.25"	VC 6	100	81.3			121
		(SP) Medium dense, dry, black medium Sand						
10		(SP) Loose, dry, medium yellow Sand	VC 7	100	172.9			118
		(SP) Black, dry, loose medium Sand and some white gravel ~0.25"						
		(SP) Loose, moist, light gray, medium Sand	VC 8	100	161.0			128

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 13.6 feet.

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BORING NUMBER VC 54

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/13/16 **COMPLETED** 12/13/16
WATER DEPTH -39.18 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.48
DRILLING METHOD Vibracore
CORE RECOVERY: 14.67
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:993370.46 Y:122845.22
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light brown medium Sand	VC 1	100	1.6			101
		(SP) Loose, alternating laminations of light gray and black fine Sand; Clay lens @ 6.5", Peat lens @1.5'	VC 2	100	3.0			121
		(SW-SM) Loose, alternating laminations of dark gray fine Sand and black sandy Silt	VC 3	100	3.0			120
		(SC) Medium dense, light gray fine Sand thinly bedded with black silty Clay	VC 4	100	2.6			106
5		(SP-SM) Medium dense, dark gray fine Sand thinly bedded with black Silt						
		(SP-SM) Medium dense, light brown Sand thinly bedded with black sandy Silt; trace shell fragments	VC 5	100	2.9			106
10			VC 6	100	4.9			121
		(SC) Soft, dark gray sandy Silt thinly bedded with black Clay	VC 7	100	3.9			
		(SP-SC) Loose, light brown fine Sand thinly bedded with dark gray soft Clay; trace shell fragments						
		(SP-SM) Loose, dark gray fine Sand with trace Silt and trace shell fragments						
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 55

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/20/16 **COMPLETED** 12/20/16
WATER DEPTH -15.69 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 29.33
DRILLING METHOD Vibracore
CORE RECOVERY: 29.33
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:997095.45 Y:124366.91
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, moist, brown medium Sand. Well sorted.	VC 1	100	1.1			102
			VC 2	100	1.6			116
		(SP) Loose, moist, dark gray medium Sand. Well sorted.	VC 3	100	4.3			126
		(SP) Medium dense, moist, light brown fine Sand with trace pebbles.	VC 4	100	6.1			115
5		(SP) Medium dense, moist, dark gray medium Sand with little coarse sand						
		(SP) Loose, moist, yellow-brown medium Sand with trace pebbles. Well-graded	VC 5	100	11.9			120
			VC 6	100	19.4			
		(SP) Medium dense, moist, dark gray medium sand with trace coarse Sand and pebbles. Well graded						
10		(SP) Unsplit sample sent to lab	VC 7	100	28.2			124
		(SP) Loose, moist, very light brown fine to medium Sand with trace laminations of greenish gray sand	VC 8	100	11.0			116
		(SP) Loose, moist, brown coarse Sand with little pebbles and trace laminations of gray sand						
15		(SP) Loose, moist, grayish green coarse Sand with trace pebbles and dark gray laminations	VC 9	100	57.2			122
		(SP) Unsplit sample sent to lab						
20			VC					

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
☐ FINES CONTENT (%) ☐ 20 40 60 80

(Continued Next Page)

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BORING NUMBER VC 55

CLIENT Williams **PROJECT NAME** NESE Pipeline

PROJECT NUMBER 1794 **PROJECT LOCATION** Raritan Bay and Lower New York Harbor

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
20		(SP) Unsplit sample sent to lab *(continued)*	10	100	169.5			117
		(SP) Loose, moist, light yellowish brown coarse Sand	VC 11	100	43.3			119
		(SP) Medium dense, moist, light yellow medium Sand with trace pebbles thinly bedded with dark greenish brown medium sand	VC 12	100				
		(SP) Medium dense, moist, dark gray coarse Sand with some pebbles and trace light brown sand laminations						
25		(SP) Medium dense, moist, light brown medium Sand with trace coarse sand and pebbles thinly bedded with dark greenish brown sand	VC 13	100				131
		(SP) Medium dense, moist, dark gray coarse Sand with trace pebbles						
		(SP) Loose, moist, light greenish brown medium Sand with trace coarse sand	VC 14	100				104
		(SP) Medium dense, moist, dark greenish brown medium to coarse Sand with trace pebbles	VC 15	100				119

Bottom of borehole at 29.3 feet.

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

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BORING NUMBER VC 56

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/13/16 **COMPLETED** 12/13/16
WATER DEPTH -21.42 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.6
DRILLING METHOD Vibracore
CORE RECOVERY: 11.79
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:998801.58 Y:125079.29
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light gray medium to coarse Sand	VC 1	100	1.6			114
		(SP) Loose, light brown medium to coarse Sand with some gravel	VC 2	100	1.6			116
		(SP) Medium dense, light gray medium Sand with trace coarse sand	VC 3	100	3.5			118
		(SP) Loose, light greenish brown fine Sand	VC 4	100	13.5			107
5		(SP) Medium dense, light brown medium Sand thinly bedded with gray sand	VC 5	100	41.0			
		(SM) Loose, light brown fine to medium Sand with few coarse sand beds	VC 6	100	20.2			108
		(SP) Loose, light brown coarse Sand						
		(SP) Medium dense, light greenish gray fine Sand						
10		(SP) Loose, light greenish brown medium to coarse Sand with trace pebbles	VC 7	100	17.4			108
			VC 8	100	6.3			108

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 13.6 feet.

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BORING NUMBER VC 57

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/13/16 **COMPLETED** 12/13/16
WATER DEPTH -25.64 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 12.24
DRILLING METHOD Vibracore
CORE RECOVERY: 12.13
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1000031.5 Y:125585.26
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, brown, fine to coarse Sand with little gravel	VC 1	100	1.4			114
		(SP) Loose, light brown fine Sand with trace gravel interbedded with light gray fine sand	VC 2	100	30.4			114
5			VC 3	100	43.4			123
			VC 4	100	59.5			
10			VC 5	100	24.8			111
			VC 6	100	61.9			126

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 12.2 feet.

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BORING NUMBER VC 58

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/13/16 **COMPLETED** 12/13/16
WATER DEPTH -33.87 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.04
DRILLING METHOD Vibracore
CORE RECOVERY: 12.79
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1002381.71 Y:126342.57
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, dark brown medium Sand with trace shell fragments and trace gravel	VC 1	100	2.6			112
			VC 2	100	2.1			122
		(SP) Loose, gray medium Sand	VC 3	100	4.2			128
		(SP) Loose, moist, brown fine Sand interbedded with black fine sand	VC 4	100	23.8			106
5			VC 5	100	108.4			>>
			VC 6	100	62.7			105
10			VC 7	100	103.6			110 >>
			VC 8	100	56.6			117

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 13.0 feet.

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BORING NUMBER VC 59

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/14/16 **COMPLETED** 12/14/16
WATER DEPTH -28.45 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 9.88
DRILLING METHOD Vibracore
CORE RECOVERY: 8.83
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1004344.28 Y:127083.47
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light brown medium Sand with trace shells. Large rock at 8 in and large shell at 1ft 5in	VC 1	100	11.8			111
		(SP) Loose, dark gray fine to medium Sand with trace shell fragments						
		(SP-SM) Loose, moist, gray fine Sand	VC 2	100	29.0			114
5		(SP) Loose, moist, dark gray fine Sand (SP) Loose, moist, dark gray medium Sand	VC 3	100	96.2			
		(SP) Very loose, moist, light brown medium Sand with trace gravel	VC 4	100	120.1			107
		(SW) Loose, moist, dark gray medium Sand with some gravel	VC 5	100	36.1			125

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 9.9 feet.

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BORING NUMBER VC 60

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/16/16 **COMPLETED** 12/16/16
WATER DEPTH -28.32 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 9.81
DRILLING METHOD Vibracore
CORE RECOVERY: 8.88
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1006157.33 Y:127789.93
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very soft, moist, shell hash in brown fine Sand	VC 1	100	1.4			106
		(SP) Very soft, moist, dark brown coarse Sand with some pebbles and gravel; piece of ceramic and glass found						
		Very hard charcoal and slag; piece of anthropogenic glass found at 0.92 ft	VC 2	100	1.2			118
		(SP) Loose, gray fine Sand with trace shell fragments; charcoal found from 0.25 - 0.42 ft	VC 3	100	4.5			130
		(SP) Dense, grey medium Sand with trace shell hash; large shell bed found from 1.83 - 2.17ft	VC 4	100	7.8			112
		(SP) Loose, brown coarse Sand with trace shell fragments						
		(SP) Loose, moist, brown fine Sand						
5		(SP) Loose, moist, black fine Sand with trace shells fragments	VC 5	100	68.1			
		(SP) Loose, moist, brown medium Sand	VC 6	100	51.1			110
		(SP) Loose, moist, black medium Sand	VC 7	100	30.6			121

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
□ FINES CONTENT (%) □ 20 40 60 80

Bottom of borehole at 9.8 feet.

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BORING NUMBER VC 61 ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/16/16 **COMPLETED** 12/16/16
WATER DEPTH -30 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 6.81
DRILLING METHOD Vibracore
CORE RECOVERY: 4.92
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1007456.99 Y:128301.93
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light brown fine Sand with trace shell fragments	VC 1	100	1.9			88
		(SP) Loose, moist, brown medium Sand with shell fragments and trace pebbles; pieces of anthropogenic glass and brick found at 1 ft	VC 2	100	12.0			126
		Granular charcoal with trace shells and pebbles						
		(SP) Loose, dark gray medium Sand with trace shells						
		(SP) Loose, brown fine Sand						
		(SP) Medium dense, black fine Sand thinly bedded with brown sand	VC 3	100	15.1			123
			VC 4	100	24.1			118
5								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 6.8 feet.

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BORING NUMBER VC 62

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/17/16 **COMPLETED** 12/17/16
WATER DEPTH -31.01 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 6.45
DRILLING METHOD Vibracore
CORE RECOVERY: 4.17
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1009834.96 Y:129210.8
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, light brown fine Sand with trace shell fragments	VC 1	100	3.5			98
		(SP-SM) Loose, gray fine Sand with trace shell fragments and trace silt	VC 2	100	4.0			111
			VC 3	100	7.9			119
		(SP) Medium dense, dark brown and gray medium Sand with few shell hash and few silt beds	VC 4	100	28.9			128
5								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 6.5 feet.

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BORING NUMBER VC 63

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/17/16 **COMPLETED** 12/17/16
WATER DEPTH -32.65 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 9.76
DRILLING METHOD Vibracore
CORE RECOVERY: 7.04
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1011696.93 Y:129929.48
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / □ FINES CONTENT (%) □ 20 40 60 80
0		(SP-SM) Medium dense, brown and black silty fine Sand; lens of shells at 0.33 ft	VC 1	100	3.3			115	▲□ ●
		(SP-SM) Medium dense, black silty fine Sand with with trace shell fragments; shell bed at 1.33 ft	VC 2	100	5.7				▲ ●
		(SP-SM) Medium dense, black fine Sand with trace silt							
			VC 3	100	6.3			124	◪ ●
5									
			VC 4	100	12.7			118	□ ▲ ●

Bottom of borehole at 9.8 feet.

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Telephone: 201-768-8000

BORING NUMBER VC 64

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/17/16 **COMPLETED** 12/17/16
WATER DEPTH -33.71 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 7.6
DRILLING METHOD Vibracore
CORE RECOVERY: 6.04
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1013550.49 Y:130644.04
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / □ FINES CONTENT (%) □ 20 40 60 80
0		(SP) Loose, brown fine Sand							
		(SP) Medium dense, dark gray fine Sand	VC 1	100	6.3			112	□▲ ●
			VC 2	100	6.8			122	▲ ●
		(SP-SM) Medium dense, moist, black fine Sand with some silt							
			VC 3	100	6.7			120	□▲ ●
			VC 4	100	9.7				▲ ●
5		(SP) Loose, medium to coarse black Sand thinly bedded with brown Sand; trace shell fragments	VC 5	100	18.0			116	□ ●▲

Bottom of borehole at 7.6 feet.

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BORING NUMBER VC 65

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/17/16 **COMPLETED** 12/17/16
WATER DEPTH -38.12 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 6.4
DRILLING METHOD Vibracore
CORE RECOVERY: 4.21
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1015604.57 Y:131158.15
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / □ FINES CONTENT (%) □ 20 40 60 80
0		(SP) Shell hash with large shells in a medium -fine yellow Sandy matrix	VC 1	100	3.5			102	
		(SP) Loose, fine yellow Sand							
		(SP) Medium dense, moist, dark gray fine Sand with large shell fragment at 2.5 ft and coarse sand lamination between 2.67 - 2.83 ft							
		(SP) Loose, coarse yellow Sand	VC 2	100	24.3			128	
5									

Bottom of borehole at 6.4 feet.

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BORING NUMBER VC 66

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/17/16 **COMPLETED** 12/17/16
WATER DEPTH -35.34 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 7.93
DRILLING METHOD Vibracore
CORE RECOVERY: 5.67
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1017720.25 Y:131268.02
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Light brown fine Sand with some small shells						
		(SP-SM) Medium dense, moist, dark gray fine Sand	VC 1	100	3.9			112
			VC 2	100	4.6			130
		(SP) Loose, dry, yellow coarse Sand	VC 3	100	8.0			127
			VC 4	100	26.4			110
5			VC 5	100	31.1			125

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 7.9 feet.

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BORING NUMBER VC 67

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/17/16 **COMPLETED** 12/17/16
WATER DEPTH -36.5 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 6.45
DRILLING METHOD Vibracore
CORE RECOVERY: 4.92
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1019741.35 Y:130898.4
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / □ FINES CONTENT (%) □ 20 40 60 80
0		(SP-SM) Loose, moist, yellow fine Sand with some shells	VC 1	100	3.3			112	
		(SP) Loose, moist, light gray fine Sand with trace shells	VC 2	100	6.0			128	
			VC 3	100	9.1			120	
			VC 4	100	19.4			120	
5			VC 5	100	30.8			127	

Bottom of borehole at 6.5 feet.

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BORING NUMBER VC 68

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/19/16 **COMPLETED** 12/19/16
WATER DEPTH -40 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 8.51
DRILLING METHOD Vibracore
CORE RECOVERY: 6.25
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1021671.36 Y:130404.59
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Loose, light yellow fine dry Sand with some large shells	VC 1	100	3.5			116
		(SP-SM) Medium dense, moist, light gray fine Sand with silt and some shells	VC 2	100	6.5			128
			VC 3	100	45.5			122
		(SP) Loose, light brown fine to medium Sand with trace shells	VC 4	100	42.5			118
		(SP) Loose, dry, black fine to medium Sand with trace shells						
5		(SP) Light brown medium Sand with few shell fragments	VC 5	100	43.6			127
		(SP-SM) Loose, dark brown silty fine Sand thinly bedded with brown medium sand and some shell hash beds	VC 6	100	134.8			124

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

>>▲

Bottom of borehole at 8.5 feet.

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BORING NUMBER VC 69 ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/19/16 **COMPLETED** 12/19/16
WATER DEPTH -41.51 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 11.25
DRILLING METHOD Vibracore
CORE RECOVERY: 9.92
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:1022403.95 Y:130263.15
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Loose, yellow fine to medium Sand with some large shells	VC 1	100	3.0			115
		(SP) Loose, moist, gray fine Sand with few shell fragments and one large shell at 2.83 ft	VC 2	100	5.1			126
			VC 3	100	8.7			121
		(SP) Loose, moist, light brown fine Sand with few shells	VC 4	100	18.4			116
		(SP) Medium dense, dry, yellow medium Sand interbedded with fine to medium black sand						
5		(SP) Loose, dry, medium gray Sand with few shell fragments and a large shell at 5.75 ft						
		(SP-SM) Medium dense, moist, fine gray Sand with shell fragments, bedded with loose, dry, yellow medium sand	VC 5	100	94.6			123
		(SP-SM) Loose, moist, light gray fine silty sand with few thin shell fragment beds	VC 6	100	108.3			
			VC 7	100	96.8			126
10								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 11.3 feet.

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BORING NUMBER VC 70

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/28/16 **COMPLETED** 11/28/16
WATER DEPTH -13.15 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.5
DRILLING METHOD Vibracore
CORE RECOVERY: 11.04
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:923401.33 Y:115464.1
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Very soft, black, wet Clay with some silt and a few shells; noncohesive, nonplastic	VC 1	100	2.6			82
						0	0	
		(CH) Very soft, greenish gray Clay with silt and trace shells; noncohesive, nonplastic	VC 2	100	1.9	0	0	92
5						0	0	
						0	0.02	
			VC 3	100	1.9	0	0.05	89
						0	0	
						0	0.04	
10			VC 4	100	1.6	0	0	88
			VC 5	100	2.0	0	0.04	89
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 71

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/28/16 **COMPLETED** 11/28/16
WATER DEPTH -13.16 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.5
DRILLING METHOD Vibracore
CORE RECOVERY: 11.63
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:925373.73 Y:115806.05
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	1.5			87
		(CL-ML) Soft, wet, greenish black Clay with silt and trace shells; noncohesive, nonplastic						
		(CH) Unsplit	VC 2	100	2.0			90
5		(CL) Very soft, bluish gray Clay; noncohesive, nonplastic				0	0.03	
		(CH) Unsplit	VC 3	100	2.0			88
		(CL) Soft, bluish gray Clay; noncohesive, nonplastic				0	0.06	
		(CH) Unsplit	VC 4	100	2.2			89
10			VC 5	100	2.1			88
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 72

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/1/16 **COMPLETED** 12/1/16
WATER DEPTH -13.09 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 18.11
DRILLING METHOD Vibracore
CORE RECOVERY: 11.58
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:927345.93 Y:116145.85
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / □ FINES CONTENT (%) □ 20 40 60 80
0		(CH) Very soft, wet, greenish gray Clay with silt, trace shells and peat; cohesive, nonplastic	VC 1	100	1.4			87	▲ □ 0
						0	0		
			VC 2	100	1.4	0	0	88	▲ 0
5						0	0		
						0	0.05		
			VC 3	100	1.3	0	0	87	▲ 1
						0	0		
			VC 4	100	1.7	0	0.07	86	▲ 0
10						0	0.03		
			VC 5	100	1.3	0	0.05	90	▲ 0
15									

Bottom of borehole at 18.1 feet.

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BORING NUMBER VC 73 ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/24/16 **COMPLETED** 11/24/16
WATER DEPTH -9.94 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.23
DRILLING METHOD Vibracore
CORE RECOVERY: 13.25
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:929447.14 Y:116490.81
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Loose, moist black Silt with trace shells and yellow fine sand with trace shells; well graded	VC 1	100	1.9			100
		(SP) Loose, yellow fine to medium Sand with some shell fragments						
		(SP) Loose, wet, black fine Sand				0.25	0	
		(SM) Loose, wet, gray fine Sand with silt and some shell fragments						
		(SM) Medium dense, dark gray Silty sand thinly bedded with red fine sand and trace pebbles						
		(GP) Loose Gravel with light gray and orange coarse sand matrix	VC 2	100	1.4			131
5		(SP) Dense, orange and dark red fine to medium Sand						
		(SP) Loose, light brown medium Sand with little pebbles						
		(SP) Dense, light brown medium to coarse Sand with trace pebbles	VC 3	100	4.0			118
		(SW) Medium dense, dark brown fine Sand with coarse sand and pebbles; well graded						
		(SP) Loose, light orange-brown medium to coarse Sand with little pebbles	VC 4	100	3.5			125
10		(SP) Medium dense, dark brown medium Sand with trace pebbles and coarse sand						
		(SP) Loose, dark brown medium Sand with some pebbles and coarse sand	VC 5	100	11.1			
		(SP) Loose, dark red medium Sand with little coarse sand and pebbles up to 1 inch	VC 6	100	13.5			119
		(SP) Very loose, dark reddish brown fine Sand with trace coarse sand and pebbles						
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 15.2 feet.

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BORING NUMBER VC 74 ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/23/16 **COMPLETED** 11/23/16
WATER DEPTH -12.64 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.83
DRILLING METHOD Vibracore
CORE RECOVERY: 11.13
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:931284.39 Y:116828.72
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Dry, orange, fine to medium Sand with large intact shell at 0.54 ft	VC 1	100	1.2			109
		(SP-SM) Moist, dark gray fine Sand with silt and some small gravel						
		(SP) Loose, orange coarse Sand and gravel						
		(SP) Dense, brown medium Sand with trace pebbles; large rock from 2.38 - 2.71 ft	VC 2	100	3.2			114
5		(SP) Medium dense, dark brown medium to coarse Sand with trace pebbles						
		(SP) Medium dense, reddish brown medium Sand with trace pebbles	VC 3	100	14.4			118
		(SP) Medium dense, dark brown medium to coarse Sand with trace pebbles	VC 4	100	22.1			
		(SP) Medium dense, reddish brown medium Sand with trace pebbles	VC 5	100	7.9			125
10		(SP) Loose, brown coarse Sand with trace pebbles	VC 6	100	9.6			122
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.8 feet.

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BORING NUMBER VC 75

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/24/16 **COMPLETED** 11/24/16
WATER DEPTH -18.6 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.01
DRILLING METHOD Vibracore
CORE RECOVERY: 11.00
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:933255.63 Y:117167.89
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Very soft, wet, black Clay with silt and some shells; noncohesive, nonplastic	VC 1	100	1.6			89
		(CL) Soft, wet, brown silty Clay with some shells and trace sand; noncohesive, nonplastic	VC 2	100	2.0			
		(SC) Dense shell hash in a silty Clay matrix, gray soft, noncohesive and nonplastic	VC 3	100	2.2			114
5								
		(SP) Loose, yellowish brown silty Sand	VC 4	100	1.6			118
		(SP) Medium dense, yellowish brown silty coarse Sand with trace shells						
10			VC 5	100	3.7			115
			VC 6	100	6.5			123
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 19.0 feet.

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Telephone: 201-768-8000

BORING NUMBER VC 76

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/24/16 **COMPLETED** 11/24/16
WATER DEPTH -17.23 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 18.36
DRILLING METHOD Vibracore
CORE RECOVERY: 13.63
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:935223.96 Y:117508.55
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	2.3			86
		(CL) Soft, moist, black Clay; noncohesive, nonplastic						
		(CH) Unsplit	VC 2	100	3.0			94
5		(CL) Very soft, gray brown Clay; noncohesive, nonplastic				0	0	
		(CH) Unsplit	VC 3	100	2.1			91
		(CL) Very soft, gray brown Clay; noncohesive, nonplastic				0	0.04	
10		(CH) Unsplit	VC 4	100	1.4			90
		(CH) Unsplit	VC 5	100	2.8			87
		(CL) Soft, gray Clay with trace shells; noncohesive, nonplastic				0	0.07	
						0	0.05	
15								

SECONDS PER FOOT: 20 40 60 80
PL MC LL: 20 40 60 80
FINES CONTENT (%): 20 40 60 80

Bottom of borehole at 18.4 feet.

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BORING NUMBER VC 77

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/24/16 **COMPLETED** 11/24/16
WATER DEPTH -21.62 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.49
DRILLING METHOD Vibracore
CORE RECOVERY: 16.83
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:937139.18 Y:118062.09
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	2.8			82
		(CL) Soft, wet, black Clay with coarse and and shells; noncohesive, nonplastic						
		(CL) Soft, wet, greenish black Clay with trace shells; noncohesive, nonplastic						
		(CH) Unsplit	VC 2	100	1.8			93
5		(CL) Soft, wet, greenish black Clay with trace shells; cohesive, nonplastic				0	0.05	
						0	0.04	
		(CH) Unsplit	VC 3	100	1.9			95
		(CL) Soft, wet, greenish black Clay with trace shells; cohesive, nonplastic				0	0.05	
						0	0.07	
10		(CH) Unsplit	VC 4	100	2.3			90
		(CL) Soft, wet, greenish black Clay with trace shells; cohesive, nonplastic				0	0.1	
						0	0.13	
		(CH) Unsplit	VC 5	100	2.3			93
		(CL) Soft, wet, greenish black Clay with trace shells; cohesive, nonplastic				0	0.1	
15		(CH) Unsplit	VC 6	100	1.8			91
		(CL) Soft, wet, greenish black Clay with trace shells; cohesive, nonplastic				0	0.12	

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 78

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/19/16 **COMPLETED** 11/19/16
WATER DEPTH -11.79 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.47
DRILLING METHOD Vibracore
CORE RECOVERY: 17.83
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:938887.92 Y:119027.08
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	2.3			84
		(CL) Soft, wet, black Clay with some shells; noncohesive, nonplastic (CL) Soft, gray, Clay; noncohesive, nonplastic						
		(CH) Unsplit	VC 2	100	2.7			90
5		(CL) Very soft, wet, gray Clay; cohesive, nonplastic				0	0.05	
						0	0.06	
		(CH) Unsplit	VC 3	100	1.9			91
		(CL) Very soft, wet, gray Clay; cohesive, nonplastic				0	0.04	
		(CH) Unsplit	VC 4	100	2.1			91
10		(CL) Soft, wet, gray Clay; cohesive, nonplastic				0	0.15	
		(CH) Unsplit	VC 5	100	2.1			93
		(CL) Soft, wet, gray Clay; cohesive, nonplastic				0	0.12	
15		(CH) Unsplit	VC 6	100	1.7			90
		(CL) Soft, wet, gray Clay; cohesive, nonplastic						

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 79 Alt

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/19/16 **COMPLETED** 11/19/16
WATER DEPTH -25.85 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.48
DRILLING METHOD Vibracore
CORE RECOVERY: 18.54
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:940475.34 Y:120460.69
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	3.7			88
		(CL) Soft, dark gray Clay; cohesive, nonplastic						
		(CH) Unsplit	VC 2	100	3.3			92
		(CL) Very soft, wet, gray Clay with trace shells				0	0.05	
5						0	0.1	
		(CH) Unsplit	VC 3	100	2.6			93
		(CL) Very soft, wet, gray Clay with trace shells				0	0.04	
		(CH) Unsplit	VC 4	100	2.9			87
10		(CL) Very soft, wet, gray Clay with trace shells				0	0.1	
		(CH) Unsplit	VC 5	100	1.9			90
		(CL) Very soft, wet, gray Clay with trace shells				0	0.02	
						0	0.07	
15						0	0.15	
						0	0.14	
						0	0.05	
						0	0.12	

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 80

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/19/16 **COMPLETED** 11/19/16
WATER DEPTH -36.89 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.47
DRILLING METHOD Vibracore
CORE RECOVERY: 16.75
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:941296.37 Y:121365.07
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	2.8			78
		(CL) Very soft, wet, black Clay; noncohesive, nonplastic						
		(CH) Unsplit	VC 2	100	2.4			82
		(CL) Very soft, wet, black Clay thinly bedded with green-gray clay; noncohesive, nonplastic				0	0.02	
5						0	0.05	
		(CH) Unsplit	VC 3	100	1.6			88
		(CL) Very soft, moist, black Clay; cohesive, nonplastic				0	0.07	
		(CL) Soft, moist, greenish- gray Clay; cohesive, nonplastic				0	0.10	
						0	0.10	
		(CH) Unsplit	VC 4	100	2.6			89
10		(CL) Soft, moist, greenish- gray Clay; cohesive, nonplastic				0	0.13	
						0	0.14	
		(CH) Unsplit	VC 5	100	2.3			89
		(CL) Soft, moist, greenish- gray Clay; cohesive, nonplastic				0	0.16	
		(CH) Unsplit	VC 6	100	2.5			91
15		(CL) Soft, moist, greenish gray Clay thinly bedded with black clay; cohesive, nonplastic				0	0.15	
						0	0.09	

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 81

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/23/16 **COMPLETED** 11/23/16
WATER DEPTH -32.6 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.48
DRILLING METHOD Vibracore
CORE RECOVERY: 16.08
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:941726.69 Y:121847.07
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	2.3			78
		(CL) Very soft, wet, dark gray, Clay with trace shells; noncohesive, nonplastic	VC 2	100	3.2			
		(CL) Very soft, wet, black Clay; noncohesive, nonplastic				0	0.01	
		(CH) Unsplit	VC 3	100	2.3			81
5		(CL) Very soft, wet, black Clay with trace thin beds of greenish gray clay; noncohesive, nonplastic				0	0.01	
						0	0.01	
		(CH) Unsplit	VC 4	100	1.9			83
		(CL) Very soft, moist, black Clay thinlybedded with greenish gray clay; noncohesive, nonplastic				0	0.03	
						0	0.07	
10		(CH) Unsplit	VC 5	100	1.6			88
		(CL) Very soft, moist, black Clay thinly bedded with greenish gray clay; noncohesive, nonplastic				0	0.07	
						0	0.05	
		(CH) Unsplit	VC 6	100	1.6			89
		(CL) Very soft, moist, black Clay thinly bedded with greenish gray clay; cohesive, nonplastic				0	0.15	
						0	0.13	
15		(CH) Unsplit	VC 7	100	1.6			95
		(CL) Very soft, moist, black Clay thinly bedded with greenish gray clay; cohesive, nonplastic				0	0.16	

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 82

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/23/16 **COMPLETED** 11/23/16
WATER DEPTH -20.26 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 19.48
DRILLING METHOD Vibracore
CORE RECOVERY: 15.46
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:943049.96 Y:123310.85
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(CH) Unsplit	VC 1	100	1.8			95
		(CL) Very soft, wet, dark gray Clay with trace shells; noncohesive, nonplastic						
		(CL) Very soft, moist, light gray silty Clay; noncohesive, nonplastic				0	0.05	
		(CH) Unsplit	VC 2	100	1.9			95
5		(CL) Very soft, moist, light gray Clay; nonplastic, noncohesive				0	0.04	
						0	0.03	
		(CH) Unsplit	VC 3	100	1.6			97
		(CL) Very soft, moist, light gray Clay; nonplastic, noncohesive				0	0.05	
						0	0.05	
10		(CH) Unsplit	VC 4	100	1.6			100
		(CL) Soft, light gray Clay; cohesive, low plasticity				0	0.05	
		(CH) Unsplit	VC 5	100	1.6			97
		(CL) Soft, light gray Clay; cohesive, low plasticity				0	0.01	
						0	0.10	
15						0	0.06	

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
☐ FINES CONTENT (%) ☐ 20 40 60 80

Bottom of borehole at 19.5 feet.

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BORING NUMBER VC 83-ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/28/16 **COMPLETED** 11/28/16
WATER DEPTH -14.65 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.58
DRILLING METHOD Vibracore
CORE RECOVERY: 10.83
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:946366.51 Y:125549.63
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Very loose, moist, light brown medium Sand with trace shells	VC 1	100	2.3			106
		(SP) Loose, moist, dark brown medium Sand with trace silt and shells						
		(SP) Medium dense, wet, dark gray medium Sand with trace shell fragments						
		(SP-SM) Loose, dry, fine Sand with trace shell fragments						
		(SP-SM) Soft, moist, shell hash in a dark gray silty fine Sand matrix	VC 2	100	1.8			121
		(SP-SM) Loose, moist, dark gray medium Sand with some coarse sand						
5		(SP-SM) Medium dense, dry, brown orange medium Sand with some silt laminations	VC 3	100	2.4			
			VC 4	100	1.6			124
		(SP) Loose, dry, orange-brown coarse Sand with few gravel; well graded						
10			VC 5	100	1.6			112
		(SP) Loose, moist, brown-orange medium to coarse Sand	VC 6	100	6.1			123
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 15.6 feet.

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BORING NUMBER VC 84-ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 11/28/16 **COMPLETED** 11/28/16
WATER DEPTH -15.89 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.25
DRILLING METHOD Vibracore
CORE RECOVERY: 11.08
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:948452.09 Y:125884.58
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Loose, moist, brown medium Sand with trace shell fragments	VC 1	100	2.3			115
		(SP) Loose, moist, reddish brown medium to coarse Sand with some gravel						
		(SP) Loose, light gray, coarse Sand with gravel	VC 2	100	2.7			126
5		(GW-GM) Medium dense, red, silty Gravel with coarse sand and cobbles; well graded	VC 3	100	29.1			130
		(SP) Loose, yellow-brown medium Sand	VC 4	100	13.9			121
10		(SP) Loose, yellow-brown medium Sand with gravel; poorly graded	VC 5	100	7.3			124
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 15.3 feet.

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BORING NUMBER VC 85

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/4/16 **COMPLETED** 12/4/16
WATER DEPTH -15.53 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.53
DRILLING METHOD Vibracore
CORE RECOVERY: 10.46
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:950325.01 Y:125844.85
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP) Medium dense, brown medium Sand with large shell from 2 - 5" and trace shell fragments	VC 1	100	2.3	0.75	0.05	109
		(SP) Medium dense, wet, gray and red clayey Sand with trace shell fragments and large shell at 11"						
		(SP) Medium dense, red medium to coarse Sand	VC 2	100	2.2	1	0.04	
		(SP) Dense, red fine Sand						
		(ML) Very dense, moist, gray sandy Silt				2.15	0.14	
		(SP) Dense, red coarse Sand with trace pebbles	VC 3	100	2.6			119
		(SP) Dense, moist, light reddish brown fine Sand						
		(SW) Loose, dry, reddish brown fine Sand with some gravel; well graded						
5		(GP) Loose, poorly graded Gravel in a fine sand matrix	VC 4	100	14.2			121
		(SP) Loose, reddish brown medium Sand						
			VC 5	100	10.1			116
10			VC 6	100	9.9			124
		(SP) Loose, reddish brown very coarse Sand						
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 15.5 feet.

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BORING NUMBER VC 86 ALT

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/4/16 **COMPLETED** 12/4/16
WATER DEPTH -17.24 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.4
DRILLING METHOD Vibracore
CORE RECOVERY: 11.04
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:952172.68 Y:125756.07
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Loose, moist, reddish brown medium to coarse Sand and trace pebbles	VC 1	100	1.4			115
		(SP) Loose, dry, light grayish brown fine to medium Sand	VC 2	100	3.3			114
5		(SP) Loose, dry, light grayish red medium to coarse Sand	VC 3	100	4.9			
		(SP) Loose, reddish brown, coarse Sand with little gravel	VC 4	100	4.6			122
		(SP) Loose, yellow brown medium Sand						
		(SP) Loose, yellow brown coarse Sand with few gravel	VC 5	100	6.0			110
10			VC 6	100	6.9			107
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 15.4 feet.

VIBRACORE COPY 2 - VIBRACORE.GDT - 4/7/17 10:29 - I:\ALPINE USA\PROJECTS\1794 WILLIAMS - VIBRACORING AND BORINGS - NY_NJ - MLK\GEOTECHNICAL\1794 CORE SPLITTING MATERIALS\WILLIAMS VIBRACORE GINT PROJECTNEW GINT PROJECT CD

Alpine Ocean Seismic Survey, Inc
155 Hudson Avenue
Norwood, NJ 07648
Telephone: 201-768-8000

BORING NUMBER VC 87

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritan Bay and Lower New York Harbor
DATE STARTED 12/4/16 **COMPLETED** 12/4/16
WATER DEPTH -21.45 ft MLLW **HOLE SIZE** 3.5 in inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.41
DRILLING METHOD Vibracore
CORE RECOVERY: 12.00
LOGGED BY CM **CHECKED BY** CD
CORE LOCATION: X:954297.38 Y:126036.15
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE TYPE NUMBER	RECOVERY % (RQD)	PENETRATION SECONDS PER FOOT	POCKET PEN. (tsf)	TORVANE (tsf)	TOTAL UNIT WT. (pcf)
0		(SM) Dark gray fine Sand, trace shell hash, trace silt	VC 1	100	3.0			114
		(SM) Dark gray fine Sand with trace silt; large amount of shell hash						
		(SM) Dark gray fine Sand, trace shell hash, trace silt				0.1	0.075	
		(SC) Unsplit	VC 2	100	1.4			114
		(CL-CH) Dark gray Clay; moderate plasticity				0.75	0.25	
		(SP) Dark gray fine to medium Sand, trace pebbles						
5		(SP) Red to light gray fine to medium Sand with trace pebbles	VC 3	100	4.4			122
		(SP) Dark red fine Sand, trace coarse sand and pebbles	VC 4	100	12.3			121
10		(SP) Red-gray medium to coarse Sand with trace pebbles	VC 5	100	10.2			
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 15.4 feet.

VIBRACORE COPY 2 - VIBRACORE.GDT - 4/7/17 10:29 - I:\ALPINE USA\PROJECTS\1794 WILLIAMS - VIBRACORING AND BORINGS - NY_NJ - MLK\GEOTECHNICAL\1794 CORE SPLITTING MATERIALS\WILLIAMS VIBRACORE GINT PROJECTNEW GINT PROJECT CD

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC

NORTHEAST SUPPLY ENHANCEMENT PROJECT

ENVIRONMENTAL REPORT

J-3. ERRATA AND ADDENDUM, OCTOBER 2017

NOVEMBER 2026

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Fall/Winter 2016 Offshore Environmental Sampling Report for the Northeast Supply Enhancement Project

New Jersey, New York

Errata Sheets

October 2017

Prepared for:

Transcontinental Gas Pipe Line Company, LLC

Prepared by:

ECOLOGY AND ENVIRONMENT, INC.
368 Pleasant View Drive
Lancaster, New York 14086

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1 Introduction

Transcontinental Gas Pipe Line Company, LLC. (Transco), an indirect wholly owned subsidiary of Williams Partners L.P. (Williams), is proposing the Northeast Supply Enhancement (NESE) Project (Project) to expand its existing interstate natural gas pipeline system in Pennsylvania and New Jersey and its existing offshore natural gas pipeline system in New Jersey and New York waters. The Project capacity is fully subscribed by two entities of National Grid: Brooklyn Union Gas Company (d/b/a [doing business as] National Grid NY) and KeySpan Gas East Corporation (d/b/a National Grid), collectively referred to herein as "National Grid."

To provide the incremental 400,000 dekatherms per day (Dth/d) of capacity, Transco plans to expand portions of its system from the existing Compressor Station 195 in York County, Pennsylvania, to the Rockaway Transfer Point in New York State waters. As defined in executed precedent agreements with National Grid, the Rockaway Transfer Point is the interconnection point between Transco's existing Lower New York Bay Lateral (LNYBL) and existing offshore Rockaway Delivery Lateral (RDL).

Transco filed its application for a Certificate of Public Convenience and Necessity with the Federal Energy Regulatory Commission (FERC) on March 27, 2017 (Certificate Application), and FERC assigned the Project Docket No. CP17-101. Transco submitted its Offshore Environmental Sampling Report as Appendix J of Transco's Joint Permit Application to the U.S. Army Corp of Engineers (USACE) and New York State Department of Environmental Conservation on June 27, 2017. Transco submitted its first Errata and Addendum to its Offshore Environmental Sampling Report as Appendix J to the Supplemental Information Filing for Transco's Joint Permit Application to the USACE on September 15, 2017. Transco submitted its Offshore Environmental Sampling Report to FERC on June 30, 2017, and its first Errata and Addendum to its Offshore Environmental Sampling report to FERC on September 7, 2017.

The following information consists of corrections to information initially presented in the Offshore Environmental Sampling Report. Please refer to the Offshore Environmental Sampling Report for the unchanged information. All revised information is listed in **red bold text** and removed features marked as ~~**strike through text**~~. Corrections to the Offshore Environmental Sampling Report

are the result of transcription and formatting errors. Additionally, for consistency any use of “VC1-ALT-B” in the Offshore Environmental Sampling Report should be updated to “VC1B-ALT”.

2 Sediment Chemistry

Results

Group A – New Jersey

Pesticides

Each sediment sample was analyzed for 27 pesticides (see Appendix C for a complete list of these pesticides). A total of seven pesticides (Dieldrin, O,P'-DDD, O,P'-DDE, O,P'-DDT, P,P'-DDD, P,P'-DDE, and P,P'-DDT) were detected in at least one sample in Group A. (Table 2-2 lists the sample sites where pesticides were detected.) Eleven pesticides tested for have available ER-L and/or ER-M thresholds and four of these (including the sum of three pesticides [DDD + DDE + DDT) exceeded ~~only~~ the ER-L **and/or ER-M** thresholds.

P,P'-DDD exceeded the ER-L threshold at the 0- to 3-foot and 3- to 6-foot depth intervals at VC1-ALT and VC1**B**-ALT~~-B~~ and at the 6- to 9-foot depth interval at VC1**B**-ALT~~-B~~, with concentrations ranging from 4.48 to 14.7 micrograms per kilogram (µg/kg). P,P'-DDE exceeded the ER-L threshold at the ~~0- to 3-foot and 3- to 6-foot depth intervals at VC1-ALT and VC1-ALT-B and at the~~ 6- to 9-foot depth interval at VC1**B**-ALT~~-B~~ with **a** concentration~~s~~ **of** ~~ranging from~~ 5.5~~ to 56.5~~ µg/kg. **P,P'-DDE exceeded the ER-M threshold at the 0- to 3-foot and 3- to 6-foot depth intervals at VC1-ALT and VC1B-ALT with concentrations ranging from 27.2 to 56.5 µg/kg.** P,P'-DDT exceeded the ER-L threshold at the 0- to 3-foot and 3- to 6 foot depth intervals at VC1-ALT and VC1**B**-ALT~~-B~~ with concentrations ranging from 2.39 to 5.37 µg/kg.

The sum of DDD + DDE + DDT exceeded the ER-L threshold value within the 0- **to** 3-f**oo**t ~~and 3- 6 ft~~ depth interval~~s~~ at VC1-ALT and ~~VC1-ALT-B and~~ the 6- **to** 9-f**oo**t depth interval at VC1**B**-ALT~~-B~~ with concentrations ranging from 14.21 to ~~92.57~~ **43.79** µg/kg. **The sum of DDD + DDE + DDT exceeded the ER-M threshold at the 0- to 3-foot depth interval at VC1B-ALT and the 3- to 6-foot depth interval at VC1-ALT and VC1B-ALT with concentrations ranging from 73.50 to 92.57 µg/kg.**

Polychlorinated Biphenyls (PCBs)

Aroclor 1254 and Aroclor 1260 exceeded the ER-L in the following samples: 0- to 3 feet and 3- to 6 feet at VC1-ALT; and 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC1B-ALT. The concentrations of Aroclor 1254 that exceeded the ER-L threshold ranged from ~~47.6~~ **439.0** µg/kg to 714.0 µg/kg. The concentrations of Aroclor 125~~4~~**60** that exceeded the ER-L threshold ranged from 149 µg/kg to 340 µg/kg. The sum of Aroclors exceeded the ER-M threshold in the following samples: 0- to 3 feet and 3- to 6 feet at VC1-ALT; and 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC1B-ALT. The concentrations of the sum of Aroclors that exceeded the ER-M threshold ranged from 291.6 µg/kg to 1,140.0 µg/kg.

Dioxins/Furans

For these sample sites, the total toxicity equivalency factor exceeded the ER-M threshold in the following samples: the 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet depth intervals at VC1-ALT and VC1B-ALT; the 12- to 15 foot depth interval at VC1-ALT; the 6-to 9 foot depth interval at VC2; and the 0-to 3 feet, **and** 3- to 6 feet~~, and 6- to 9 feet~~ depth intervals at VC4. Concentrations in these samples ranged from 3.81 pg/g to 81.5 pg/g.

2.3.2 Group B – New York

Metals

Each sediment sample was analyzed for 27 metals, including mercury (see Appendix C for a complete list of these metals). Each of the 27 metals was detected in at least one sample in Group B (see Table B-7 for the sample sites where they were detected; due to the number of sample sites in Group B, this table is included in Appendix B). ~~Seven~~**Nine** of the detected metals exceeded Class A thresholds. Of these metals, nine have available Class A and/or Class C thresholds and all nine of these metals exceeded available NYSDEC thresholds. The metals detected within this segment that exceeded Class A and/or Class C thresholds are presented in Table B-8.

Cyanide

Total cyanide was detected in the following samples: 0- to 3 feet at VC7 and 9- to 12 feet at VC16 and VC17; 3- to 6 feet at VC3**5**; and 3- to 6 feet and 9- to 12 feet at VC36. Detected concentrations ranged from 0.25 mg/kg to 0.97 mg/kg.

Pesticides

<u>Non-Clamshell Dredge Sample Sites</u>

Of the 11 pesticides tested for in samples from sample sites where clamshell dredging would not occur, Class A and**/or** Class C thresholds are available for three pesticides and the sum of DDT+DDE+DDD. No pesticides tested for were detected in samples from these sample sites.

Possible Clamshell Dredge Sample Sites

Of the pesticides tested for in samples from sample sites where clamshell dredging may occur, 1**1**~~2~~ have available Class A and/or Class C thresholds. A total of 8 pesticides (aldrin, endosulfan sulfate, endrin, O,P'-DDD, O,P'-DDE, P,P'-DDD, P,P'-DDE, and P,P'-DDT) were detected in at least one sample in Group B. (See Table B-7 for a list of the sample sites where pesticides were detected.) Of the detected pesticides, only endrin and the sum of DDT + DDE + DDD have available Class A and Class C thresholds, and only the sum of DDT + DDE + DDD exceeded the Class A threshold. The sum of DDT + DDE + DDD exceeded the Class A threshold in the 0- to 3-foot sample~~s~~ at VC6 ~~and VC7; all samples from VC16 and VC17; and 0- to 3 foot sample at VC41-ALT. Concentrations that exceeded the Class A threshold ranged from 8.6 µg/kg to~~ **with a concentration of** 99.0 µg/kg.

Polycyclic Aromatic Hydrocarbons (PAHs)

Total PAHs detected only exceed the Class A criteria in the following samples: 0- **to** 3 **feet**, 3- to 6 feet, 6- to 9 feet, 9-to 12 feet at VC16 and VC17; ~~and~~ 0- to 3feet at VC37 and VC38**; and 3- to 6 feet and 6- to 9 feet at VC42**. The concentrations that exceeded the Class A threshold ranged from 4,540.0 µg/kg to 21,860.5 µg/kg.

Dioxins/Furans

Possible Clamshell Dredge Sample Sites

The total toxicity equivalency factor only exceeded the Class A threshold in samples from sites where clamshell dredging may occur. 2,3,7,8-tetrachlorodibenzo-P-Dioxin ~~did not~~ exceed**ed** the Class A threshold **in the 0- to 3 feet samples at VC6, VC7, and VC41-ALT; and the 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, 9- to 12 feet, and 12- to 15 feet samples at VC16 and VC17. The concentrations that exceeded the Class A threshold ranged from 0.53 pg/g to 13.2 pg/g**. The samples where the total toxicity equivalency factor exceeded the Class A threshold were **0- to 3 feet, and** 3- to 6 feet at VC6~~; 3- to 6 feet at~~ **and** VC7; 9- to 12 feet at VC8; 0- to 3 feet and 6- to 9 feet at VC15; **3- to 6 feet, 6- to 9 feet, and 9- to 12 feet at VC16 and VC17; 12- to 15 feet at VC16;** 0- to 3 feet and 6- to 9 feet at VC18; **0- to 3 feet at VC37;** and 3- to 6 feet at VC39. The concentrations that exceeded the Class A threshold ranged from 4.6 pg/g to ~~8.01~~ **39.7** pg/g.

2.3.3 Group C – New Jersey

Metals

Each sediment sample was analyzed for 27 metals, including mercury (see Appendix C for a complete list of these metals). Of these metals, 16 ER-L and/or ER-M thresholds are available. Within this sample segment, 25 metals tested for were detected in at least one sample in Group C (see Table 2-6 for the sample sites where metals were detected). The only two metals not detected were

antimony and **silver** ~~molybdenum~~. ~~Three~~ **Four** of the metals detected exceeded available NJDEP thresholds.

Selenium exceeded the ER-M threshold in the following samples: 0- to 3 feet at VC46; 9- to 12 feet at VC47; 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, 9- to 12 feet at VC50, 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, 9- to 12 feet at VC51; 3- to 6 feet, and 6- to 9 feet at VC52; 0- to 3 feet, 3- to 6 feet, **and** 6- to 9 feet~~, 9- to 12 feet~~ at VC53; 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, 9- to 12 feet, 12- to 15 feet, and 15- to 17.63 feet at VC54; 0- to 3 feet, 3- to 6 feet, 6- to 9 feet, ~~and~~ 9- to 12**, 12- to 15 feet, and 15- to 16** feet at VC54B; and 9- to 12 feet at VC55. Concentrations that exceeded the ER-M threshold ranged from 1.00 mg/kg to 4.13 mg/kg.

Cyanide

Samples from all sample sites in New Jersey were tested for cyanide. Cyanide was measured as total and amenable concentrations. There are no standard or guidance values available for total or amenable cyanide. Total cyanide was detected in three of the sample sites. Detections occurred at the 6- to 9.7-foot interval at location VC48-ALT, the 9- to 12-foot interval at location VC51, and the **0- to 3-foot,** 3- to 6-foot and 6- to ~~9~~9-foot intervals at location VC55 with concentrations ranging from 0.21 to 0.62 mg/kg.

Polychlorinated Biphenyls (PCBs)

Each sediment sample was analyzed for PCBs expressed as seven Aroclor compounds. Testing for ~~22~~**31** PCB congeners was also performed for samples from all sample sites in New Jersey. A complete list of the Aroclor compounds and PCB congeners included in the analysis is provided in Appendix C. ER-L thresholds are available for four Aroclor compounds and for the sum of Aroclor compounds, and an ER-M threshold is also available for the sum of Aroclor compounds. While there are no available thresholds for individual PCB congeners, there are ER-L and ER-M thresholds for total PCB congeners.

Polycyclic Aromatic Hydrocarbons (PAHs)

Each sediment sample was analyzed for 18 PAH compounds and 17 PAHs were detected (see Table 2-8 for a list of detected ~~SVOCs~~ **PAHs** and Table 2-6 for the sample sites where they were detected). The total toxicity equivalency factor exceeded the ER-M threshold in the 6-to 9 foot sample from sample site VC54 with a concentration of 5.83 pg/g.

Semi-Volatile Organic Compounds (SVOCs)

An additional 46 SVOCs were tested in samples from all sample sites in New Jersey (see Appendix C for a complete list of these SVOCs). ~~Five~~ **Six** of the 46 SVOCs were detected (4-chloroaniline, 4-methylphenol, benzyl butyl phthalate, **carbazole,** dibenzofu**r**an, and phenol). ER-L and/or ER-M thresholds for 16 SVOCs are available. No SVOCs exceeded the ER-L and/or ER-M thresholds.

2.3.4 Group D – New York

Dioxins/Furans

Three dioxins (1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin, 2,3,7,8-tetrachlorodibenzo-p-dioxin, and octachlorodibenzo-p-dioxin) and two furans (1,2,3,4,6,7,8-heptachlorodibenzofuran and 2,3,7,8-tetrachlorodibenzofuran) were detected in at least one sample in Group D (see Table 2.10 for the sample sites where they were detected). No detected dioxins or furans exceeded the Class A thresholds. The total toxicity equivalency factor did not exceed the Class A threshold.

2.3.5 Alternative Route

Metals

Table 2-11 Number of Detections at Sample Sites Along Alternative Route

	Number of Detections							
Sample Site[1]	VOCs	PAHs	Other SVOCs	Dioxins/ Furans	Pesticides	PCB Congener	PCB Aroclor	Metals
VC70	0	17	0	17	5	0	2	27
VC71	0	8	0	2	0	0	0	26
VC72	0	12	0	3	0	0	0	23
VC73-ALT	0	16	0	2	~~4~~ 3	0	~~3~~ 0	24
VC74	0	14	0	2	0	0	~~1~~ 0	22
VC75	0	17	0	2	~~1~~ 0	0	0	26
VC76	5	17	0	7	~~5~~ 4	0	~~3~~ 2	26
VC77	0	17	~~1~~ 5	7	~~4~~ 3	29	~~3~~ 2	26
VC78	0	17	~~5~~ 6	14	~~2~~ 1	28	0	27
VC79-ALT	0	16	~~2~~ 5	15	~~2~~ 1	27	0	26
VC80	0	16	~~0~~ 2	16	~~11~~ 10	30	2	27
VC81	0	17	~~0~~ 7	17	5	31	2	26
VC82	0	17	0	2	0	0	~~1~~ 0	25
VC83-ALT	0	0	0	2	0	0	0	24
VC84-ALT	0	0	0	2	0	0	0	24
VC85	0	11	0	2	0	0	0	24
VC86-ALT	0	11	0	1	0	0	0	24
VC87	0	2	0	2	0	0	0	24

Key:

[1] Dioxins and furans were analyzed only in the samples taken from 0- to 3 feet at sites in New York where clamshell dredging is not anticipated to occur. Dioxin/furan samples for additional depths at these sites were archived. PCB congeners were not tested at sites in New York where clamshell dredging is not anticipated to occur.

Cyanide

Samples from sampling sites in New York where clamshell dredging may occur were tested for cyanide (see Table B-1 for these locations). See above for discussion of analytes included in the analysis of samples at clamshell sites. Cyanide was measured as total and amenable concentrations. No standard or

guidance values are available for total or amenable cyanide. Total cyanide was detected in ~~three~~ **two** of the sample sites. Total cyanide was detected in the following samples: 0- to 3 feet, 3- to 6 feet, and 6- to 9 feet at VC80; and 0- to 3 feet, 6-to 9 feet, 9- to 12 feet, and 12- to 14.75 feet at VC81. Concentrations in these samples ranged from 0.38 mg/kg to 0.66 mg/kg.

Polychlorinated Biphenyls (PCBs)

Non-Clamshell Dredge Sample Sites

Three Aroclor compounds (Aroclor 1016, Aroclor 1254, and Aroclor 1260) were detected in at least one sample from the alternative route (see Table 2-11 for the sample sites where PCBs were detected). Class A thresholds were exceeded only for the sum of Aroclor compounds in the following samples: 0- to 3 feet at VC70 and VC76~~ and 9- to 12 feet at VC73 and VC74~~. Concentrations that exceeded the Class A threshold ranged from 300 µg/kg to 554 µg/kg.

Possible Clamshell Dredge Sample Sites

~~Three~~ **Two** Aroclor compounds (~~Aroclor 1016,~~ Aroclor 1254, and Aroclor 1260) and all 31 PCB congeners were detected in at least one sample in the alternative route (see Table 2.11 for the sample sites where they were detected). Class A thresholds were exceeded for both the sum of Aroclor compounds and total PCB congeners. The Class C threshold for total PCB congeners was also exceeded.

Semi-Volatile Organic Compounds (SVOCs)

Samples from sample sites in New York where clamshell dredging may occur (see Table B-1 for these locations) were tested for an additional 46 SVOCs. See above for discussion of analytes included in the analysis of samples at clamshell sites. See Appendix C for a complete list of these SVOCs. Six SVOCs have available Class A and/or Class C thresholds. Of those samples tested, ~~five~~ **nine** SVOCs were detected (**1,2,4-trichlorobenzene,** 4-methylphenol, **benzyl butyl phthalate,** bis(2-ethylhexyl) phthalate, carbazole, **dibenzofuran, diethyl phthalate, di-n-butyl phthalate,** ~~hexachlorocyclopentadiene,~~ and phenol) in at least one sample in the alternative route (see Table 2-11 for the sample sites where SVOCs were detected). None exceeded Class A thresholds.

3 Tables

Table B-3 Summary of Sediment Sample Analytical Results that Exceeded Available Thresholds for the Proposed Route

Analyte	No. of Sites (and Samples) Included in Analysis	No. of Sites (and Samples) Where Analyte was Detected	Maximum Result	Location of Maximum Result	No. of Exceedances (and Range of Values in Exceedance)[3]			
					NJDEP ER-L	NJDEP ER-M	NYSDEC Class A	NYSDEC Class C
Total BTEX (µg/kg)	69(314)	6(12)	9.81	VC16-D9-12E	N/A	N/A	0	0
Total PAHs (µg/kg)	69(313)	56(155)	21860.5	VC38-D0-3E	24(4230-21860.5)	0	24(4230-21860.5)	0
Dioxins and Furans Total Toxicity Equivalency Factor[1] (pg/g)	69(219)	66(202)	81.499	VC1-ALT-D0-3E	N/A	45(3.70104-81.499)	26(4.60107-81.499)	1(81.499-81.499)
P,P'-DDE (µg/kg)	69(313)	7(21)	56.5	VC1B-ALT-D3-6E	19(4.1-56.5)	N/A	0	N/A
Sum of DDT + DDE + DDD (µg/kg)	69(313)	7(21)	99.003	VC6-D0-3E	21(3.393-99.003)	~~N/A~~ **4(73.497-99.003)**	~~0~~ **4(8.672-99.003)**	0
Total PCB Congeners[2] (pg/g)	37(177)	28(135)	2000000	VC16-D9-12E	19(28800-2000000)	~~N/A~~ **15(233000-2000000)**	~~0~~ **16(149000-2000000)**	3(1160000-2000000)
Total PCB Aroclors (µg/kg)	69(313)	6(19)	1140	VC1B-ALT-D0-3E	19(93.9-1140)	~~0~~ **17(239.8-1140)**	~~0~~ **18(161.6-1140)**	1(1140-1140)
Aluminum (mg/kg)	69(314)	69(314)	18000	VC16-D0-3E	N/A	~~N/A~~ **0**	~~0~~ **N/A**	N/A
Antimony (mg/kg)	69(314)	8(23)	15.8	VC6-D0-3E	N/A	~~4(73.497-99.003)~~ **1(15.8-15.8)**	~~4(8.672-99.003)~~ **N/A**	N/A
Arsenic (mg/kg)	69(314)	69(314)	108	VC1-ALT-D3-6E	86(8.27-108)	~~15(233000-2000000)~~ **3(87.8-108)**	~~16(149000-2000000)~~ **86(8.27-108)**	3(87.8-108)
Barium (mg/kg)	69(314)	68(300)	135	VC1B-ALT-D0-3E	N/A	~~17(239.8-1140)~~ **17(48.8-135)**	~~18(161.6-1140)~~ **N/A**	N/A
Cadmium (mg/kg)	69(314)	43(146)	3.49	VC16-D6-9E	12(1.3-3.49)	0	~~N/A~~ **12(1.3-3.49)**	0
Chromium, Total (mg/kg)	69(314)	65(296)	202	VC16-D6-9E	14(86.5-202)	~~1(15.8-15.8)~~ **0**	~~N/A~~ **14(86.5-202)**	0
Cobalt (mg/kg)	69(314)	69(313)	16.3	VC16-D6-9E	N/A	~~3(87.8-108)~~ **45(10.1-16.3)**	~~86(8.27-108)~~ **N/A**	N/A
Copper (mg/kg)	69(314)	65(281)	504	VC1B-ALT-D0-3E	22(37.3-504)	~~17(48.8-135)~~ **2(307-504)**	~~N/A~~ **22(37.3-504)**	2(307-504)

Table B-3 Summary of Sediment Sample Analytical Results that Exceeded Available Thresholds for the Proposed Route

Analyte	No. of Sites (and Samples) Included in Analysis	No. of Sites (and Samples) Where Analyte was Detected	Maximum Result	Location of Maximum Result	No. of Exceedances (and Range of Values in Exceedance)[3]			
					NJDEP ER-L	NJDEP ER-M	NYSDEC Class A	NYSDEC Class C
Lead (mg/kg)	69(314)	69(314)	285	VC16-D6-9E	24(47.4-285)	~~0~~ **4(235-285)**	~~12(1.3-3.49)~~ **24(47.4-285)**	4(235-285)
Manganese (mg/kg)	69(314)	69(314)	906	VC16-D6-9E	N/A	~~0~~ **94(263-906)**	~~14(86.5-202)~~ **N/A**	N/A
Nickel (mg/kg)	69(314)	68(301)	53.5	VC16-D6-9E	86(21.4-53.5)	~~45(10.1-16.3)~~ **1(53.5-53.5)**	~~N/A~~ **86(21.4-53.5)**	1(53.5-53.5)
Selenium (mg/kg)	69(314)	46(167)	18.6	VC1-ALT-D3-6E	N/A	~~2(307-504)~~ **156(1.01-18.6)**	~~22(37.3-504)~~ **N/A**	N/A
Silver (mg/kg)	69(314)	13(34)	8.52	VC16-D9-12E	18(1.06-8.52)	~~4(235-285)~~ **8(3.92-8.52)**	~~24(47.4-285)~~ **18(1.06-8.52)**	8(3.92-8.52)
Vanadium (mg/kg)	69(314)	64(279)	94.4	VC22-D9-12E	N/A	~~94(263-906)~~ **6(61.7-94.4)**	N/A	N/A
Zinc (mg/kg)	69(314)	66(270)	494	VC16-D6-9E	18(154-494)	~~1(53.5-53.5)~~ **3(427-494)**	~~86(21.4-53.5)~~ **18(154-494)**	3(427-494)
Mercury (mg/kg)	69(314)	53(196)	5.28	VC1B-ALT-D0-3E	36(0.169-5.28)	~~156(1.01-18.6)~~ **21(0.904-5.28)**	~~N/A~~ **36(0.169-5.28)**	21(0.904-5.28)

Sources: NJDEP 2009, NYSDEC 2004, 2014c.

Notes:

1 Dioxins/Furans only analyzed in top 3 feet in NY non-clamshell.
2 Not analyzed in NY non-clamshell.
3 N/A indicates that there is no guidance value for that analyte, 0 indicates no exceedances of the guidance value. All measured concentrations of analytes detected in samples from New Jersey and New York were compared to both the SGVs and ESC.

Key:
DDD = dichloro-diphenyl-dichloroethane
DDE = dichloro-diphenyl-dichloroethylene
DDT = dichloro-diphenyl-trichloroethane
µg/kg = micrograms per kilogram
mg/kg = milligrams per kilogram
PCBs = polychlorinated biphenyls
PCBs = polychlorinated biphenyls
pg/g = picogram/gram

Table B-4 Count of Exceedances in New York and New Jersey per Number of Available SGVs and ESCs

Analyte Group	VC1-ALT[1]		VC1B-ALT[1]		VC2		VC3-ALT		VC4		VC5		VC6		VC7		VC8		VC9		VC10		VC11[2]		VC12[2]	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	4/15	0/6	3/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	1/15	0/6	2/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/0	0/15	0/0
PAHs[3]	16/18	1/1	13/18	1/1	0/18	0/1	0/18	0/1	1/18	0/1	0/18	0/1	7/18	0/1	8/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1
Dioxins/ Furans[3]	1/1	2/2	1/1	2/2	1/1	1/2	0/1	0/2	1/1	1/2	1/1	0/2	1/1	2/2	1/1	2/2	1/1	1/2	1/1	0/2	0/1	0/2	1/1	0/2	1/1	0/2
Pesticides[3]	4/11	1/11	4/11	1/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	4/11	1/11	4/11	~~1~~0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/4	0/11	0/4
PCB Congener[3]	1/1	1/1	1/1	1/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	1/1	1/1	1/1	1/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/0	0/1	0/0
PCB Aroclor[3]	3/5	1/1	3/5	1/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	3/5	1/1	3/5	1/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	14/16	9/9	14/16	9/9	2/16	1/9	5/16	2/9	7/16	4/9	5/16	2/9	15/16	9/9	13/16	9/9	5/16	2/9	4/16	2/9	0/16	0/9	0/16	0/9	2/16	1/9
Total	**43/71**	**15/36**	**39/71**	**15/36**	**3/71**	**2/36**	**5/71**	**2/36**	**9/71**	**5/36**	**6/71**	**2/36**	**32/71**	**14/36**	**32/71**	**14/36**	**6/71**	**3/36**	**5/71**	**2/36**	**0/71**	**0/36**	**1/71**	**0/22**	**3/71**	**1/22**

Analyte Group	VC13[2]		VC14[2]		VC15		VC16		VC17		VC18		VC19[2]		VC20[2]		VC21[2]		VC22[2]		VC23[2]		VC24[2]		VC25[2]	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	0/15	0/0	0/15	0/0	0/15	0/6	3/15	0/6	4/15	0/6	0/15	0/6	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0
PAHs[3]	0/18	0/1	0/18	0/1	0/18	0/1	17/18	1/1	17/18	1/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1
Dioxins/ Furans[3]	0/1	0/2	1/1	0/2	1/1	1/2	1/1	2/2	1/1	2/2	1/1	1/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2
Pesticides[3]	0/11	0/4	0/11	0/4	0/11	0/11	4/11	~~1~~0/11	4/11	1/11	~~1~~0/11	0/11	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4
PCB Congener[3]	0/1	0/0	0/1	0/0	0/1	0/1	1/1	1/1	1/1	1/1	0/1	0/1	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0
PCB Aroclor[3]	0/5	0/1	0/5	0/1	0/5	0/1	3/5	1/1	3/5	1/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	3/16	2/9	3/16	2/9	4/16	2/9	14/16	9/9	13/16	9/9	4/16	2/9	4/16	2/9	4/16	2/9	0/16	0/9	3/16	1/9	3/16	2/9	0/16	0/9	1/16	1/9
Total	**3/71**	**2/22**	**4/71**	**2/22**	**5/71**	**3/36**	**43/71**	**15/36**	**43/71**	**15/36**	**5/71**	**3/36**	**4/71**	**2/22**	**4/71**	**2/22**	**0/71**	**0/22**	**3/71**	**1/22**	**3/71**	**2/22**	**0/71**	**0/22**	**1/71**	**1/22**

Analyte Group	VC26[2]		VC27[2]		VC28[2]		VC29[2]		VC30[2]		VC31-ALT[2]		VC32[2]		VC33-ALT[2]		VC34[2]		VC35		VC36		VC37		VC38	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6
PAHs[3]	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	17/18	1/1	18/18	1/1
Dioxins/ Furans[3]	0/1	0/2	1/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	1/1	1/2	1/1	0/2
Pesticides[3]	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11
PCB Congener[3]	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/1	0/1	0/1	0/1	0/1	1/1	0/1
PCB Aroclor[3]	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	1/16	0/9	2/16	1/9	1/16	0/9	3/16	1/9	1/16	0/9	2/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9	5/16	4/9	11/16	7/9
Total	**1/71**	**0/22**	**3/71**	**1/22**	**1/71**	**0/22**	**3/71**	**1/22**	**1/71**	**0/22**	**2/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/36**	**1/71**	**0/36**	**23/71**	**6/36**	**31/71**	**8/36**

Analyte Group	VC39		VC40		VC41-ALT		VC42[2]		VC43[2]		VC44[2]		VC45-ALT		VC46		VC47		VC48-ALT		VC49		VC50		VC51	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6
PAHs[3]	0/18	0/1	0/18	0/1	0/18	0/1	17/18	1/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1
Dioxins/ Furans[3]	1/1	1/2	0/1	0/2	1/1	1/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2
Pesticides[3]	0/11	0/11	0/11	0/11	2/11	~~1~~0/11	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11
PCB Congener[3]	0/1	0/1	0/1	0/1	1/1	0/1	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1
PCB Aroclor[3]	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	1/16	0/9	1/16	0/9	2/16	1/9	8/16	6/9	1/16	0/9	0/16	0/9	0/16	0/9	1/16	0/9	3/16	2/9	1/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9
Total	**2/71**	**1/36**	**1/71**	**0/36**	**6/71**	**3/36**	**25/71**	**7/22**	**1/71**	**0/22**	**0/71**	**0/22**	**0/71**	**0/36**	**1/71**	**0/36**	**3/71**	**2/36**	**1/71**	**0/36**	**1/71**	**0/36**	**1/71**	**0/36**	**1/71**	**0/36**

Table B-4 Count of Exceedances in New York and New Jersey per Number of Available SGVs and ESCs

Analyte Group	VC52		VC53		VC54[1]		VC54B[1]		VC55[1]		VC55B[1]		VC56[2]		VC57[2]		VC58[2]		VC59[2]		VC60[2]		VC61-ALT[2]		VC62[2]	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/6	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0
PAHs[3]	0/18	0/1	0/18	0/1	15/18	1/1	15/18	1/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	1/18	~~1~~0/1	0/18	0/1	9/18	1/1
Dioxins/ Furans[3]	0/1	0/2	0/1	0/2	1/1	1/2	1/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2
Pesticides[3]	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4
PCB Congener[3]	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0
PCB Aroclor[3]	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	1/16	0/9	1/16	0/9	2/16	1/9	2/16	1/9	1/16	0/9	0/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9	1/16	0/9	5/16	4/9	2/16	1/9	2/16	2/9
Total	**1/71**	**0/36**	**1/71**	**0/36**	**18/71**	**3/36**	**18/71**	**2/36**	**1/71**	**0/36**	**0/71**	**0/36**	**1/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/22**	**6/71**	**5/22**	**2/71**	**1/22**	**11/71**	**3/22**

Analyte Group	VC63[2]		VC64[2]		VC65[2]		VC66[2]		VC67[2]		VC68[2]		VC69-ALT[2]	
	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY	NJ	NY
VOCs[3]	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5	0/4	0/5
SVOCs (excluding PAHs)[3]	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0	0/15	0/0
PAHs[3]	11/18	1/1	15/18	1/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1	0/18	0/1
Dioxins/ Furans[3]	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2	0/1	0/2
Pesticides[3]	1/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4	0/11	0/4
PCB Congener[3]	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0	0/1	0/0
PCB Aroclor[3]	2/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1	0/5	0/1
Metals[3]	0/16	0/9	1/16	1/9	1/16	1/9	0/16	0/9	0/16	0/9	1/16	0/9	1/16	0/9
Total	**14/71**	**1/22**	**16/71**	**2/22**	**1/71**	**1/22**	**0/71**	**0/22**	**0/71**	**0/22**	**1/71**	**0/22**	**1/71**	**0/22**

Key

[1] Both samples of each set (VC1-ALT/VC1B-ALT, VC54/VC54B, and VC55/VC55B) were collected in the same location.

[2] Dioxins and furans were tested for only in the samples taken from 0-3 feet at sites in New York where clamshell dredging is not anticipated to occur. Dioxin/furan samples for additional depths at these sites were archived. PCB Congeners were not tested for at sites in New York where clamshell dredging is not anticipated to occur.

[3] Highlighted cells indicate that ER-M or Class C thresholds were also exceeded.

Table B-7 Number of Detections at Sample Sites in Group B

Sample Site	Number of Detections							
	VOCs	PAHs	Other SVOCs	Dioxins/ Furans	Pesticides	PCB Congener	PCB Aroclor	Metals
VC5	0	7	2	2	0	24	0	26
VC6	0	17	7	14	5	30	2	27
VC7	0	17	9	14	5	29	2	27
VC8	0	0	2	4	0	5	0	25
VC9	0	2	1	2	0	13	0	23
VC10	0	7	1	1	0	2	0	21
VC11[1]	0	14	0	2	0	0	0	23
VC12[1]	0	16	0	2	0	0	0	25
VC13	0	17	0	2	0	0	0	24
VC14[1]	0	15	0	2	0	0	0	23
VC15	0	12	2	3	0	14	0	24
VC16	5	17	6	16	5	30	2	26
VC17	1	17	7	16	~~7~~ 6	30	2	26
VC18	0	11	2	3	0	20	0	24
VC19[1]	0	16	0	2	0	0	0	24
VC20[1]	0	17	0	2	0	0	0	24
VC21[1]	0	15	0	2	0	0	0	23
VC22[1]	0	15	0	3	0	0	0	23
VC23[1]	0	14	0	3	0	0	0	24
VC24[1]	0	15	0	2	0	0	0	24
VC25[1]	0	18	0	1	0	0	0	22
VC26	0	1	0	2	0	0	0	23
VC27[1]	0	6	0	2	0	0	0	25
VC28[1]	0	10	0	2	0	0	0	24
VC29[1]	0	4	0	2	0	0	0	24
VC30[1]	0	5	0	1	0	0	0	23
VC31-ALT[1]	0	1	0	1	0	0	0	24
VC32[1]	0	0	0	2	0	0	0	23
VC33-ALT[1]	0	1	0	1	0	0	0	23
VC34[1]	0	0	0	2	0	0	0	23
VC35	0	0	0	2	0	3	0	24
VC36	0	11	0	2	0	4	0	26
VC37	0	17	5	5	0	14	0	27
VC38	0	17	4	5	2	28	0	27
VC39	~~NY~~ 0	9	1	2	0	24	0	26
VC40	~~NY~~ 0	1	1	3	0	2	0	25
VC41-ALT	~~NY~~ 0	17	3	6	3	27	0	25
VC42[1]	~~NY~~ 0	18	0	3	0	0	0	25
VC43[1]	~~NY~~ 0	3	0	1	0	0	0	19
VC44[1]	~~NY~~ 0	3	0	1	0	0	0	19

Key:

1 - Dioxins and furans were only analyzed in the samples taken from 0-3 feet at sites in New York where clamshell dredging is not anticipated to occur. Dioxin/furan samples for additional depths at these sites were archived. PCB Congeners were not tested at sites in New York where clamshell dredging is not anticipated to occur.

Table B-8 Sample Sites and Samples in Group B Exceeding Class A and/or Class C Thresholds for Metals

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded Class A (mg/kg)	Exceeded Class C (mg/kg)		
Arsenic	8.61-37.54	87.8	VC5	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC6	***0-3 ft***, 3-6ft, 6-9 ft, 9-12 ft
			VC7	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC8	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC9	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC12	0-3 ft
			VC13	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC14	3-6 ft, 6-9 ft
			VC15	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16.5 ft
			VC16	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft
			VC17	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-17.5 ft
			VC18	3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16.2 ft
			VC19	3-6 ft, 6-9 ft, 9-12 ft
			VC20	6-9 ft, 9-12 ft
			VC22	9-12 ft
			VC23	9-12 ft
			VC25	9-11.5 ft
			VC37	0-3 ft
			VC38	0-3 ft
			VC42	3-6 ft
Cadmium	1.3-3.5	N/A	VC6	0-3 ft
			VC7	0-3 ft
			VC16	3-6 ft, 6-9 ft, 9-12 ft
			VC17	6-9 ft, 9-12 ft, 12-15 ft
Chromium	86.5-202.0	N/A	VC6	0-3 ft
			VC7	0-3 ft
			VC16	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC17	3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft
Copper	37.3-265.0	N/A	VC6	0-3 ft
			VC7	0-3 ft, 3-6 ft
			VC16	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft
			VC17	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-17.5 ft
			VC37	0-3 ft
			VC38	0-3 ft
			VC42	3-6 ft
Lead	51.0-198.0	235.0-285.0	VC6	0-3 ft
			VC7	0-3 ft, 3-6 ft
			VC16	0-3 ft, 3-6ft, ***6-9 ft, 9-12 ft***, 12-15 ft
			VC17	0-3 ft, 3-6ft, 6-9 ft, ***9-12 ft,*** 12-15 ft, 15-17.5 ft
			VC37	0-3 ft
			VC38	0-3 ft
			VC42	3-6 ft
Nickel	21.4-48.7	53.5	VC5	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC6	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft
			VC7	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC8	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC9	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC13	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC14	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft
			VC15	0-3 ft, f-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16.5 ft

Table B-8 Sample Sites and Samples in Group B Exceeding Class A and/or Class C Thresholds for Metals

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded Class A (mg/kg)	Exceeded Class C (mg/kg)		
			VC16	0-3 ft, 3-6ft, ***6-9 ft,*** 9-12 ft, 12-15 ft
			VC17	0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-17.5 ft
			VC18	**0-3 ft,** 3-6 ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-16.2 ft
			VC19	3-6 ft, 6-9 ft, 9-12 ft
			VC20	3-6 ft, 6-9 ft, 9-12 ft
			VC23	6-9 ft
			VC27	0-3 ft, 3-6 ft
			VC29	9-12 ft
			VC38	0-3 ft
			VC42	3-6 ft
Silver	1.06-3.55	4.78-8.52	VC6	0-3 ft
			VC7	***0-3 ft***
			VC16	0-3 ft, ***3-6 ft, 6-9 ft, 9-12 ft,*** 12-15 ft
			VC17	0-3 ft, 3-6 ft, ***6-12 ft,*** 12-17.5 ft
			VC38	0-3 ft
Zinc	154-390	427-494	VC6	0-3 ft
			VC7	0-3 ft, 3-6 ft
			VC16	0-3 ft, 3-6 ft, ***6-9 ft, 9-12 ft***
			VC17	**0-3 ft,** 3-6 ft, 6-9 ft, ***9-12 ft,*** 12-~~17.5~~ **15** ft
			VC38	0-3 ft
			VC42	**3-6 ft**
Mercury	0.265-0.515	0.904-4.22	VC6	***0-3 ft,*** 3-6 ft
			VC7	***0-3 ft, 3-6 ft***
			VC16	***0-3 ft, 3-6 ft, 6-9 ft, 9-12 ft,*** 12-15 ft
			VC17	***0-3 ft, 3-6ft, 6-9 ft, 9-12 ft, 12-15 ft, 15-17.5 ft***
			VC37	***0-3 ft***
			VC38	***0-3 ft***
			VC41-ALT	0-3 ft
			VC42	***3-6 ft,*** **6-9 ft**

Key:
* Sample depths where Class C thresholds were exceeded are in bold and italics.

Table B-9 Sample Sites and Samples in Group C Exceeding ER-L and/or ER-M Thresholds for PAHs

Analyte	Concentrations that Exceeded Thresholds		Sample Site	Sample Depth(s)*
	Exceeded ER-L (µg/kg)	Exceeded ER-M (µg/kg)		
Acenaphthene	39.4-172	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	0-3 ft, 3-6 ft, 6-9 ft
Acenaphthylene	46.3-131	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Anthracene	122-989	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	0-3 ft, 3-6 ft, 6-9 ft
Benzo(A)Anthracene	282-1580	1670	VC54	0-3 ft, 3-6 ft, ***6-9 ft***
			VC54B	0-3 ft, 3-6 ft, 6-9 ft
Benzo(A)Pyrene	861-1330	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Benzo(G,H,I)Perylene	440-708	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Benzo(K)Fluoranthene	369-488	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Chrysene	733-1160	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Dibenz(A,H)Anthracene	134-211	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Fluoranthene	1680-2490	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Fluorene	46-263	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	**0-3 ft,** 3-6 ft, 6-9 ft
Indeno(1,2,3-C,D)Pyrene	457-753	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Phenanthrene	324-1440	1540-1570	VC54	0-3 ft, ***3-6 ft,*** 6-9 ft
			VC54B	**0-3 ft,** 3-6 ft, ***6-9 ft***
Pyrene	1530-2270	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft
Total PAHs	10400-15400	N/A	VC54	0-3 ft, 3-6 ft, 6-9 ft
			VC54B	3-6 ft, 6-9 ft

Key:
* Sample depths where ER-M thresholds were exceeded are in bold and italics.
N/A = Detected concentrations did not exceed the ER-M thresholds.

Addendum to the Fall/Winter 2016 Offshore Environmental Sampling Report for the Northeast Supply Enhancement Project New Jersey, New York

October 2017

Prepared for:

Transcontinental Gas Pipe Line Company, LLC

Prepared by:

ECOLOGY AND ENVIRONMENT, INC.
368 Pleasant View Drive
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List of Abbreviations and Acronyms

E & E	Ecology and Environment, Inc.
ETRA	Environmental Toxicology and Risk Assessment
FERC	Federal Energy Regulatory Commission
Project	Northeast Supply Enhancement Project
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan
Transco	Transcontinental Gas Pipe Line Company, LLC
USACE	U.S. Army Corps of Engineers

1 Introduction

1.1 Project Description

Transcontinental Gas Pipe Line Company, LLC (Transco), has filed an application with the Federal Energy Regulatory Commission (FERC) for a Certificate of Public Convenience and Necessity (Certificate) for the Northeast Supply Enhancement Project (Project) (Docket No. CP17-101-000). The Offshore Environmental Sampling Report was prepared to facilitate FERC's review and development of an environmental impact statement for the Project and to support the corresponding state-level environmental evaluation of the Project. On June 27, 2017, Transco submitted its Offshore Environmental Sampling Report as Appendix J of Transco's application for a U.S. Army Corps of Engineers (USACE) permit. On June 30, 2017, Transco submitted its Offshore Environmental Sampling Report to FERC. See the Offshore Environmental Sampling Report for a Project Description. On September 7, 2017, Transco submitted an initial Addendum to the sampling report as Attachment 6 to Volume 1 of Supplemental Information Filing 3 to Transco's application for a FERC Certificate.

1.2 Scope of Work

Transco conducted a supplemental field survey and laboratory analysis in May 2017 to evaluate the physical and chemical characteristics of sediment at the proposed anode sled installation and to further evaluate chemical data along the Raritan Bay Loop route. The survey was conducted to support evaluation of the route for both engineering design and environmental review purposes. Supplementary vibracore samples were collected from sample sites in the 2016 survey where recovery to depth was incomplete. Laboratory analysis was completed for a focused list of contaminants, specifically metals, based on historical contamination documented in the area. Transco contracted Ecology and Environment, Inc. (E & E) to support the environmental permitting effort for the Project. The Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP) developed by E & E and reviewed by regulatory agencies for the previous offshore survey was used for this supplemental field survey. The SAP/QAPP prepared for the offshore component of the Project is provided in Appendix C of the Offshore Environmental Sampling Report. Agency comments on initial versions of the SAP/QAPP are provided in Appendix D of the Offshore Environmental Sampling Report. Transco contracted Alpine Ocean (a Gardline company) to conduct the survey and arrange for sediment testing at onshore laboratories.

1.3 Spring 2017 Survey Results

Attached to this report are grain size statistics (see Appendix A), and the previously unsubmitted core logs for samples collected in the 2017 survey (see Appendix B).

A Grain Size Results

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC11	VC11	VC11	VC11	VC11	VC11	VC11	VC25	VC25	VC25	VC25
Sample or Section No		0-2	0-2	2-4	4-6	6-8	8-10	10-12	0-2	0-2	2-4	4-6
Depth in ft		0.45	1.90	4.00	6.00	8.00	10.00	12.00	0.50	2.00	4.00	6.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100	100.0	100	100	100	100.0	100	100.0	100	100	100
1 1/2 inch	38.1	100	100.0	100	100	100	100.0	100	100.0	100	100	100
1 inch	25.4	100	100.0	99	100	100	100.0	100	100.0	100	100	100
3/4 inch	19.05	100	100.0	95	100	100	100.0	100	100.0	100	100	100
1/2 inch	12.7	100	100.0	91	98	95	100.0	100	100.0	100	96	100
3/8 inch	9.525	100	100.0	87	93	92	100.0	99	100.0	97	84	98
1/4 inch	6.35	97	100.0	83	88	86	100.0	96	99.5	93	79	96
4	4.75	96	100.0	79	85	84	99.7	95	99.2	87	74	96
10	2.00	89	99.6	68	65	77	98.8	88	97.0	70	65	93
20	0.85	69	98.5	54	29	48	95.5	71	82.7	55	56	81
40	0.425	40	81.4	38	6	7	60.9	37	50.8	32	35	63
60	0.250	18	30.3	12	2	2	16.6	10	36.3	15	16	35
100	0.150	12	5.3	3	1	1	3.1	2	23.9	7	6	7
140	0.106	11	2.0	2	1	1	2.4	2	14.2	5	4	3
200	0.075	**10**	**1.5**	**2**	**1**	**1**	**2.1**	**1**	**10.6**	**4**	**4**	**2**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC25	VC25	VC25	VC26	VC26	VC26	VC26	VC26	VC26	VC26	VC28
Sample or Section No		6-8	8-10	10-12	0-2	0-2	2-4	4-6	6-8	8-10	10-12	0-2
Depth in ft		8.00	10.00	12.00	0.50	2.00	4.00	6.00	8.00	10.00	12.00	0.60
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100	100	100	100.0	100	100	100	100	100.0	100.0	100.0
1 1/2 inch	38.1	100	100	100	100.0	100	100	100	100	100.0	100.0	100.0
1 inch	25.4	100	100	100	100.0	100	100	100	100	100.0	100.0	100.0
3/4 inch	19.05	100	100	100	100.0	100	94	100	100	100.0	100.0	100.0
1/2 inch	12.7	97	100	98	100.0	100	80	100	100	100.0	100.0	100.0
3/8 inch	9.525	95	95	98	99.8	97	72	99	95	99.2	100.0	100.0
1/4 inch	6.35	92	92	97	99.8	89	67	98	92	99.2	100.0	99.5
4	4.75	89	90	96	99.8	87	64	96	87	99.1	99.4	99.0
10	2.00	76	85	92	99.6	82	54	92	70	97.8	97.3	97.6
20	0.85	57	68	78	98.8	75	41	86	53	93.5	86.0	92.2
40	0.425	27	27	34	89.9	58	24	73	31	70.6	43.6	72.0
60	0.250	6	4	5	36.1	35	10	57	17	19.2	8.0	50.8
100	0.150	3	2	3	4.7	13	5	46	8	4.9	1.9	34.3
140	0.106	3	1	3	2.6	8	4	42	6	3.6	1.3	22.1
200	0.075	**3**	**1**	**3**	**1.9**	**7**	**4**	**39**	**5**	**3.2**	**1.2**	**16.9**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC28	VC28	VC28	VC28	VC28	VC28	VC30	VC30	VC30	VC30	VC30
Sample or Section No		0-2	2-4	4-6	6-8	8-10	10-12	0-2	0-2	2-4	4-6	6-8
Depth in ft		2.00	4.00	6.00	8.00	10.00	12.00	0.60	2.00	4.00	6.00	8.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100	100.0	100	100	100	100	100.0	100	100	100	100.0
1 1/2 inch	38.1	100	100.0	100	100	100	100	100.0	100	100	100	100.0
1 inch	25.4	100	100.0	100	100	100	100	100.0	100	100	100	100.0
3/4 inch	19.05	100	100.0	100	100	100	100	100.0	100	100	100	100.0
1/2 inch	12.7	100	100.0	97	100	100	100	100.0	100	100	100	100.0
3/8 inch	9.525	98	100.0	97	99	100	100	100.0	98	100	87	100.0
1/4 inch	6.35	97	100.0	97	94	99	100	100.0	96	95	78	100.0
4	4.75	95	99.6	97	91	97	98	99.5	96	94	74	100.0
10	2.00	91	98.6	93	83	88	96	97.8	90	86	67	99.9
20	0.85	85	93.9	78	72	69	95	92.5	85	65	58	99.0
40	0.425	71	82.3	53	59	24	89	85.7	78	36	32	91.8
60	0.250	53	33.6	27	24	9	61	77.4	71	22	11	52.1
100	0.150	33	20.6	16	9	4	8	56.7	53	11	3	10.2
140	0.106	19	18.7	14	7	3	3	17.2	20	7	2	4.3
200	0.075	**14**	**17.6**	**13**	**6**	**3**	**2**	**10.5**	**11**	**6**	**2**	**3.1**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC30	VC30	VC33	VC33	VC33	VC33	VC33	VC33	VC33	VC34	VC34
Sample or Section No		8-10	10-12	0-2	0-2	2-4	4-6	6-8	8-10	10-12	0-2	0-2
Depth in ft		10.00	12.00	0.70	2.00	4.00	6.00	8.00	10.00	11.70	0.70	2.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100	100.0	100.0	100.0	100	100	100	100	100	100.0
1 1/2 inch	38.1	100.0	100	100.0	100.0	100.0	100	100	100	100	100	100.0
1 inch	25.4	100.0	100	100.0	100.0	100.0	100	100	100	100	100	100.0
3/4 inch	19.05	100.0	100	100.0	100.0	100.0	100	100	100	100	100	100.0
1/2 inch	12.7	100.0	100	100.0	100.0	100.0	100	100	100	100	100	100.0
3/8 inch	9.525	100.0	100	100.0	100.0	100.0	98	94	98	98	99	100.0
1/4 inch	6.35	100.0	98	100.0	99.4	100.0	95	87	97	94	99	100.0
4	4.75	99.9	96	100.0	99.1	99.8	94	83	95	91	98	100.0
10	2.00	99.6	90	99.9	96.2	99.1	92	76	91	82	97	98.4
20	0.85	97.4	77	99.0	86.6	97.2	85	67	85	75	88	90.2
40	0.425	53.7	51	71.5	72.3	90.2	50	32	64	42	48	67.1
60	0.250	12.8	22	17.0	56.5	66.3	17	9	39	11	17	26.5
100	0.150	3.6	6	3.5	28.4	26.9	6	2	9	4	4	3.6
140	0.106	1.6	3	2.5	7.3	18.5	5	2	4	3	3	2.1
200	0.075	**1.3**	**3**	**1.9**	**5.3**	**14.9**	**4**	**2**	**3**	**3**	**2**	**1.8**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC34	VC34	VC34	VC34	VC34	VC42	VC42	VC42	VC42	VC42	VC42
Sample or Section No		2-4	4-6	6-8	8-10	10-12	0-2	0-2	2-4	4-6	6-8	8-10
Depth in ft		4.00	6.00	8.00	10.00	12.00	0.50	2.00	4.00	5.90	8.00	10.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100.0	100.0	100	100.0	100	100.0	100	100	100	100.0
1 1/2 inch	38.1	100.0	100.0	100.0	100	100.0	100	100.0	100	100	100	100.0
1 inch	25.4	100.0	100.0	100.0	100	100.0	100	100.0	100	100	100	100.0
3/4 inch	19.05	100.0	100.0	100.0	100	100.0	100	100.0	100	100	100	100.0
1/2 inch	12.7	100.0	100.0	100.0	100	100.0	99	100.0	99	99	99	100.0
3/8 inch	9.525	100.0	100.0	100.0	100	100.0	98	100.0	99	97	98	100.0
1/4 inch	6.35	100.0	100.0	99.9	99	99.6	97	100.0	97	92	95	100.0
4	4.75	100.0	100.0	99.7	98	99.3	97	99.9	97	91	93	99.9
10	2.00	99.8	99.9	97.7	96	97.5	95	99.7	95	90	89	99.6
20	0.85	97.2	99.2	78.5	92	84.5	95	99.6	94	90	89	99.4
40	0.425	52.5	83.6	26.7	79	41.5	88	93.8	92	90	88	98.8
60	0.250	15.6	49.2	7.9	25	7.9	46	51.0	81	89	84	96.4
100	0.150	5.2	22.2	5.0	4	3.2	4	4.4	20	84	38	24.6
140	0.106	2.3	12.8	3.7	2	2.1	2	2.2	15	83	27	6.0
200	0.075	**1.9**	**8.1**	**3.2**	**2**	**1.8**	**2**	**1.9**	**14**	**82**	**22**	**4.3**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC42	VC43	VC43	VC43	VC43	VC43	VC43	VC43	VC46	VC46	VC46
Sample or Section No		10-12	0-2	0-2	2-4	4-6	6-8	8-10	10-12	0-2	0-2	2-4
Depth in ft		12.00	0.50	2.00	4.00	6.00	8.00	10.00	12.00	0.50	2.00	4.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100	100.0	100.0
1 1/2 inch	38.1	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100	100.0	100.0
1 inch	25.4	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	96	100.0	100.0
3/4 inch	19.05	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	91	100.0	100.0
1/2 inch	12.7	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	88	100.0	100.0
3/8 inch	9.525	100.0	100.0	100.0	100.0	100.0	99.9	100.0	100.0	84	100.0	100.0
1/4 inch	6.35	100.0	100.0	100.0	100.0	100.0	99.9	99.9	99.6	83	100.0	100.0
4	4.75	100.0	100.0	100.0	100.0	99.9	99.8	99.4	99.5	82	99.9	100.0
10	2.00	99.6	100.0	100.0	100.0	99.9	99.4	98.5	97.8	80	99.8	99.9
20	0.85	99.2	100.0	99.9	99.9	99.9	99.1	98.0	94.2	79	99.6	99.8
40	0.425	97.1	96.2	97.0	95.1	97.0	92.6	97.2	82.1	68	84.1	88.1
60	0.250	89.3	50.0	54.4	49.5	60.5	52.4	91.7	43.7	47	21.6	23.8
100	0.150	21.0	2.6	3.3	2.7	5.2	4.6	16.1	9.8	21	2.0	2.3
140	0.106	4.7	1.4	1.1	1.3	1.4	1.8	2.6	4.0	17	1.0	1.3
200	0.075	**3.1**	**1.3**	**1.0**	**1.1**	**1.1**	**1.4**	**1.8**	**3.3**	**15**	**0.9**	**1.1**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC46	VC46	VC46	VC46	VC47	VC47	VC47	VC47	VC47	VC47	VC47
Sample or Section No		4-6	6-8	8-10	10-12	0-2	0-2	2-4	4-6	6-8	8-10	10-12
Depth in ft		6.00	8.00	10.00	12.00	0.70	2.00	4.00	6.00	8.00	10.00	12.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100.0	100.0	100.0	100.0	100	100	100.0	100.0	100	100.0
1 1/2 inch	38.1	100.0	100.0	100.0	100.0	100.0	100	100	100.0	100.0	100	100.0
1 inch	25.4	100.0	100.0	100.0	100.0	100.0	100	100	100.0	100.0	100	100.0
3/4 inch	19.05	100.0	100.0	100.0	100.0	100.0	100	100	100.0	100.0	100	100.0
1/2 inch	12.7	100.0	100.0	100.0	100.0	98.5	98	96	100.0	100.0	100	100.0
3/8 inch	9.525	100.0	100.0	100.0	100.0	98.5	98	93	100.0	100.0	100	100.0
1/4 inch	6.35	100.0	100.0	100.0	100.0	97.9	97	89	100.0	100.0	99	100.0
4	4.75	99.9	100.0	100.0	100.0	97.9	96	86	100.0	100.0	99	99.9
10	2.00	99.8	99.4	100.0	99.8	97.7	94	81	99.6	99.6	95	99.2
20	0.85	99.7	98.6	99.8	98.1	97.2	92	79	99.3	98.7	88	91.4
40	0.425	94.4	93.9	93.7	52.5	78.7	76	74	89.6	87.3	64	84.1
60	0.250	35.9	48.5	44.5	12.0	25.4	26	69	31.6	35.4	28	46.5
100	0.150	3.6	6.6	2.8	2.2	2.6	4	57	4.2	4.1	4	4.6
140	0.106	1.3	2.3	1.4	1.5	1.7	3	54	2.3	2.0	2	3.3
200	0.075	**1.1**	**1.9**	**1.2**	**1.3**	**1.6**	**3**	**50**	**2.0**	**1.7**	**2**	**3.1**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC49	VC49	VC49	VC49	VC49	VC49	VC49	VC50	VC50	VC50	VC50
Sample or Section No		0-2	0-2	2-4	4-6	6-8	8-10	10-12	0-2	0-2	2-4	4-6
Depth in ft		0.10	2.00	4.00	6.00	8.00	10.00	12.00	0.70	2.00	4.00	6.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1 1/2 inch	38.1	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1 inch	25.4	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
3/4 inch	19.05	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1/2 inch	12.7	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
3/8 inch	9.525	100.0	100.0	100.0	100.0	100.0	100.0	100.0	99.3	100.0	100.0	100.0
1/4 inch	6.35	100.0	100.0	100.0	100.0	100.0	100.0	100.0	97.3	100.0	100.0	100.0
4	4.75	100.0	100.0	100.0	100.0	100.0	100.0	100.0	96.7	100.0	100.0	100.0
10	2.00	99.7	100.0	100.0	100.0	100.0	100.0	100.0	95.3	100.0	99.7	99.5
20	0.85	99.6	99.9	99.9	99.9	100.0	99.9	100.0	90.2	99.8	97.1	93.9
40	0.425	99.5	99.8	99.5	97.8	99.7	99.5	99.9	68.2	98.6	71.2	57.1
60	0.250	82.0	65.7	58.7	17.5	90.2	78.5	89.2	37.0	85.6	30.8	14.7
100	0.150	1.9	1.4	1.5	0.8	3.7	3.7	5.8	2.9	5.4	4.0	3.7
140	0.106	0.6	0.6	0.5	0.6	1.1	1.2	1.3	1.7	1.1	1.8	2.3
200	0.075	**0.5**	**0.6**	**0.5**	**0.5**	**1.0**	**1.0**	**1.1**	**1.5**	**0.9**	**1.5**	**2.1**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC50	VC50	VC50	VC51	VC51	VC51	VC51	VC51	VC51	VC52	VC52
Sample or Section No		6-8	8-10	10-12	0-2	0-2	2-4	4-6	6-8	8-10	0-2	0-2
Depth in ft		8.00	10.00	12.00	0.70	2.00	4.00	6.00	8.00	9.30	0.70	2.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100.0	100.0	100.0	100.0	100.0	100	100.0	100.0	100.0	100.0
1 1/2 inch	38.1	100.0	100.0	100.0	100.0	100.0	100.0	100	100.0	100.0	100.0	100.0
1 inch	25.4	100.0	100.0	100.0	100.0	100.0	100.0	100	100.0	100.0	100.0	100.0
3/4 inch	19.05	100.0	100.0	100.0	100.0	100.0	100.0	100	100.0	100.0	100.0	100.0
1/2 inch	12.7	100.0	100.0	100.0	99.6	100.0	100.0	100	100.0	100.0	100.0	100.0
3/8 inch	9.525	100.0	100.0	100.0	99.6	100.0	100.0	99	100.0	100.0	100.0	100.0
1/4 inch	6.35	100.0	100.0	100.0	99.4	100.0	100.0	99	100.0	100.0	99.7	100.0
4	4.75	100.0	100.0	100.0	99.3	100.0	100.0	99	99.9	100.0	99.7	99.9
10	2.00	99.9	99.9	100.0	99.0	99.9	99.9	97	99.7	99.6	99.3	99.8
20	0.85	99.6	99.6	99.8	97.4	98.6	99.8	90	99.0	97.8	97.4	99.7
40	0.425	93.9	96.1	97.1	73.9	91.8	97.5	63	66.3	80.8	87.5	98.5
60	0.250	63.9	72.6	80.9	21.9	53.2	75.6	27	20.3	35.1	45.0	88.4
100	0.150	5.5	8.6	11.3	1.6	6.9	13.5	17	2.1	4.5	1.8	11.2
140	0.106	1.7	2.4	2.0	1.0	1.8	5.0	9	1.4	2.6	1.0	1.8
200	0.075	**1.4**	**1.8**	**1.3**	**0.9**	**1.6**	**4.2**	**7**	**1.3**	**2.4**	**1.0**	**1.2**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC52	VC52	VC52	VC52	VC52	VC53	VC53	VC53	VC53	VC53	VC53
Sample or Section No		2-4	4-6	6-8	8-10	10-12	0-2	0-2	2-4	4-6	6-8	8-10
Depth in ft		4.00	6.00	8.00	10.00	12.00	0.70	2.00	4.00	6.00	8.00	10.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1 1/2 inch	38.1	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1 inch	25.4	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
3/4 inch	19.05	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1/2 inch	12.7	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
3/8 inch	9.525	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1/4 inch	6.35	100.0	100.0	100.0	100.0	99.9	99.1	100.0	100.0	100.0	100.0	100.0
4	4.75	100.0	100.0	100.0	99.9	99.7	99.1	100.0	100.0	100.0	100.0	100.0
10	2.00	99.9	100.0	100.0	99.9	95.7	98.8	99.9	100.0	100.0	100.0	99.9
20	0.85	98.5	99.9	100.0	99.8	85.1	97.9	99.8	100.0	100.0	100.0	99.8
40	0.425	95.6	99.8	99.8	96.7	62.1	92.6	94.2	99.0	99.5	99.4	99.1
60	0.250	91.3	98.6	99.0	92.2	32.3	56.5	49.6	69.4	74.9	87.4	89.2
100	0.150	22.1	21.8	26.8	19.5	15.4	1.7	3.2	4.9	6.4	11.6	16.4
140	0.106	3.4	2.5	2.9	3.2	10.8	0.9	1.1	2.1	2.0	3.8	6.1
200	0.075	**2.5**	**1.6**	**2.0**	**2.2**	**9.7**	**0.9**	**0.8**	**1.6**	**1.5**	**2.0**	**3.6**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC55	VC55	VC55	VC55	VC55	VC55	VC55	VC56	VC56	VC56	VC56
Sample or Section No		0-2	0-2	2-4	4-6	6-8	8-10	10-12	0-2	0-2	2-4	4-6
Depth in ft		0.70	2.00	4.00	6.00	8.00	10.00	11.90	0.70	2.00	4.00	6.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100	100.0	100.0	100	100	100	100	100.0	100	100.0	100.0
1 1/2 inch	38.1	100	100.0	100.0	100	100	100	100	100.0	100	100.0	100.0
1 inch	25.4	100	100.0	100.0	100	100	100	100	100.0	100	100.0	100.0
3/4 inch	19.05	100	100.0	100.0	100	100	100	100	100.0	98	100.0	100.0
1/2 inch	12.7	100	100.0	100.0	100	100	98	100	100.0	98	100.0	100.0
3/8 inch	9.525	100	100.0	100.0	100	100	96	100	98.7	95	100.0	100.0
1/4 inch	6.35	99	100.0	100.0	98	99	92	98	98.7	86	99.7	100.0
4	4.75	99	100.0	100.0	96	98	86	94	98.2	77	99.7	99.6
10	2.00	96	99.2	99.9	94	92	70	80	97.4	61	98.4	97.6
20	0.85	78	85.0	94.0	92	77	48	64	92.8	45	94.6	93.8
40	0.425	33	29.2	54.6	63	36	17	28	23.5	24	64.9	72.4
60	0.250	8	6.7	13.3	20	9	4	5	3.3	10	17.0	14.1
100	0.150	1	1.5	2.2	3	4	1	1	1.2	1	2.2	1.4
140	0.106	1	0.9	1.0	1	4	1	1	0.9	1	1.0	1.1
200	0.075	**1**	**0.8**	**0.8**	**1**	**3**	**1**	**1**	**0.8**	**1**	**0.9**	**1.1**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC56	VC56	VC56	VC58	VC58	VC58	VC58	VC58	VC58	VC58	VC59
Sample or Section No		6-8	8-10	10-12	0-2	0-2	2-4	4-6	6-8	8-10	10-12	0-2
Depth in ft		8.00	10.00	12.00	0.20	2.00	4.00	6.00	8.00	10.00	12.00	0.70
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100	100.0	100	100.0	100	100	100	100.0	100.0	100
1 1/2 inch	38.1	100.0	100	100.0	100	100.0	100	100	100	100.0	100.0	100
1 inch	25.4	100.0	100	100.0	100	100.0	100	100	100	100.0	100.0	100
3/4 inch	19.05	100.0	100	100.0	100	100.0	100	100	100	100.0	100.0	100
1/2 inch	12.7	100.0	100	100.0	100	100.0	100	96	100	100.0	100.0	95
3/8 inch	9.525	100.0	100	100.0	97	100.0	100	87	100	100.0	100.0	94
1/4 inch	6.35	100.0	99	100.0	92	99.2	95	72	100	100.0	99.8	91
4	4.75	100.0	96	100.0	90	99.2	90	64	98	99.8	99.4	89
10	2.00	99.2	82	99.1	85	99.2	75	55	92	98.8	98.9	86
20	0.85	95.2	61	94.8	82	98.6	64	49	80	94.9	98.7	83
40	0.425	52.4	21	43.7	73	83.7	43	28	38	44.9	86.2	73
60	0.250	20.5	5	9.7	51	18.9	4	5	9	8.8	26.0	34
100	0.150	1.4	1	1.7	16	1.2	1	1	1	1.1	5.7	5
140	0.106	1.0	1	1.0	3	0.8	1	1	1	0.9	3.9	2
200	0.075	**0.9**	**1**	**1.0**	**1**	**0.8**	**1**	**1**	**1**	**0.9**	**3.2**	**2**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC59	VC59	VC59	VC59	VC59	VC59	VC60	VC60	VC60	VC60	VC60
Sample or Section No		0-2	2-4	4-6	6-8	8-10	10-12	0-2	0-2	2-4	4-6	6-8
Depth in ft		2.00	4.00	6.00	8.00	10.00	12.00	0.30	2.00	4.00	6.00	8.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100.0	100.0	100	100	100	100	100	100	100.0	100.0
1 1/2 inch	38.1	100.0	100.0	100.0	100	100	100	100	100	100	100.0	100.0
1 inch	25.4	100.0	100.0	100.0	100	100	100	100	100	100	100.0	100.0
3/4 inch	19.05	100.0	100.0	100.0	100	100	100	100	100	100	100.0	100.0
1/2 inch	12.7	100.0	100.0	100.0	100	100	100	98	92	100	100.0	100.0
3/8 inch	9.525	100.0	100.0	100.0	100	100	100	98	87	100	100.0	100.0
1/4 inch	6.35	99.7	99.9	100.0	99	84	100	97	79	97	99.9	100.0
4	4.75	99.4	99.9	99.8	98	66	99	96	76	97	99.8	100.0
10	2.00	97.7	99.4	99.1	93	44	97	93	69	96	99.7	99.9
20	0.85	90.1	98.8	96.3	75	34	93	90	64	78	99.4	99.3
40	0.425	66.5	94.5	82.6	33	12	80	73	50	39	87.2	80.6
60	0.250	43.0	71.5	23.3	17	3	14	32	32	15	38.2	17.0
100	0.150	9.4	12.6	2.7	1	1	2	3	3	2	3.8	2.1
140	0.106	2.2	2.2	1.5	1	1	1	1	1	1	2.8	1.7
200	0.075	**1.2**	**1.4**	**1.3**	**1**	**1**	**1**	**1**	**1**	**1**	**2.5**	**1.6**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC60	VC61	VC61	VC61	VC61	VC61	VC61	VC61	VC62	VC62	VC62
Sample or Section No		8-10	0-2	0-2	2-4	4-6	6-8	8-10	10-12	0-2	0-2	2-4
Depth in ft		10.00	0.70	2.00	4.00	6.00	8.00	10.00	12.00	0.70	2.00	4.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	97	100	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100
1 1/2 inch	38.1	100.0	97	100	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100
1 inch	25.4	100.0	96	100	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100
3/4 inch	19.05	100.0	93	99	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100
1/2 inch	12.7	100.0	89	87	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100
3/8 inch	9.525	100.0	87	78	99.6	100.0	100.0	100.0	100.0	100.0	100.0	100
1/4 inch	6.35	100.0	84	66	99.6	100.0	100.0	100.0	100.0	100.0	99.9	99
4	4.75	100.0	84	61	99.3	100.0	100.0	100.0	100.0	100.0	99.8	98
10	2.00	99.6	81	51	98.8	100.0	100.0	100.0	99.9	99.9	99.2	96
20	0.85	99.6	80	40	97.8	99.9	99.4	99.3	99.8	99.8	98.8	95
40	0.425	94.8	71	26	70.2	98.8	69.2	32.5	71.4	99.7	98.5	81
60	0.250	22.2	48	14	31.0	66.8	9.5	4.3	8.0	98.7	97.2	54
100	0.150	3.3	4	4	4.7	3.1	1.9	0.9	1.0	46.8	41.9	13
140	0.106	2.6	1	3	1.6	1.2	1.8	0.7	0.7	8.6	11.1	3
200	0.075	**2.5**	**1**	**2**	**1.1**	**1.1**	**1.7**	**0.6**	**0.7**	**2.1**	**2.7**	**1**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC62	VC62	VC62	VC62	VC64	VC64	VC64	VC64	VC64	VC64	VC64
Sample or Section No		4-6	6-8	8-10	10-12	0-2	0-2	2-4	4-6	6-8	8-10	10-12
Depth in ft		6.00	8.00	10.00	12.00	0.70	2.00	4.00	6.00	8.00	10.00	11.50
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100	100.0	100.0	100.0	100.0	100.0	100.0	100	100	100.0	100.0
1 1/2 inch	38.1	100	100.0	100.0	100.0	100.0	100.0	100.0	100	100	100.0	100.0
1 inch	25.4	100	100.0	100.0	100.0	100.0	100.0	100.0	100	100	100.0	100.0
3/4 inch	19.05	100	100.0	100.0	100.0	100.0	100.0	100.0	100	100	100.0	100.0
1/2 inch	12.7	100	100.0	100.0	100.0	100.0	100.0	100.0	100	100	100.0	100.0
3/8 inch	9.525	100	100.0	100.0	100.0	100.0	100.0	100.0	100	100	100.0	100.0
1/4 inch	6.35	99	100.0	100.0	100.0	99.8	100.0	100.0	98	100	100.0	100.0
4	4.75	99	100.0	100.0	100.0	99.8	100.0	100.0	97	99	99.8	100.0
10	2.00	98	100.0	99.5	99.1	98.6	99.9	99.8	89	91	98.9	99.9
20	0.85	93	99.9	98.3	94.4	97.9	99.7	99.3	54	72	93.8	95.8
40	0.425	42	83.8	73.3	29.7	96.9	99.5	95.4	19	25	54.5	50.4
60	0.250	5	22.0	18.9	8.1	95.5	99.2	84.8	5	4	10.7	9.0
100	0.150	2	5.3	5.2	1.4	78.8	94.2	61.2	3	2	3.9	3.2
140	0.106	1	3.1	2.6	1.0	26.5	34.2	20.5	1	1	2.8	2.5
200	0.075	**1**	**2.1**	**2.2**	**0.9**	**6.2**	**4.6**	**3.7**	**1**	**1**	**2.4**	**2.3**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC66	VC66	VC66	VC66	VC66	VC66	VC66	VC67	VC67	VC67	VC67
Sample or Section No		0-2	0-2	2-4	4-6	6-8	8-10	10-12	0-2	0-2	2-4	4-6
Depth in ft		0.70	2.00	4.00	6.00	8.00	10.00	12.00	0.70	2.00	4.00	6.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1 1/2 inch	38.1	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1 inch	25.4	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
3/4 inch	19.05	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1/2 inch	12.7	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	98.5
3/8 inch	9.525	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	98.5
1/4 inch	6.35	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	98.5
4	4.75	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	99.9	100.0	98.2
10	2.00	100.0	99.3	99.1	99.2	99.8	99.8	99.9	99.9	99.5	100.0	97.7
20	0.85	99.7	81.9	83.0	80.7	86.6	94.7	98.4	99.8	98.9	99.8	97.4
40	0.425	99.0	37.6	35.9	37.8	45.4	59.6	77.5	99.4	98.5	99.4	97.0
60	0.250	98.0	6.8	4.8	8.9	8.4	14.8	22.2	98.0	97.0	98.1	95.1
100	0.150	86.4	1.7	1.1	1.9	1.4	2.5	1.9	81.1	70.3	69.2	69.9
140	0.106	32.3	0.9	0.8	1.0	1.0	1.3	1.2	31.4	19.7	20.0	20.2
200	0.075	**5.0**	**0.7**	**0.7**	**0.8**	**0.9**	**1.0**	**1.0**	**5.0**	**2.1**	**3.7**	**4.2**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC67	VC67	VC67	VC68	VC68	VC68	VC68	VC68	VC68	VC68	VC93
Sample or Section No		6-8	8-10	10-12	0-2	0-2	2-4	4-6	6-8	8-10	10-12	0-2
Depth in ft		7.80	10.00	12.00	0.70	2.00	4.00	6.00	8.00	10.00	12.00	0.08
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100.0	100.0	100	100.0	100.0	100.0	100	100.0	100.0	100.0
1 1/2 inch	38.1	100.0	100.0	100.0	100	100.0	100.0	100.0	100	100.0	100.0	100.0
1 inch	25.4	100.0	100.0	100.0	100	100.0	100.0	100.0	99	100.0	100.0	100.0
3/4 inch	19.05	100.0	100.0	100.0	100	100.0	100.0	100.0	98	100.0	100.0	100.0
1/2 inch	12.7	100.0	100.0	100.0	99	100.0	100.0	100.0	96	100.0	100.0	100.0
3/8 inch	9.525	100.0	100.0	100.0	95	100.0	100.0	100.0	95	100.0	100.0	100.0
1/4 inch	6.35	100.0	100.0	99.0	93	99.9	99.6	100.0	92	100.0	99.5	99.6
4	4.75	100.0	99.9	99.0	92	99.7	99.6	100.0	90	100.0	99.2	99.6
10	2.00	99.9	99.0	98.3	90	99.0	98.8	99.9	81	99.7	98.4	99.4
20	0.85	99.6	97.5	97.6	88	98.0	98.4	99.6	74	99.3	97.4	99.3
40	0.425	98.8	76.0	96.7	87	96.8	98.2	97.9	70	98.8	96.6	95.5
60	0.250	95.0	19.0	95.3	81	93.2	96.2	61.0	59	98.1	95.8	59.5
100	0.150	80.9	6.5	75.4	44	53.6	55.6	31.7	17	90.5	92.6	4.8
140	0.106	31.6	3.3	19.6	12	12.2	14.2	10.9	6	30.2	37.9	1.4
200	0.075	**7.7**	**1.9**	**5.3**	**3**	**2.6**	**2.8**	**2.8**	**3**	**8.8**	**8.9**	**1.0**

Summary of Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC93	VC93	VC93	VC93	VC93	VC93	VC93	VC93
Sample or Section No		0-2	0-2	2-4	4-6	6-8	6-8	8-10	8-10
Depth in ft		1.00	2.00	3.00	5.00	7.00	8.00	9.00	10.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**	**% Passing**
2 inch	50.8	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1 1/2 inch	38.1	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1 inch	25.4	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
3/4 inch	19.05	100.0	100.0	97.6	100.0	100.0	100.0	100.0	100.0
1/2 inch	12.7	100.0	100.0	97.6	100.0	100.0	100.0	100.0	100.0
3/8 inch	9.525	100.0	100.0	96.3	100.0	100.0	100.0	100.0	100.0
1/4 inch	6.35	100.0	100.0	95.1	100.0	100.0	100.0	100.0	100.0
4	4.75	100.0	100.0	94.3	100.0	100.0	100.0	100.0	100.0
10	2.00	99.7	99.1	92.0	100.0	100.0	100.0	100.0	100.0
20	0.85	97.8	96.7	89.3	100.0	99.8	99.9	100.0	100.0
40	0.425	76.8	85.0	83.9	99.9	99.7	99.9	99.9	98.0
60	0.250	34.3	46.8	49.4	99.8	99.5	99.5	97.9	78.9
100	0.150	4.0	5.1	13.5	87.5	82.2	61.4	33.7	19.3
140	0.106	1.6	3.0	7.7	25.4	15.2	14.0	12.5	7.9
200	0.075	**1.3**	**2.5**	**4.9**	**7.7**	**4.1**	**7.1**	**5.6**	**4.7**

Summary Of Hydrometer and Sieve Results

Project Number: 17-1052

Alpine - NESE II

Boring or Core Number		VC93
Sample or Section No		2-4
Depth in ft		4.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**
2 inch	50.8	100.0
1 1/2 inch	38.1	100.0
1 inch	25.4	100.0
3/4 inch	19.05	100.0
1/2 inch	12.7	100.0
3/8 inch	9.525	100.0
1/4 inch	6.35	100.0
4	4.75	100.0
10	2.00	99.9
20	0.85	99.8
40	0.425	99.6
60	0.250	99.1
100	0.150	92.3
140	0.106	56.5
200	0.075	**16.6**
Hyd.	0.04981	6.4
Hyd.	0.03562	4.4
Hyd.	0.02533	4.2
Hyd.	0.01791	3.9
Hyd.	0.01308	3.7
Hyd.	0.00925	3.2
Hyd.	0.00654	2.7
Hyd.	0.00450	2.6
Hyd.	0.00329	2.3
Hyd.	0.00233	2.2
Hyd.	0.00134	1.7

% passing 2µ	**2.1**

D_{100} (mm)	4.750
D_{60} (mm)	0.110
D_{30} (mm)	0.085

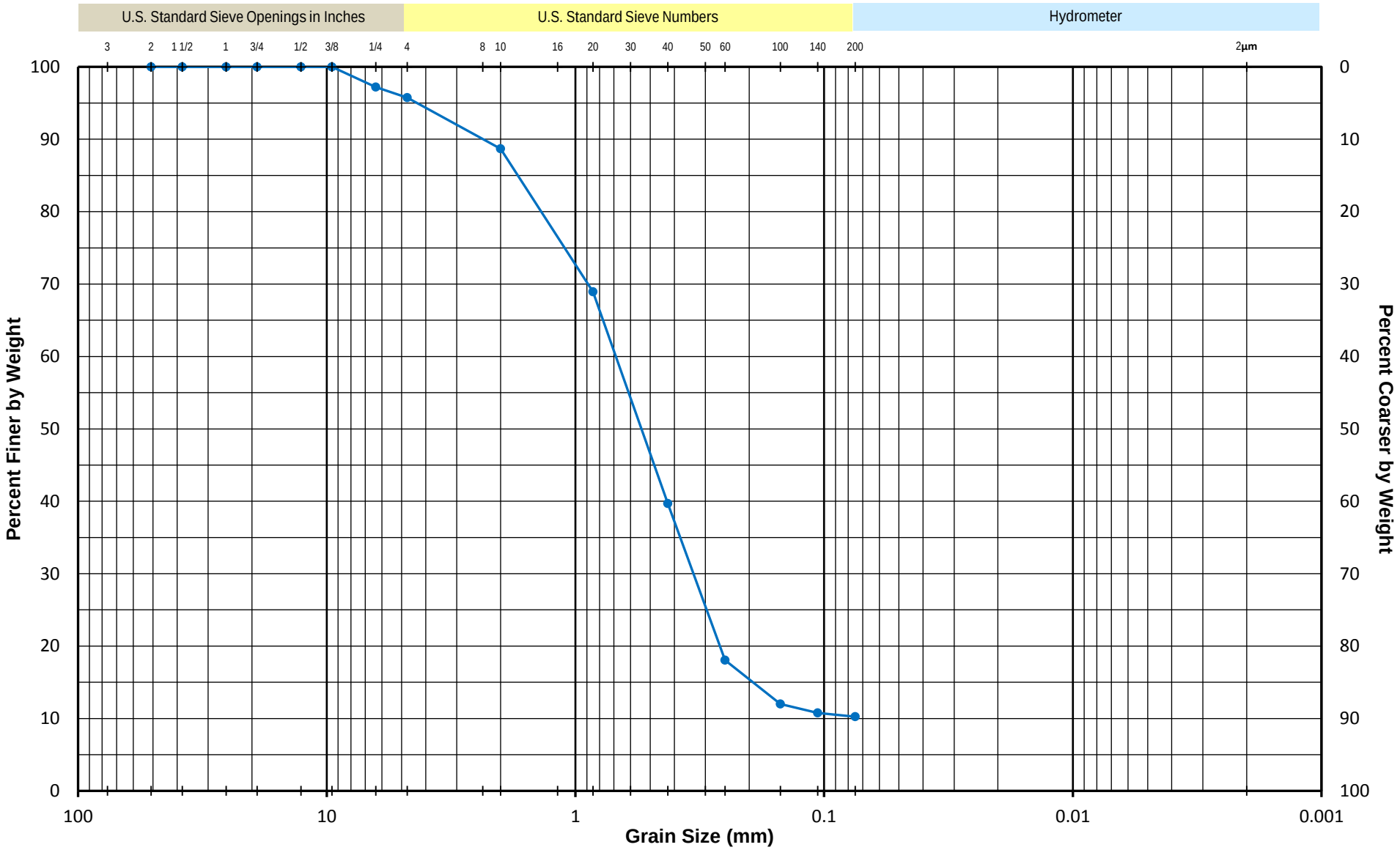
Boring or Core Number		VC93
Sample or Section No		4-6
Depth in ft		6.00
Sieve Size/No.	**Sieve Size (mm)**	**% Passing**
2 inch	50.8	100.0
1 1/2 inch	38.1	100.0
1 inch	25.4	100.0
3/4 inch	19.05	100.0
1/2 inch	12.7	100.0
3/8 inch	9.525	100.0
1/4 inch	6.35	100.0
4	4.75	100.0
10	2.00	100.0
20	0.85	100.0
40	0.425	99.9
60	0.250	99.9
100	0.150	96.8
140	0.106	41.7
200	0.075	**19.3**
Hyd.	0.04687	16.4
Hyd.	0.03336	15.9
Hyd.	0.02374	14.6
Hyd.	0.01689	13.4
Hyd.	0.01241	11.9
Hyd.	0.00888	10.7
Hyd.	0.00628	9.7
Hyd.	0.00440	8.7
Hyd.	0.00318	7.8
Hyd.	0.00226	7.2
Hyd.	0.00131	5.5

% passing 2µ	**6.8**

D_{100} (mm)	0.850
D_{60} (mm)	0.121
D_{30} (mm)	0.090

D_{10} (mm)	0.059
C_c	-
C_u	-

D_{10} (mm)	0.007
C_c	-
C_u	-

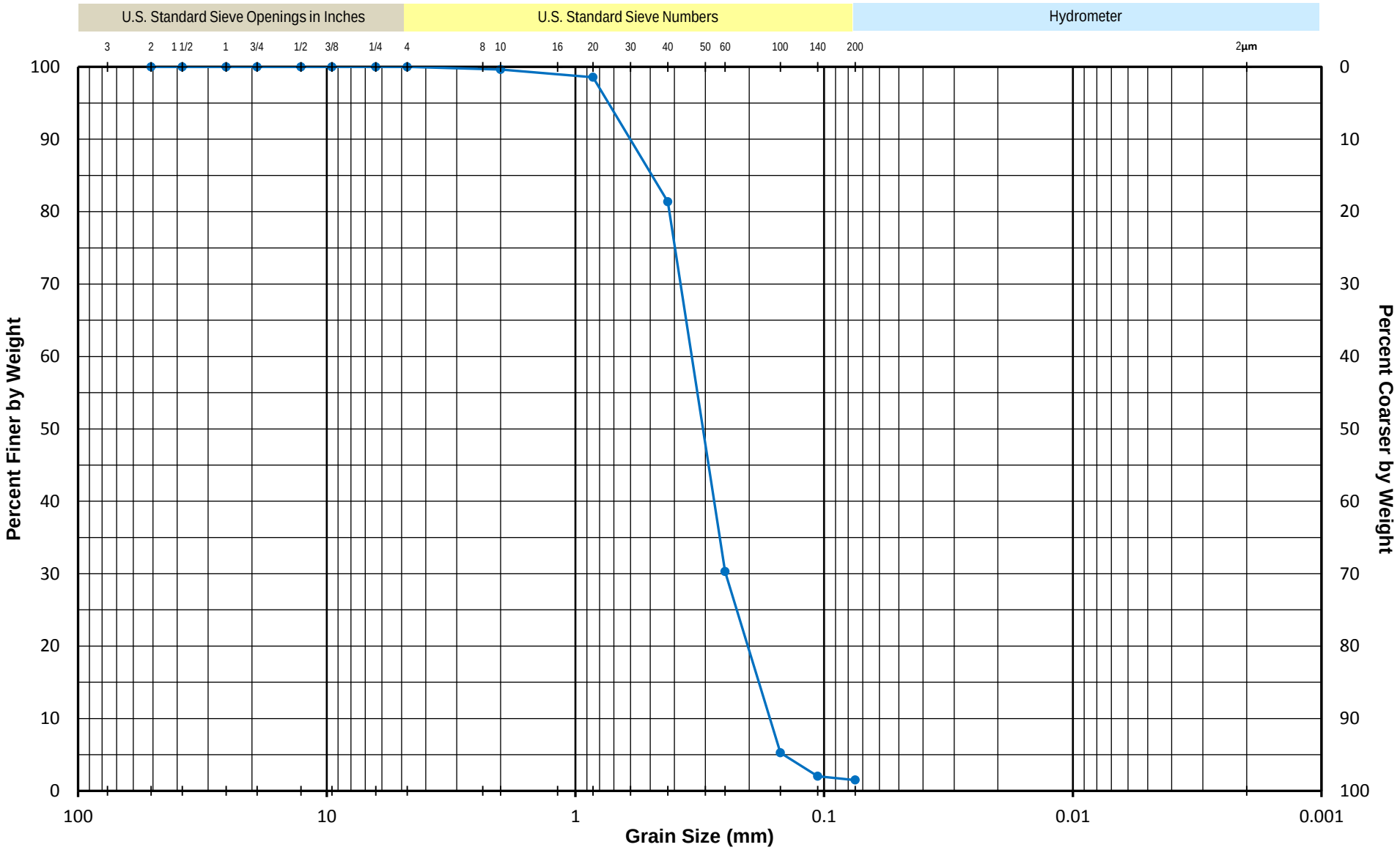


Test Depth	Core ID Section/Sample	Material Description
0.45 ft	VC11 0-2	Very Dark Gray Medium to Fine Sand with silt

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

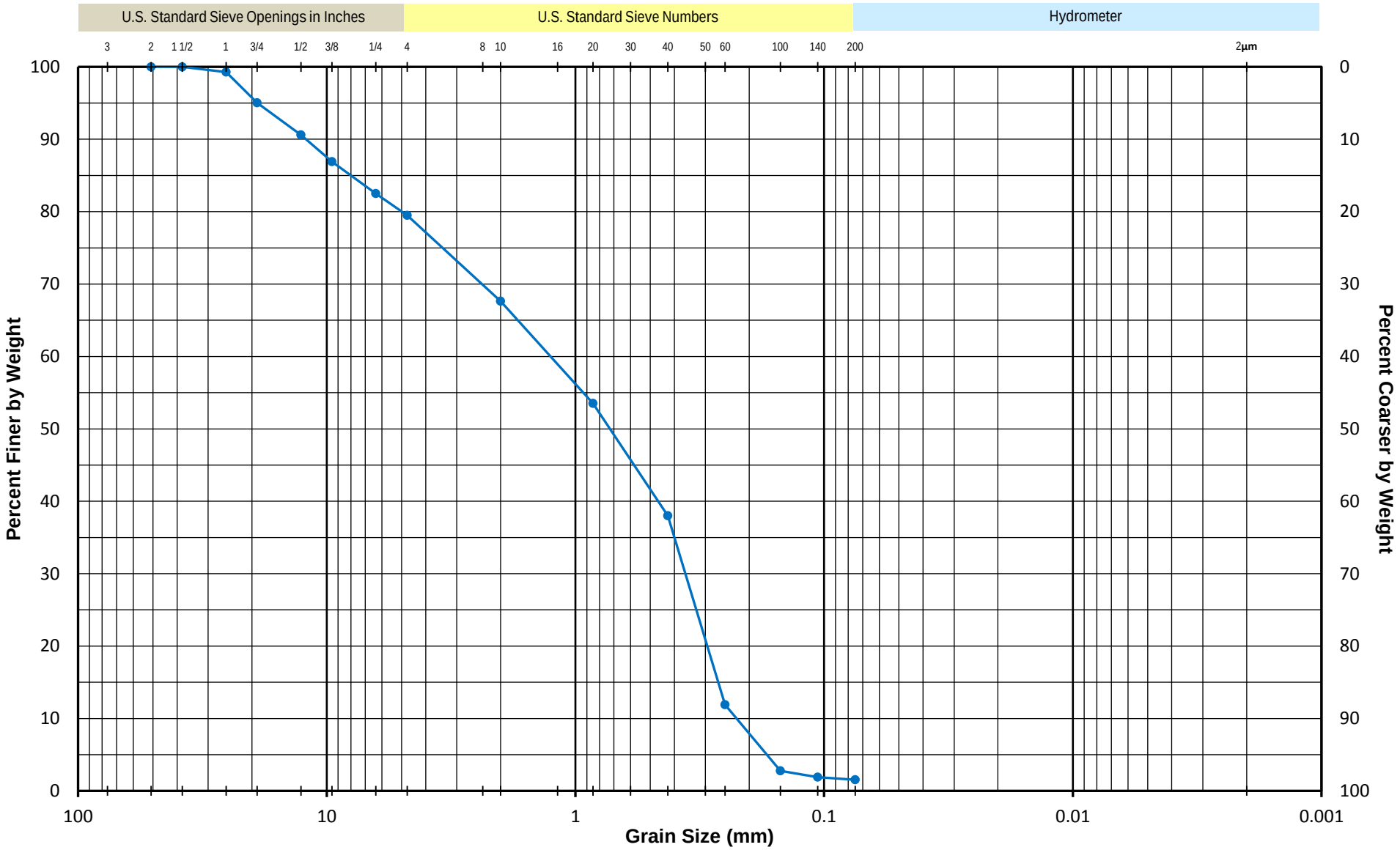


Test Depth	Core ID Section/Sample	Material Description
1.9 ft	VC11 0-2	Dark Brown Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

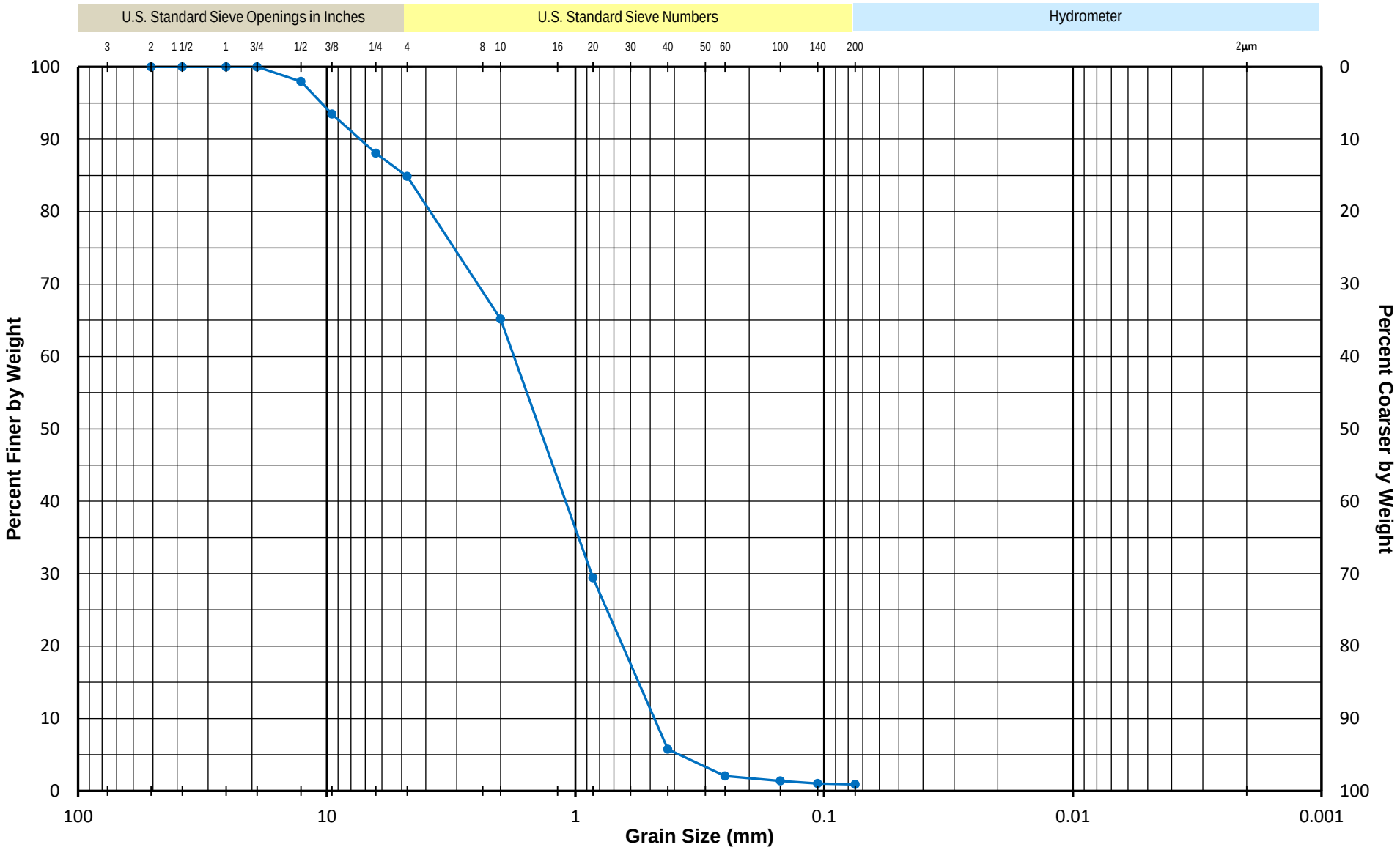


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC11 2-4	Dark Brown Fine to Coarse Sand with gravel

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

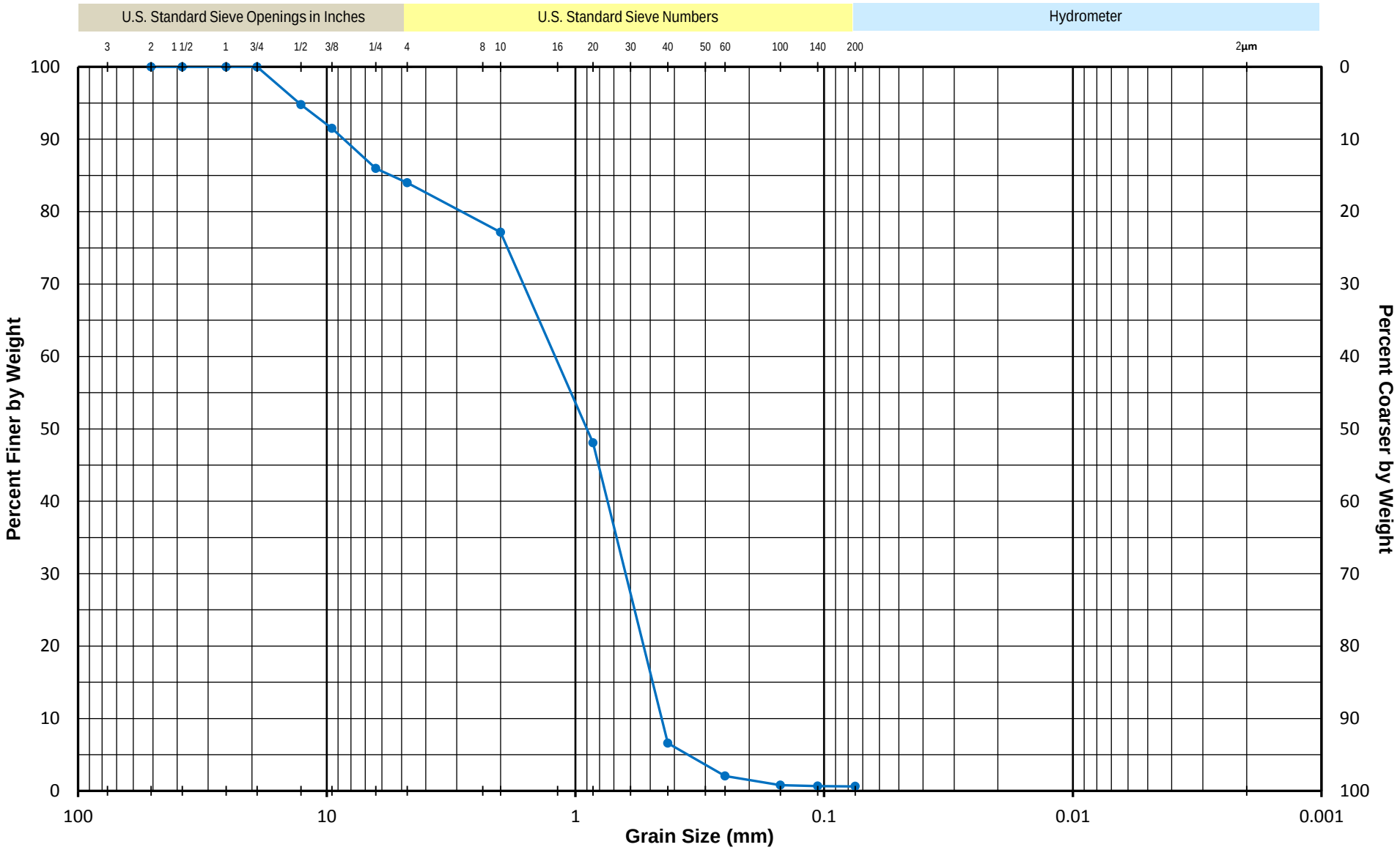


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC11 4-6	Dark Brown Coarse to Medium Sand with gravel

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

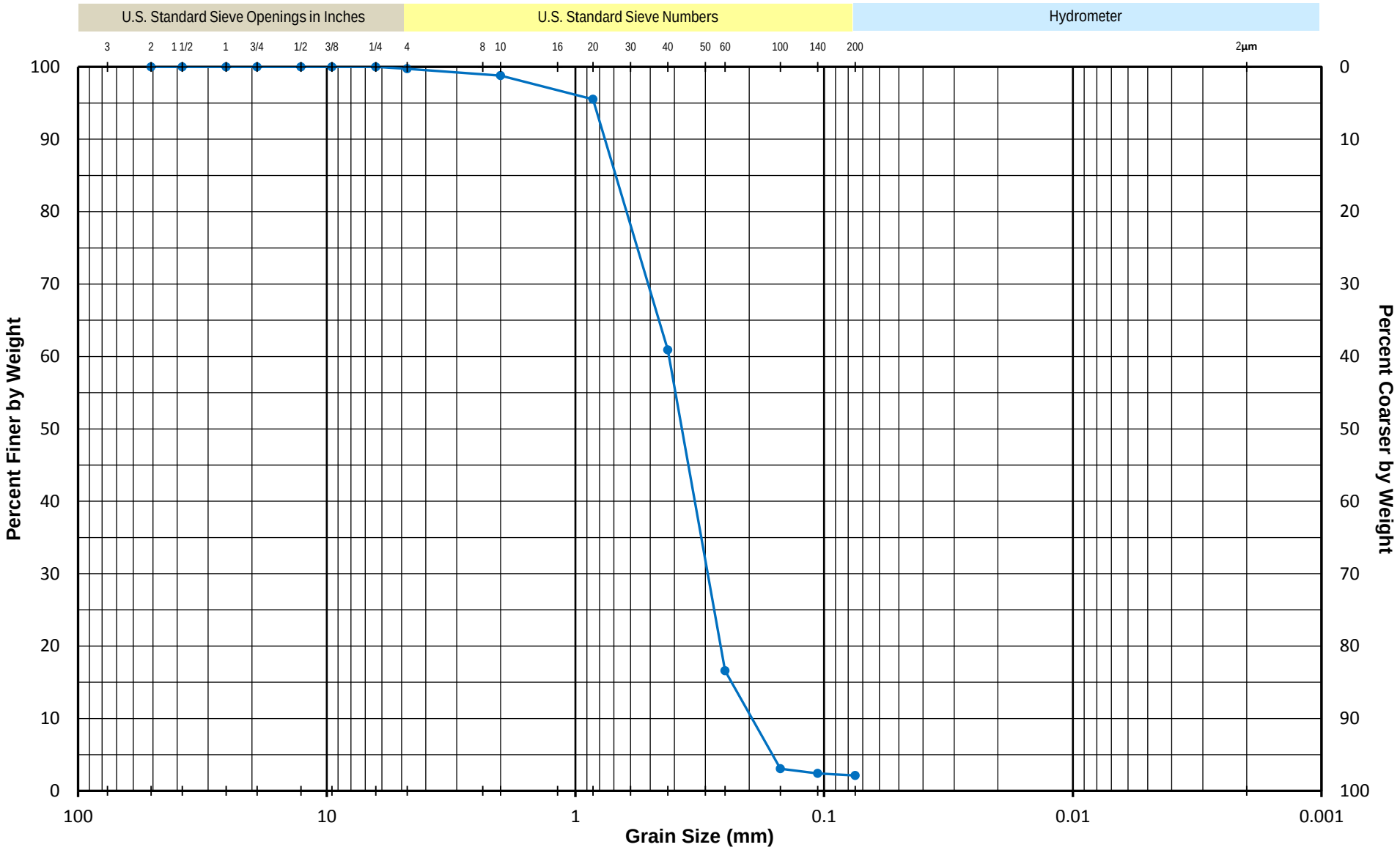


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC11 6-8	Brown Coarse to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

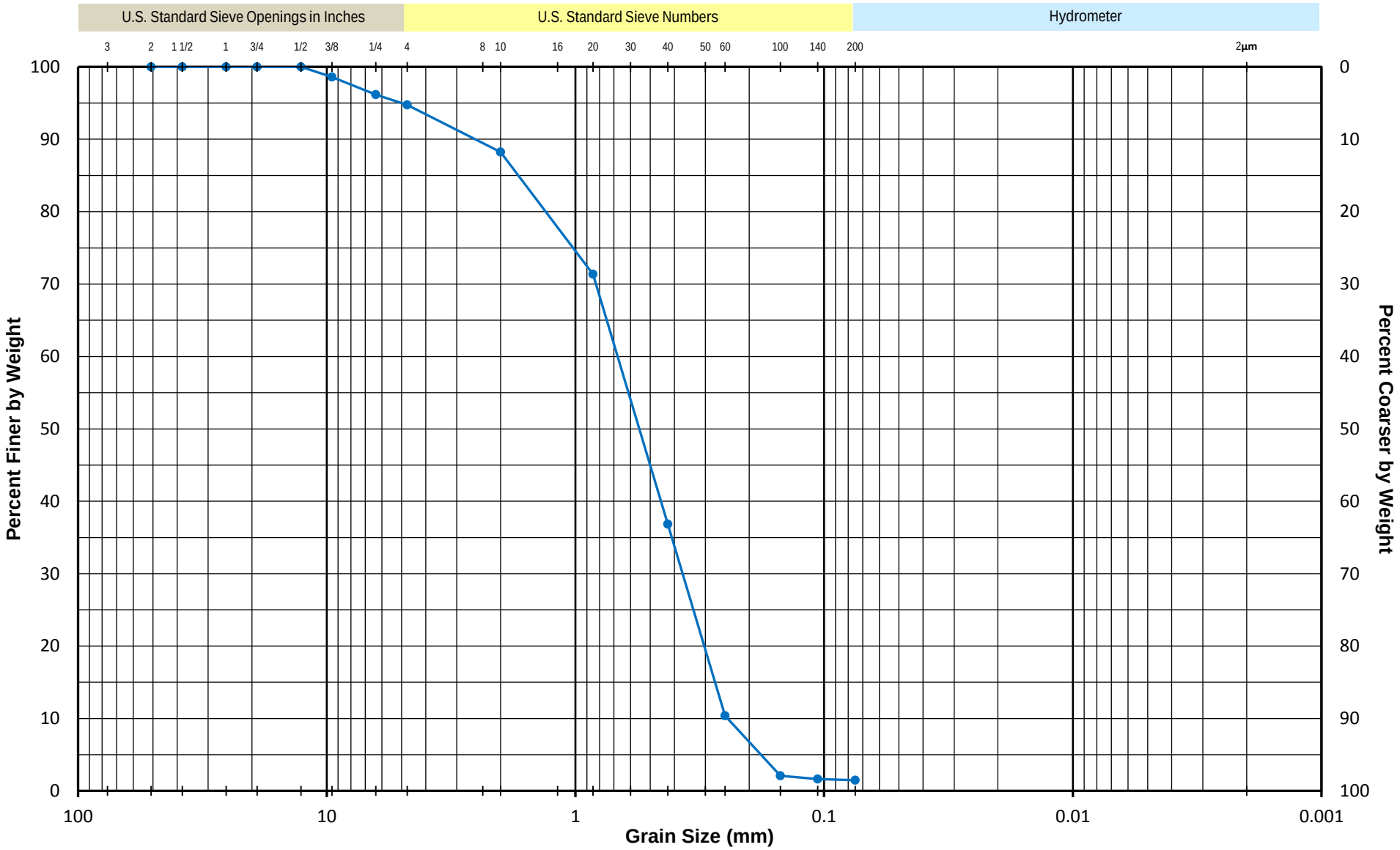


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC11 8-10	Brown Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

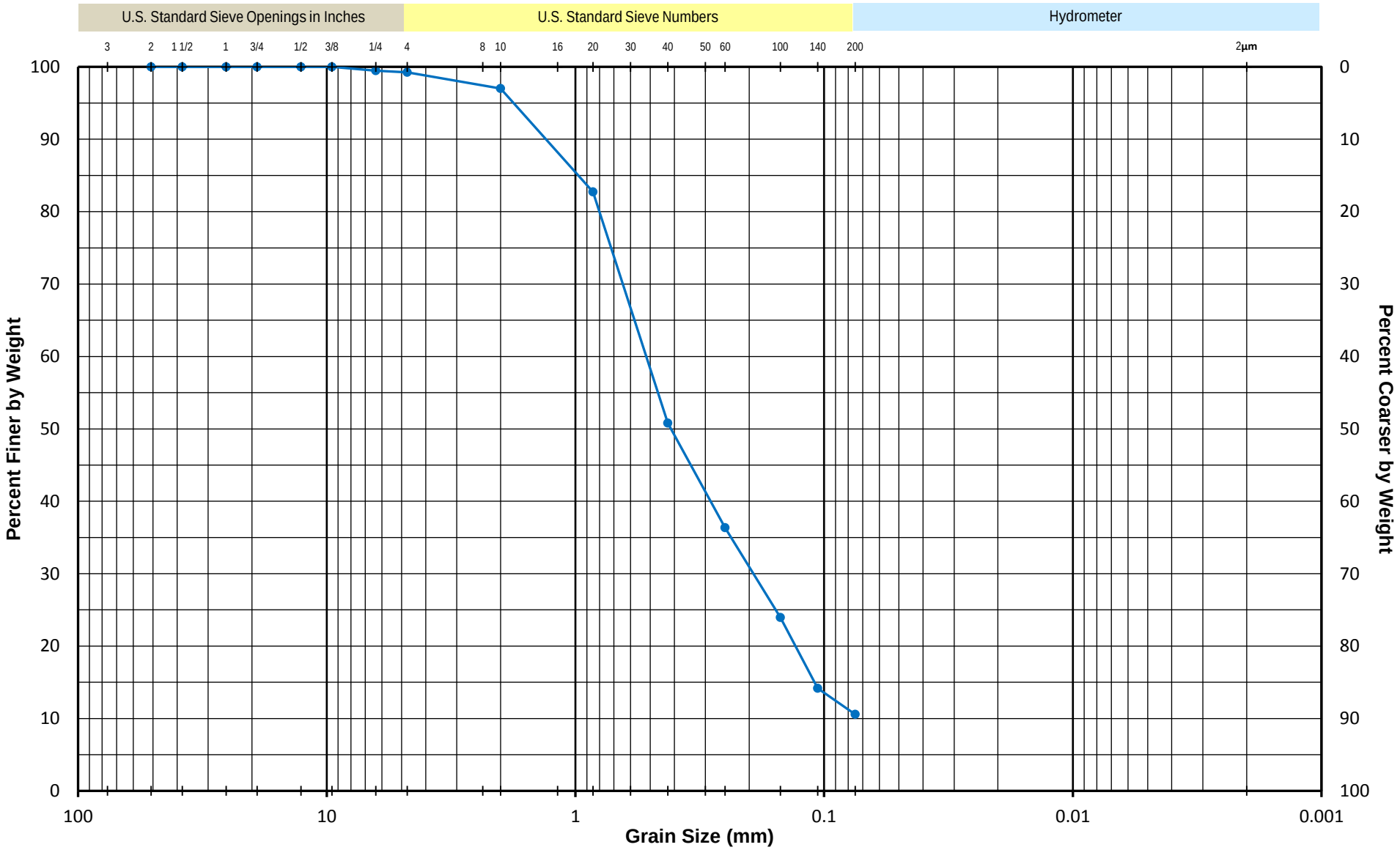


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC11 10-12	Dark Brown Coarse to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

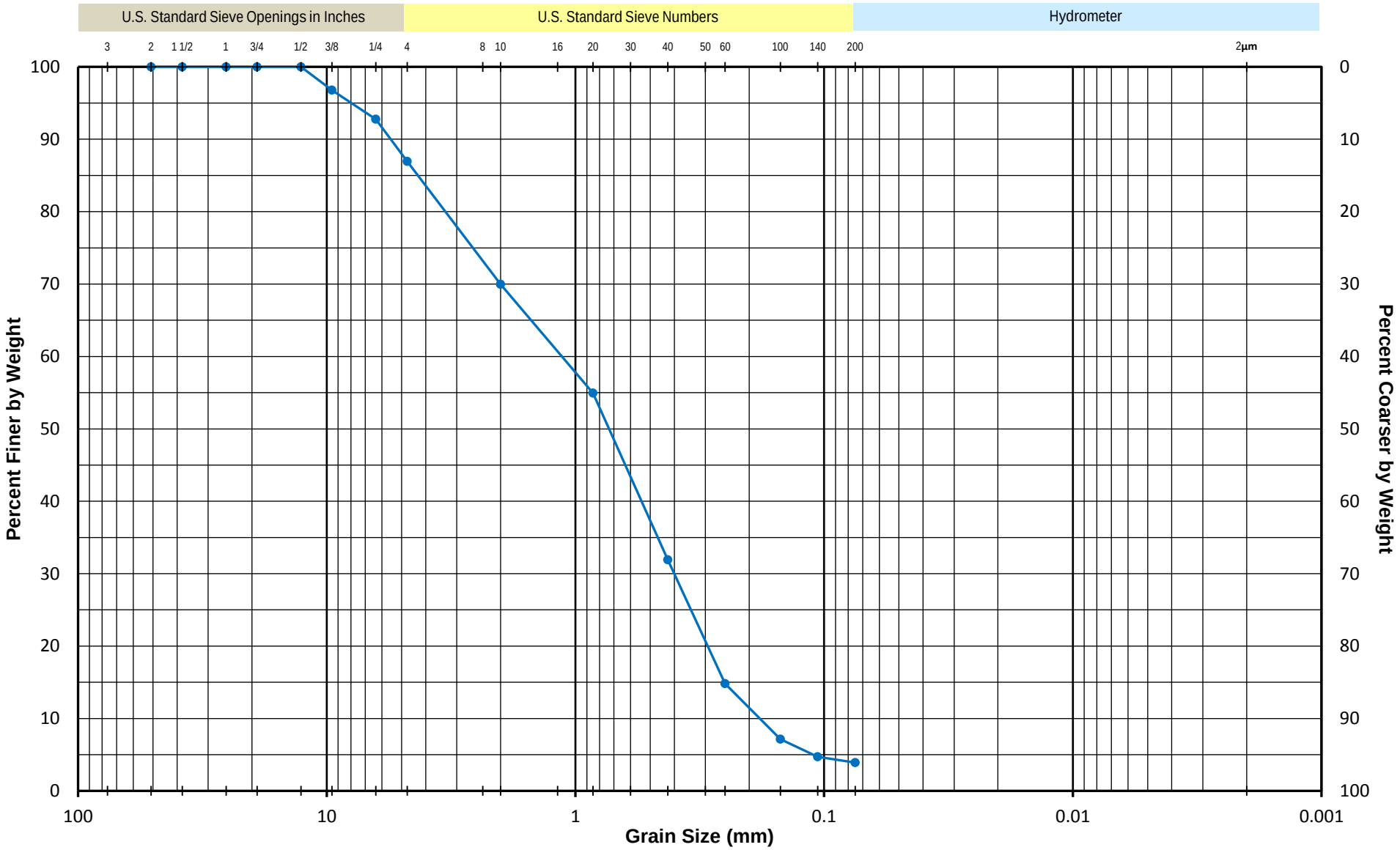


Test Depth	Core ID Section/Sample	Material Description
0.5 ft	VC25 0-2	Very Dark Gray Sand with silt and shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

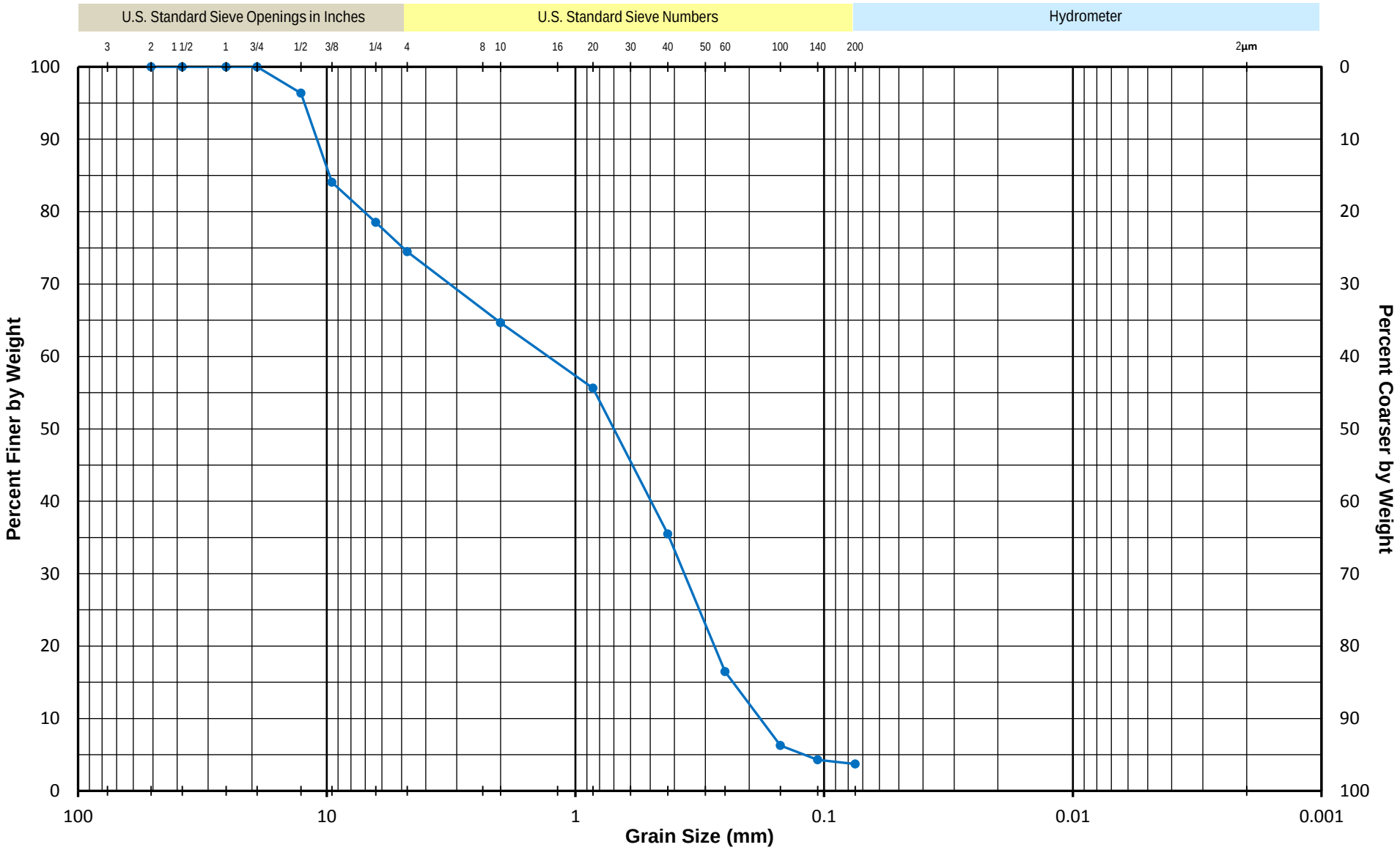


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC25 0-2	Brown Coarse to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

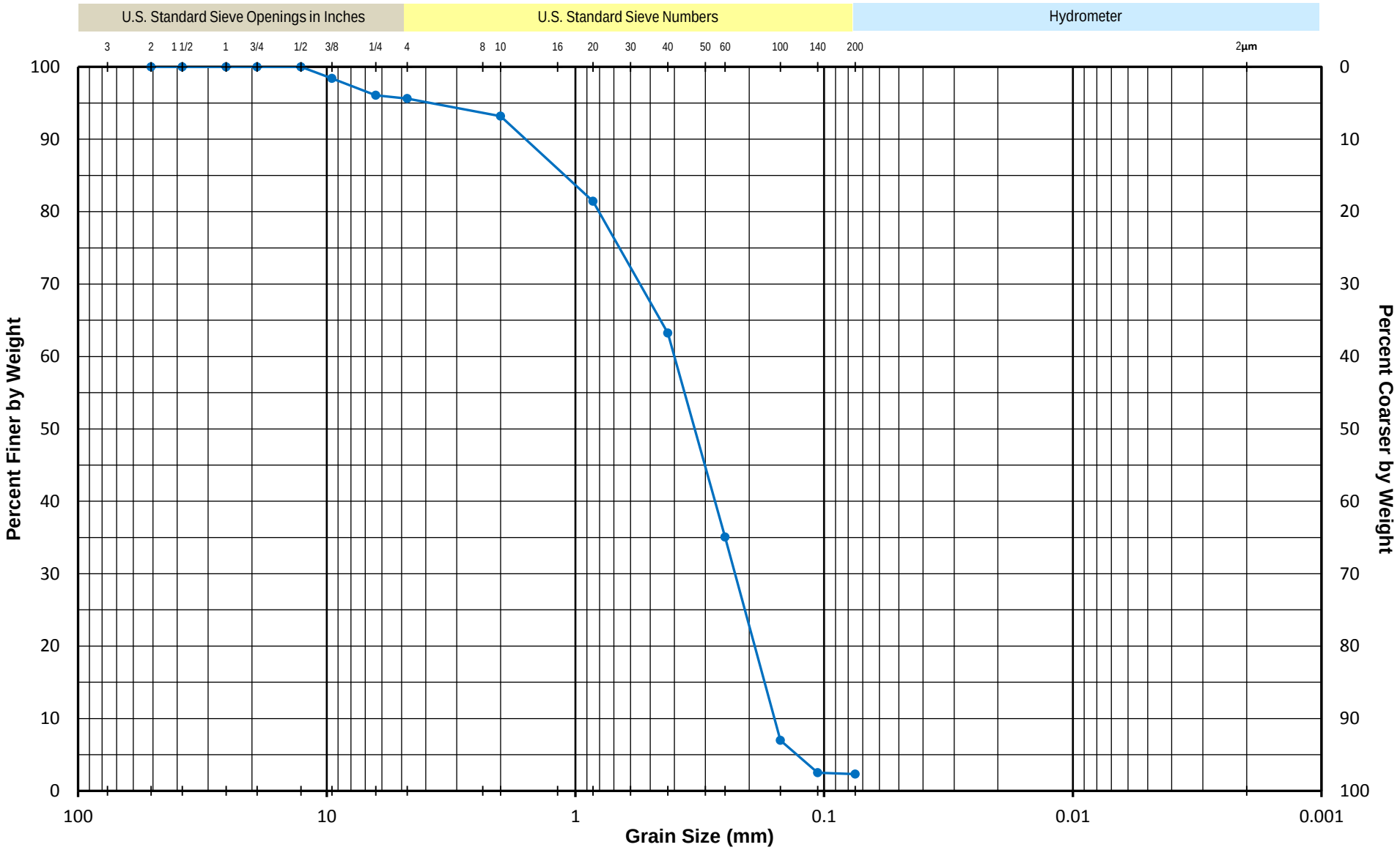


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC25 2-4	Dark Brown Coarse to Medium Sand with gravel

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

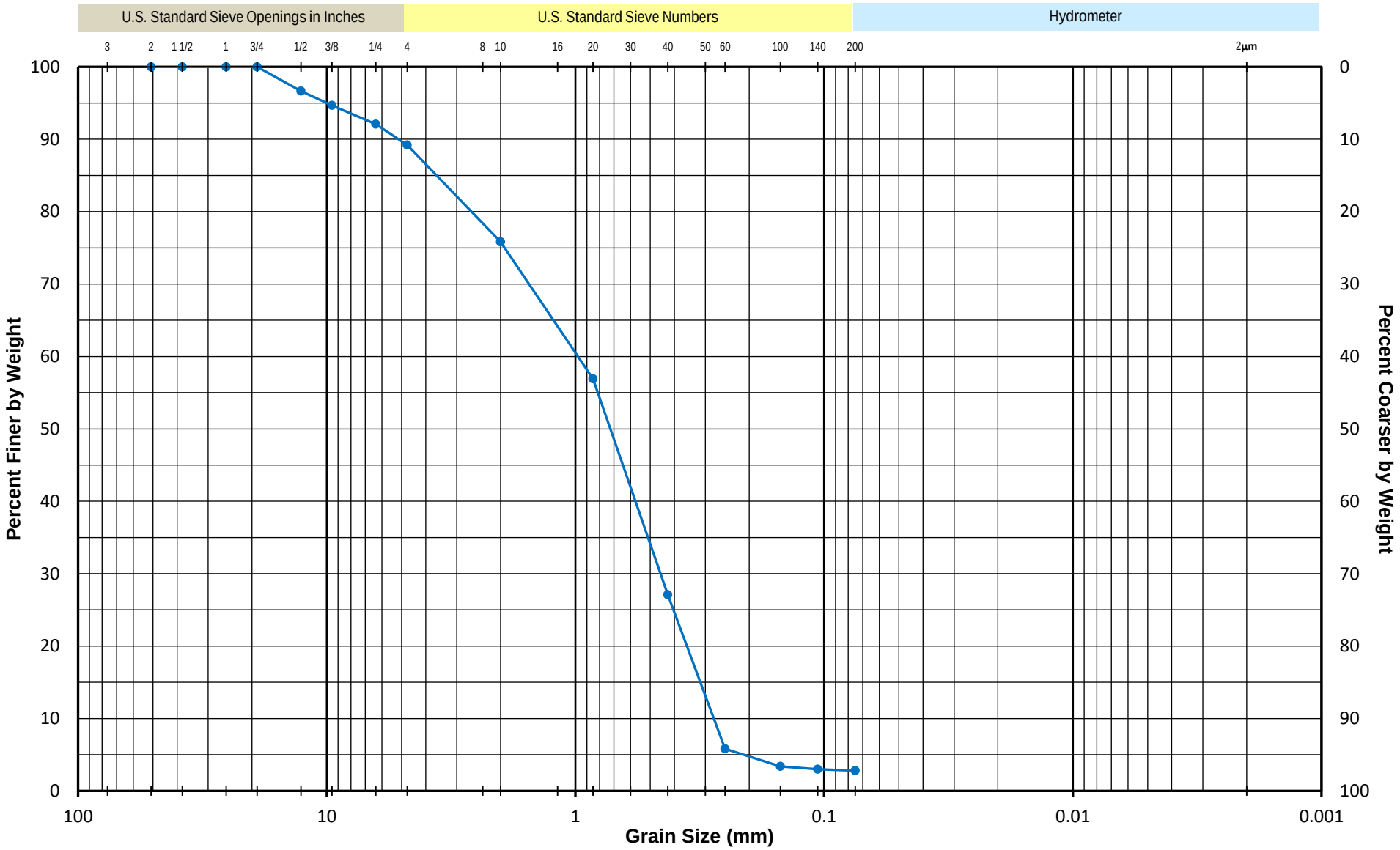


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC25 4-6	Dark Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

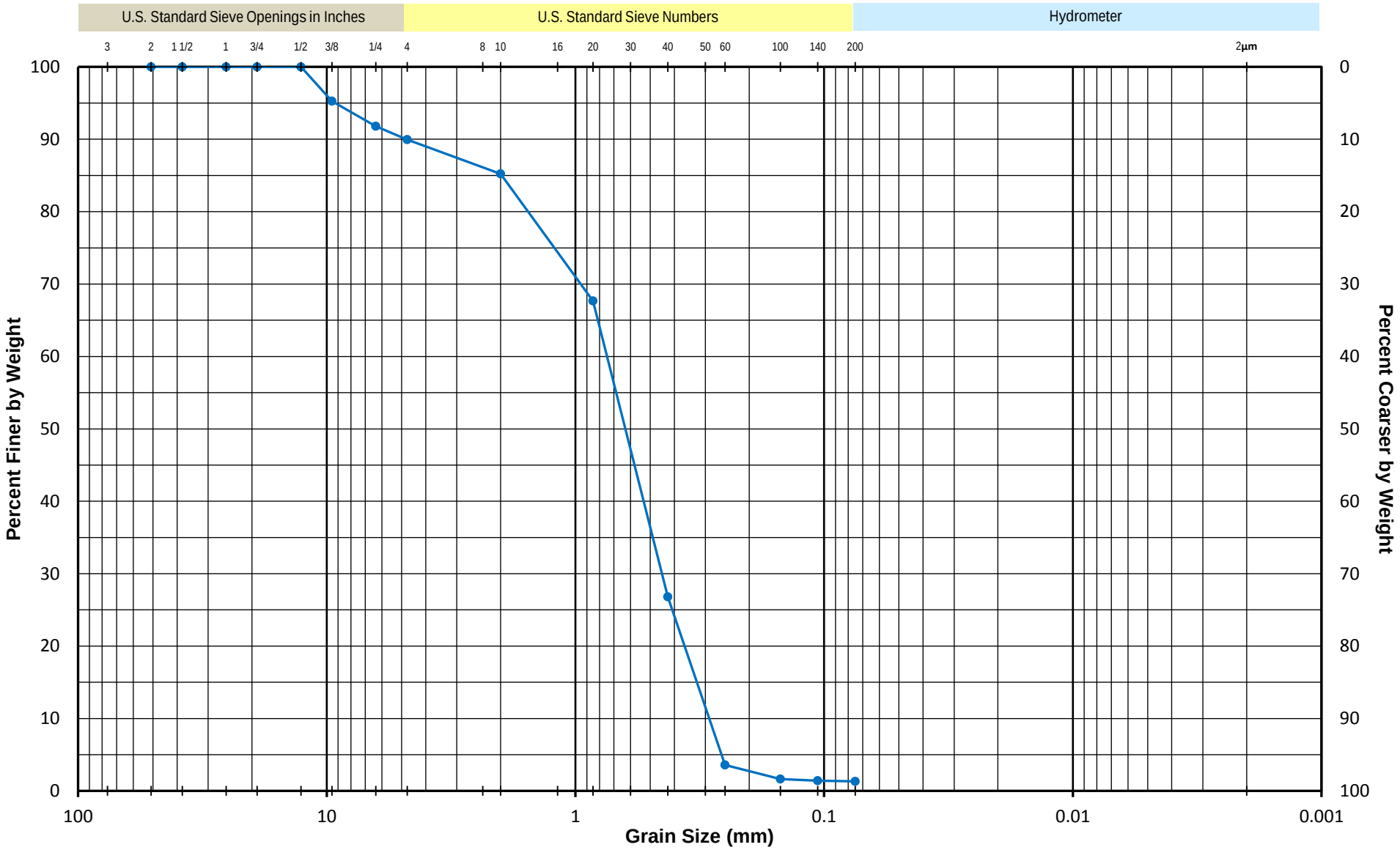


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC25 6-8	Dark Brown Medium to Coarse Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

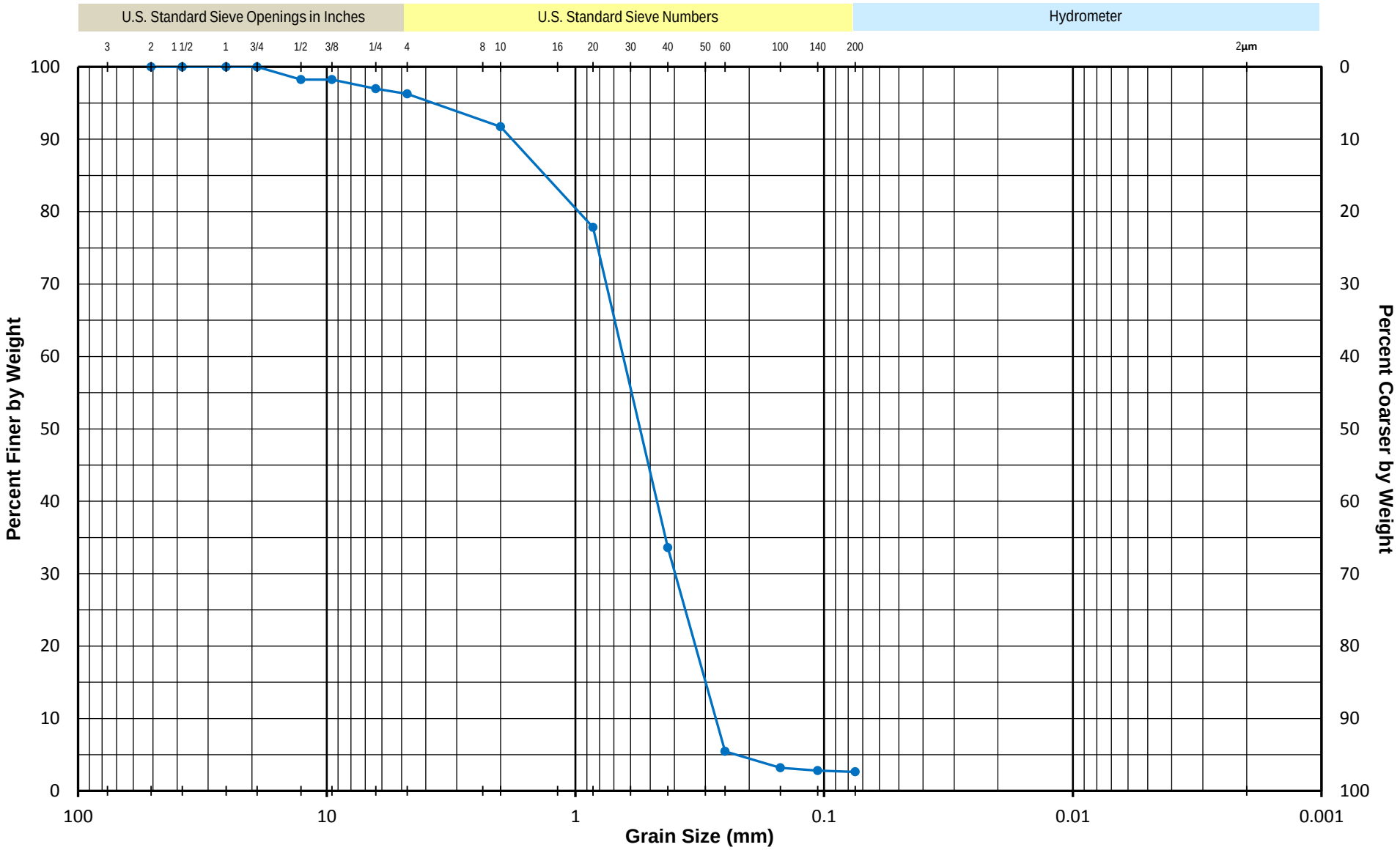


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC25 8-10	Dark Brown Coarse to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

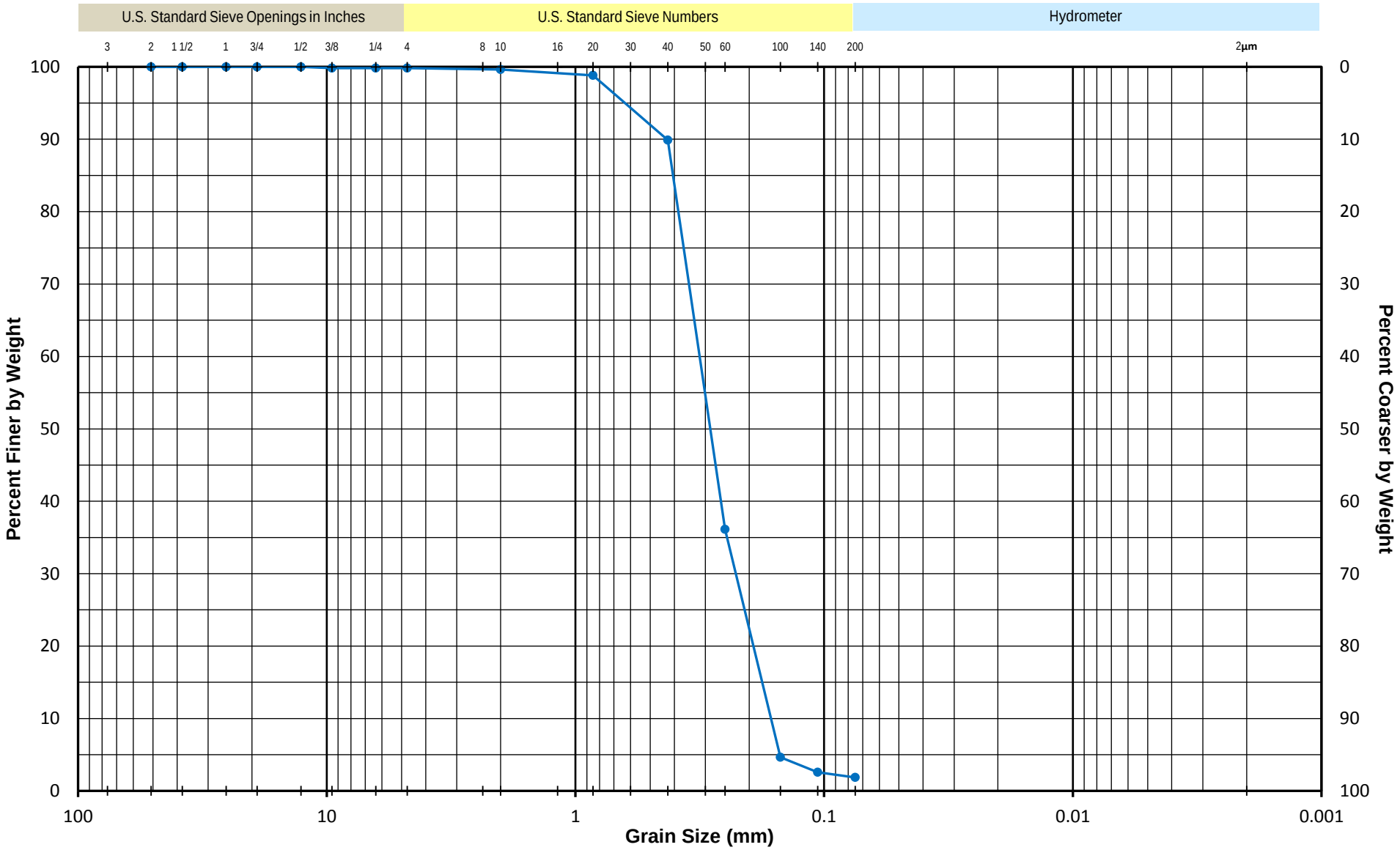


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC25 10-12	Dark Brown Coarse to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

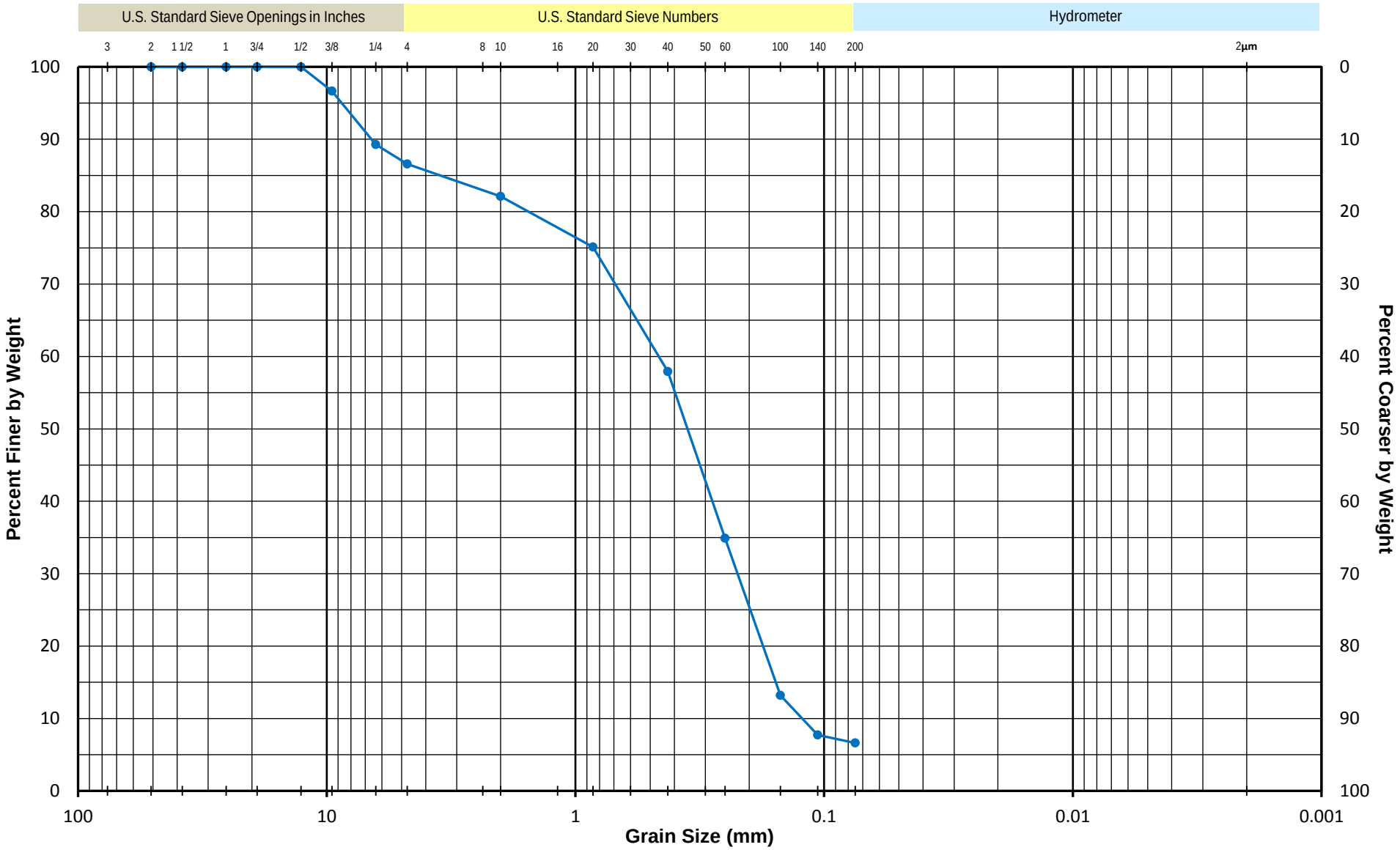


Test Depth	Core ID Section/Sample	Material Description
0.5 ft	VC26 0-2	Very Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

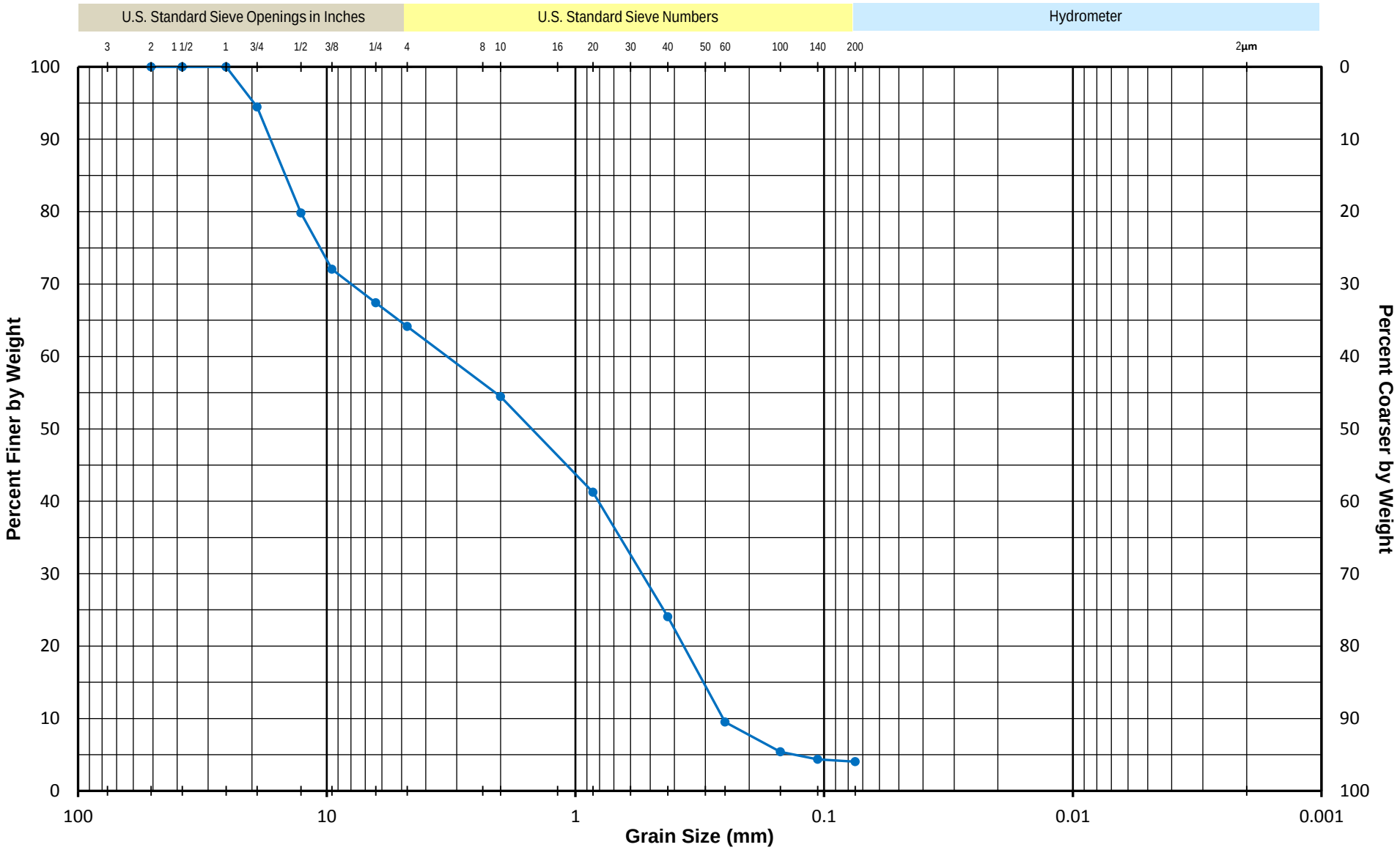


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC26 0-2	Very Dark Gray Coarse to Fine Sand with clay

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

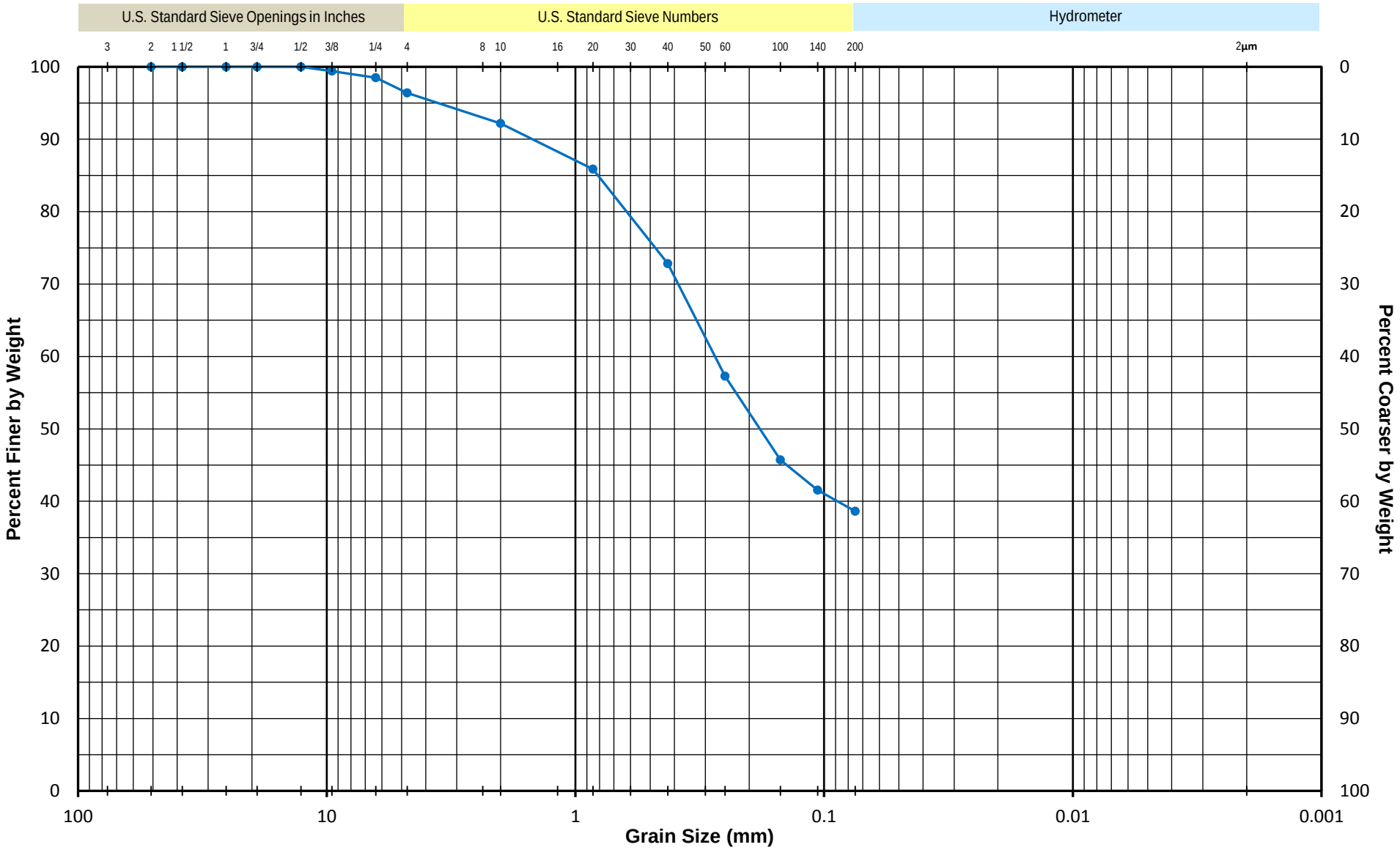


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC26 2-4	Very Dark Gray Fine to Coarse Sand with gravel

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

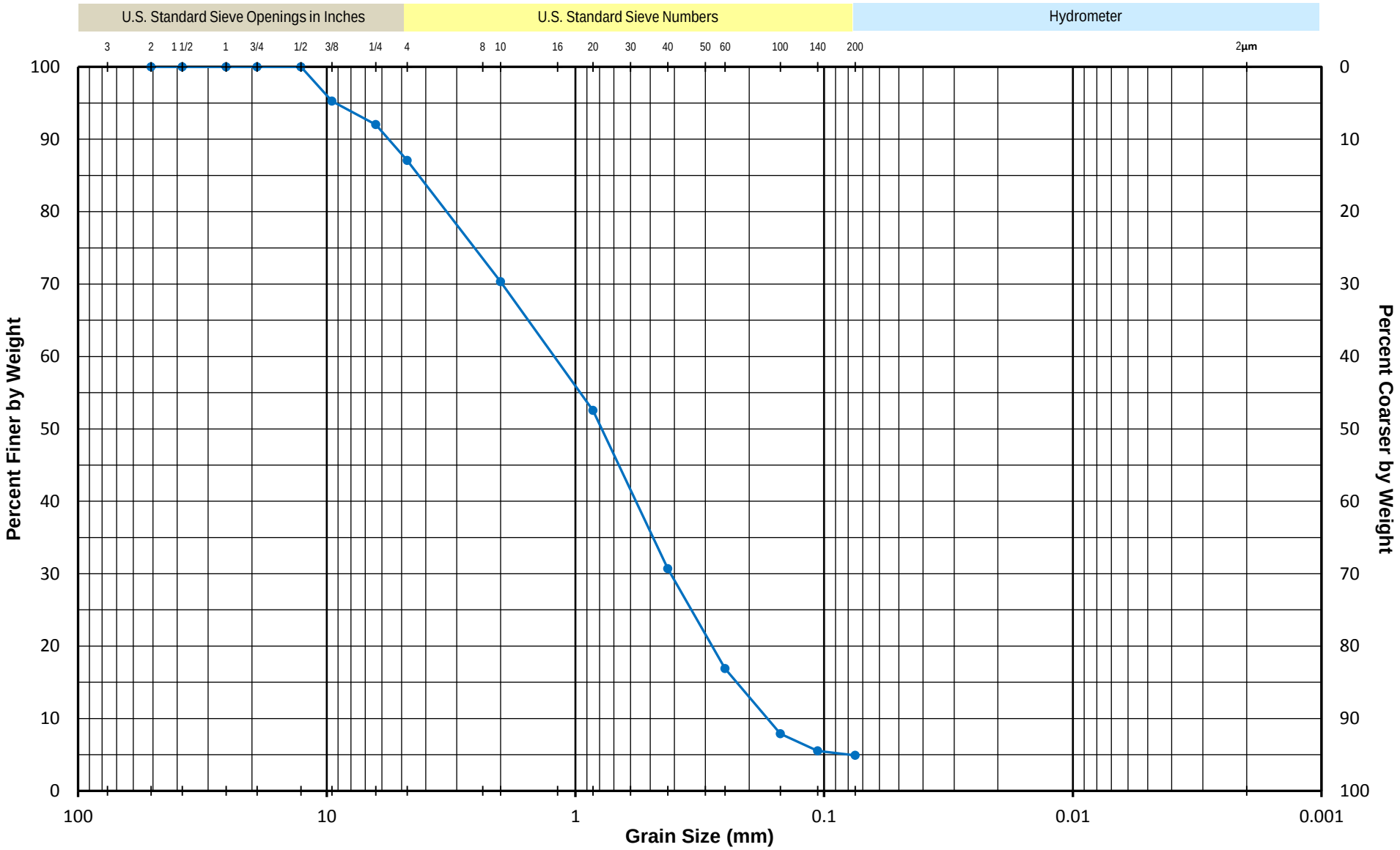


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC26 4-6	Strong Brown Silty Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

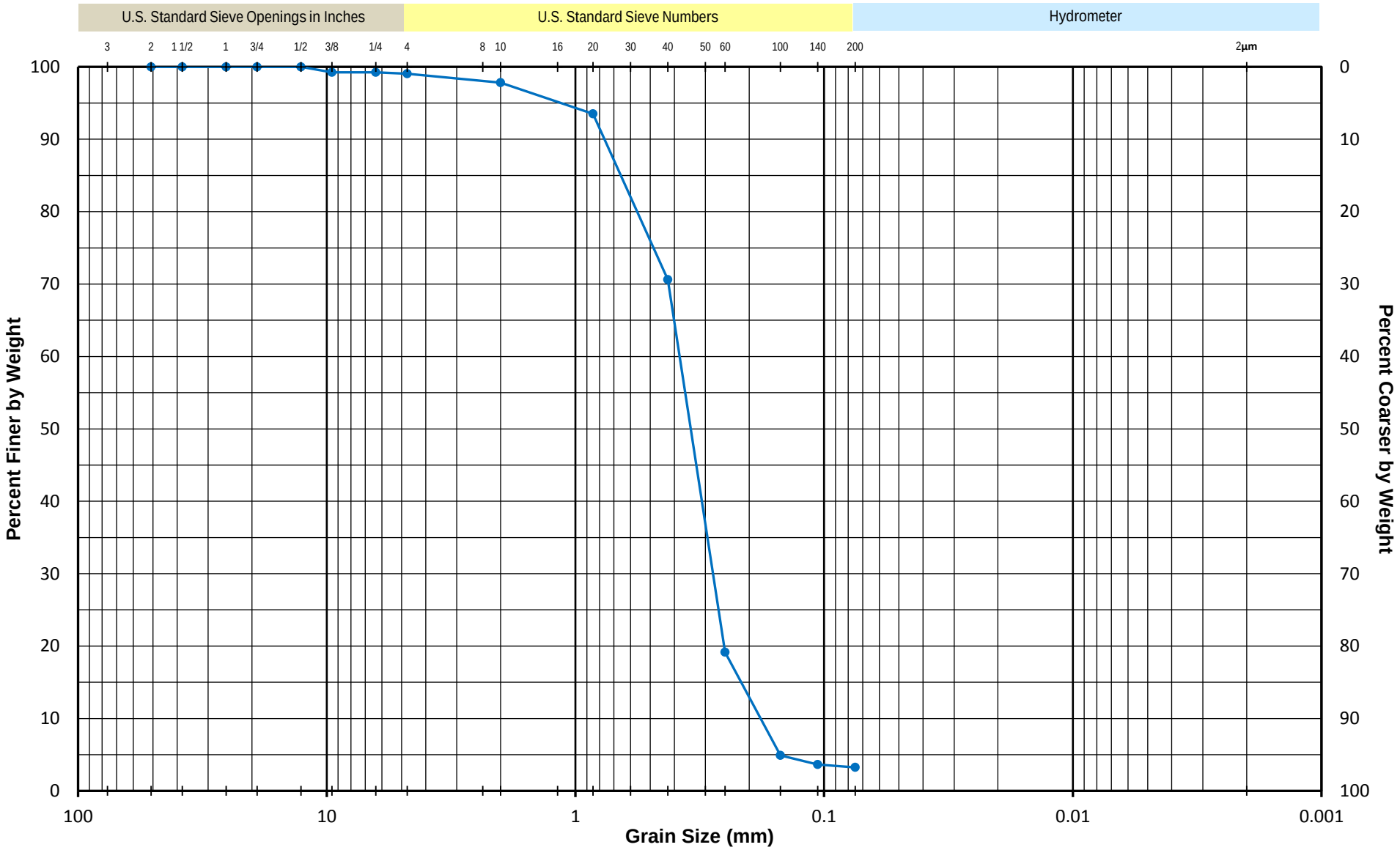


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC26 6-8	Strong Brown Coarse to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

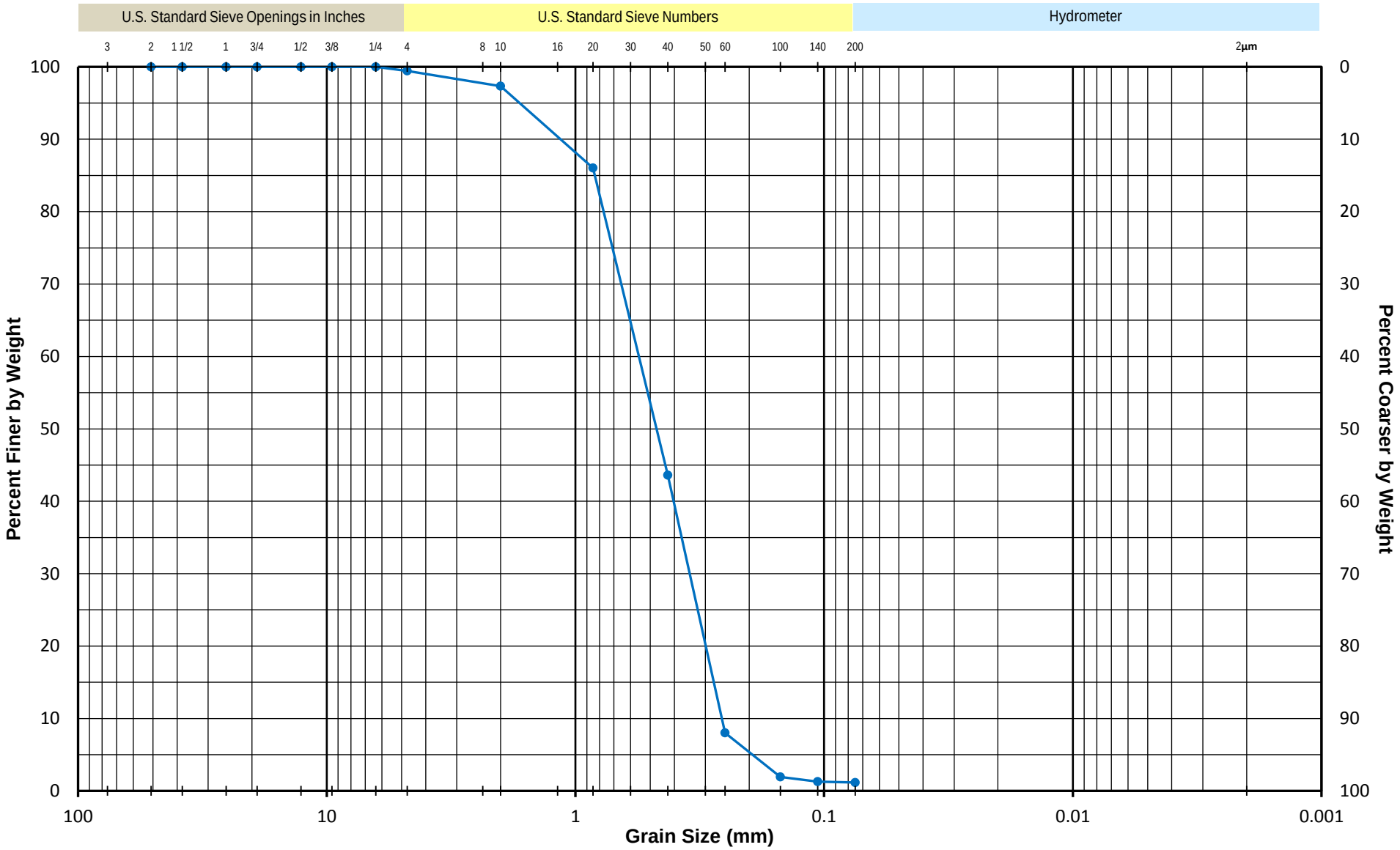


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC26 8-10	Dark Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

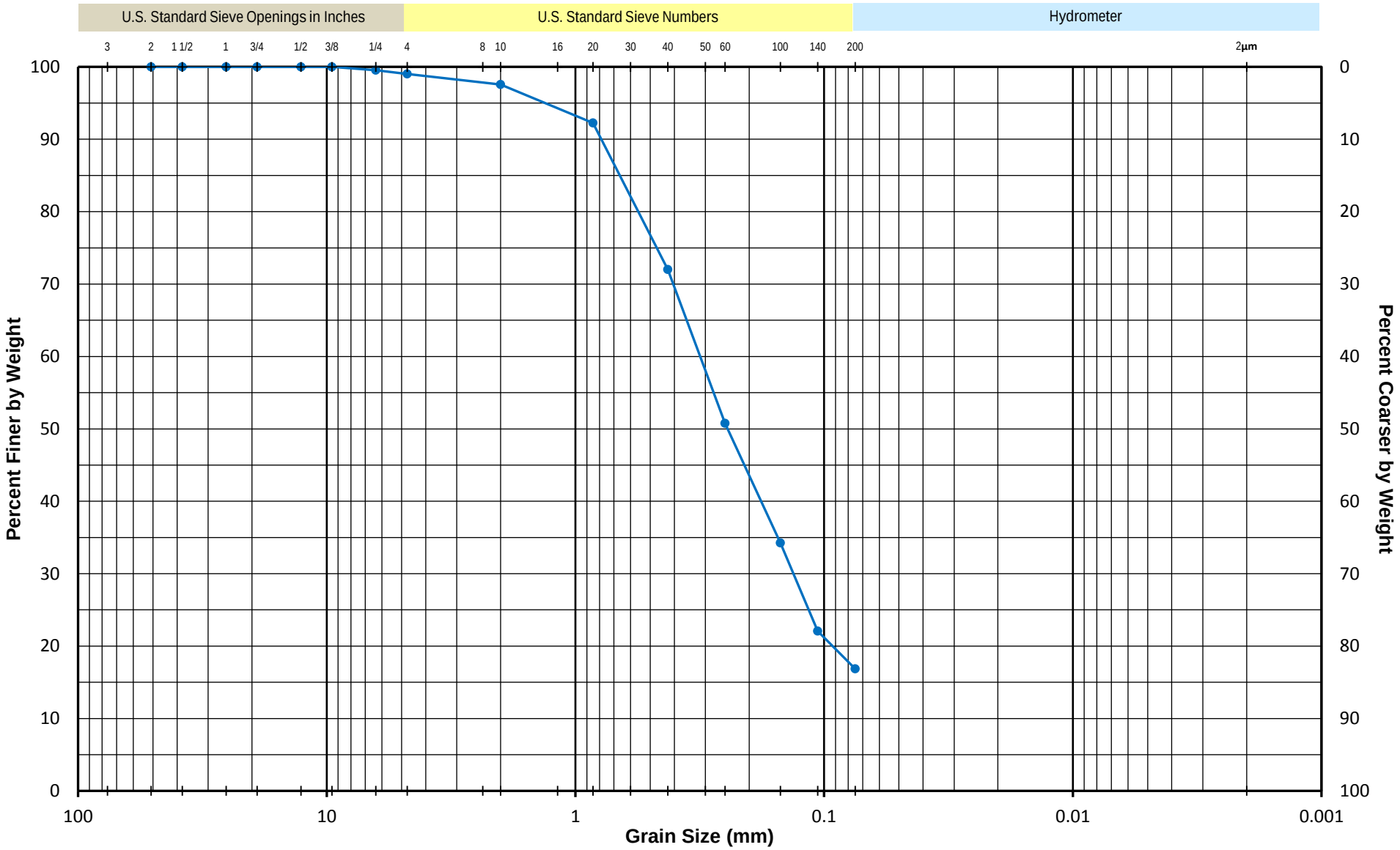


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC26 10-12	Dark Brown Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

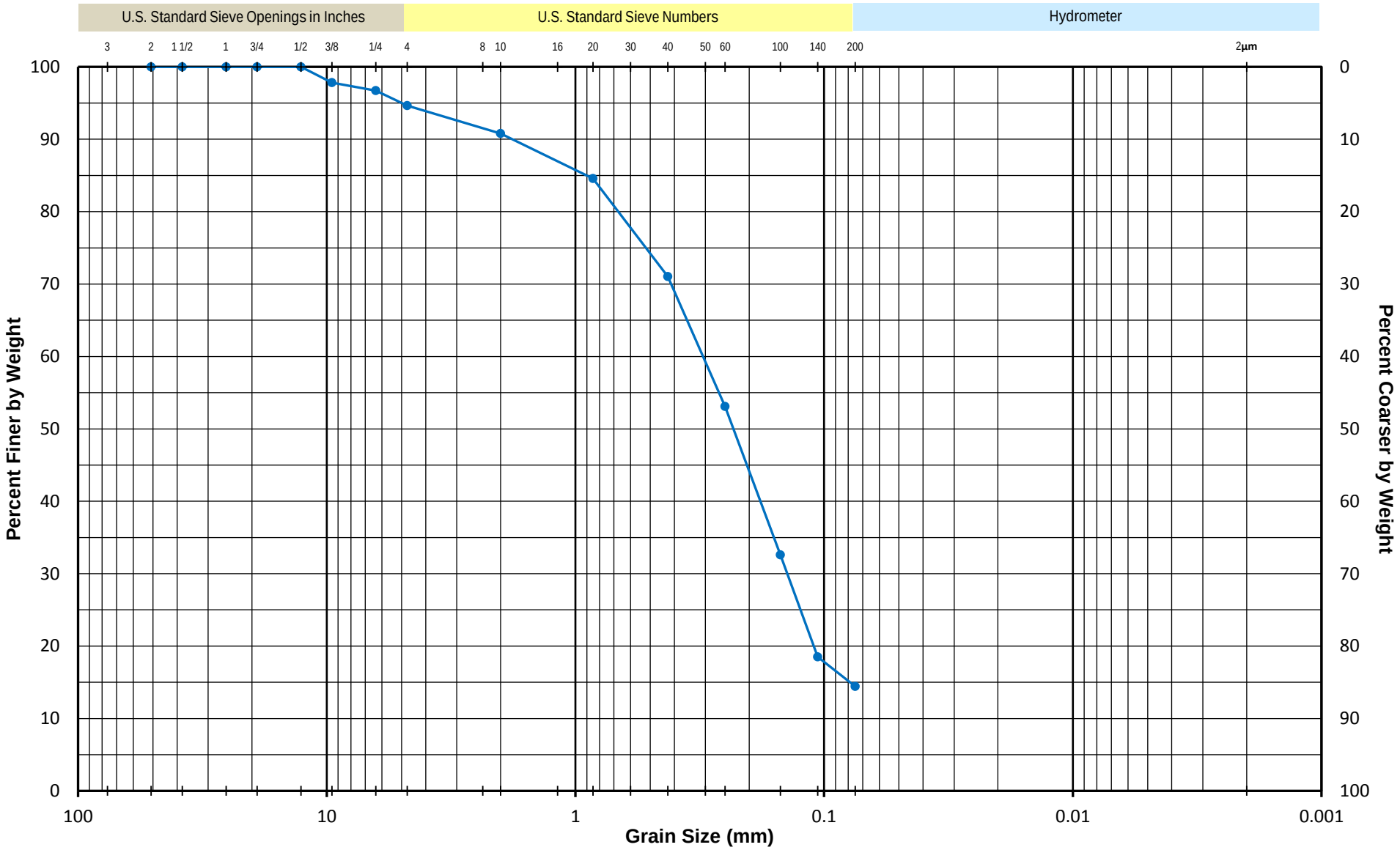


Test Depth	Core ID Section/Sample	Material Description
0.6 ft	VC28 0-2	Black Clayey Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

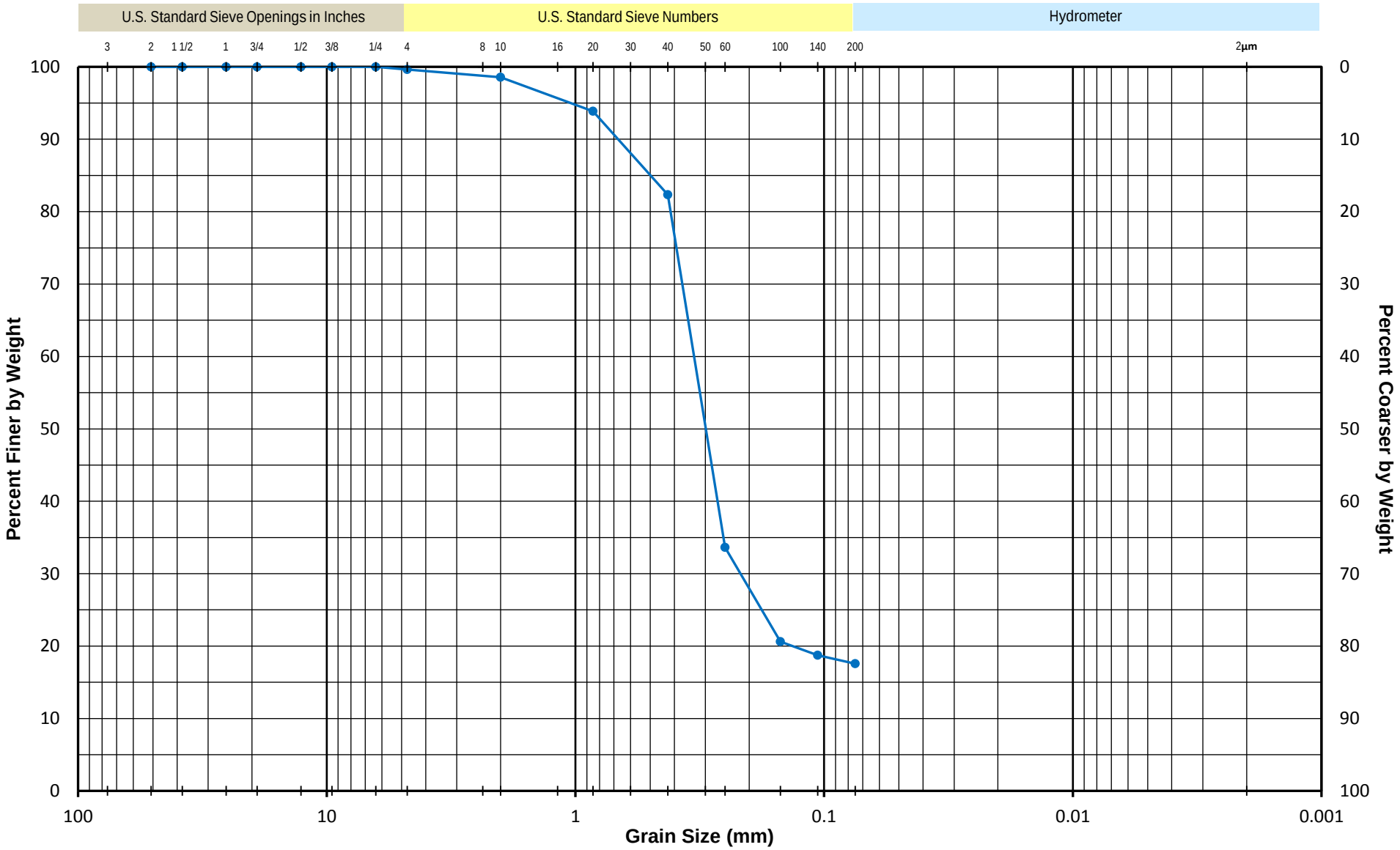


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC28 0-2	Very Dark Gray Clayey Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

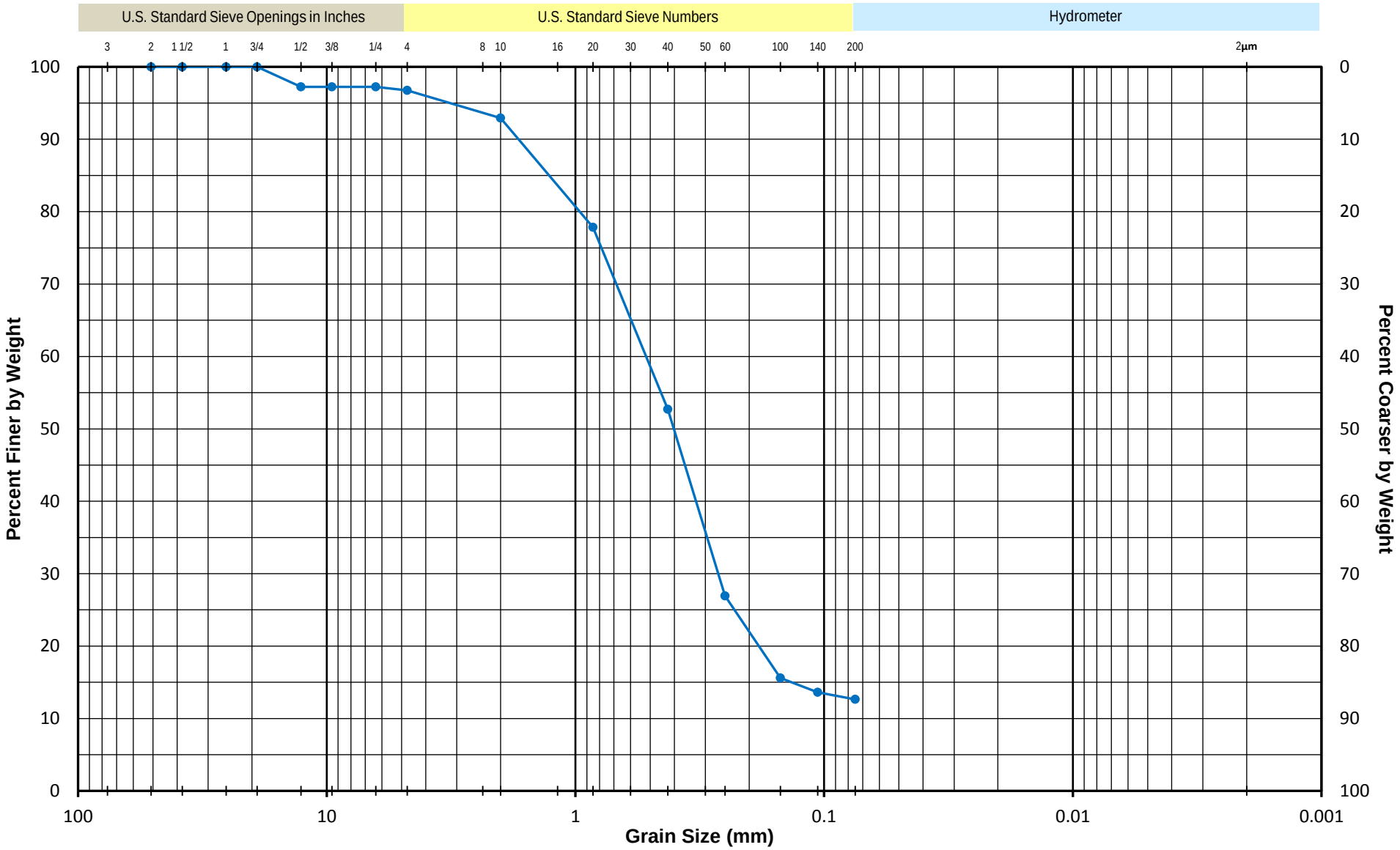


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC28 2-4	Dark Gray Silty Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

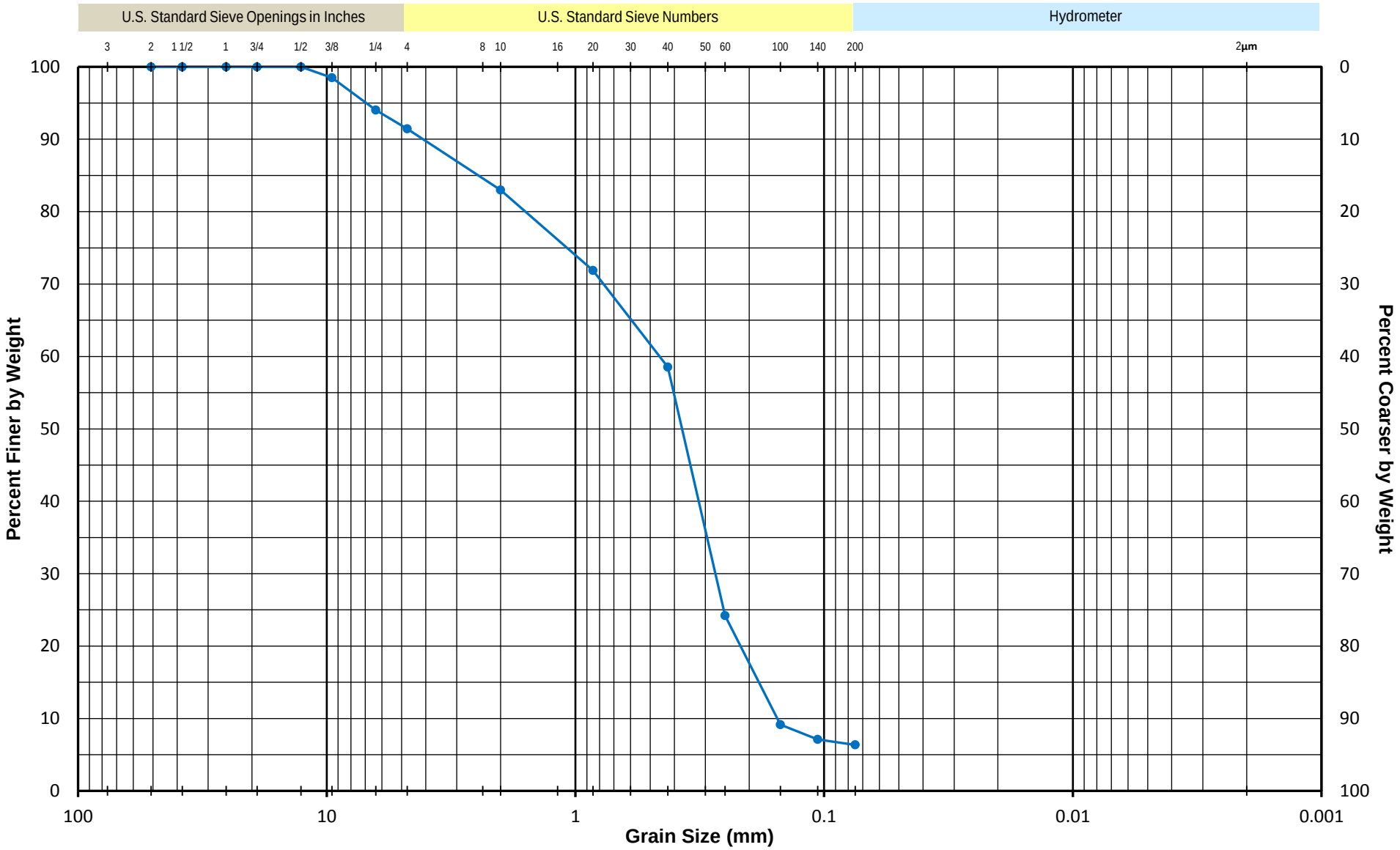


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC28 4-6	Dark Gray Silty Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

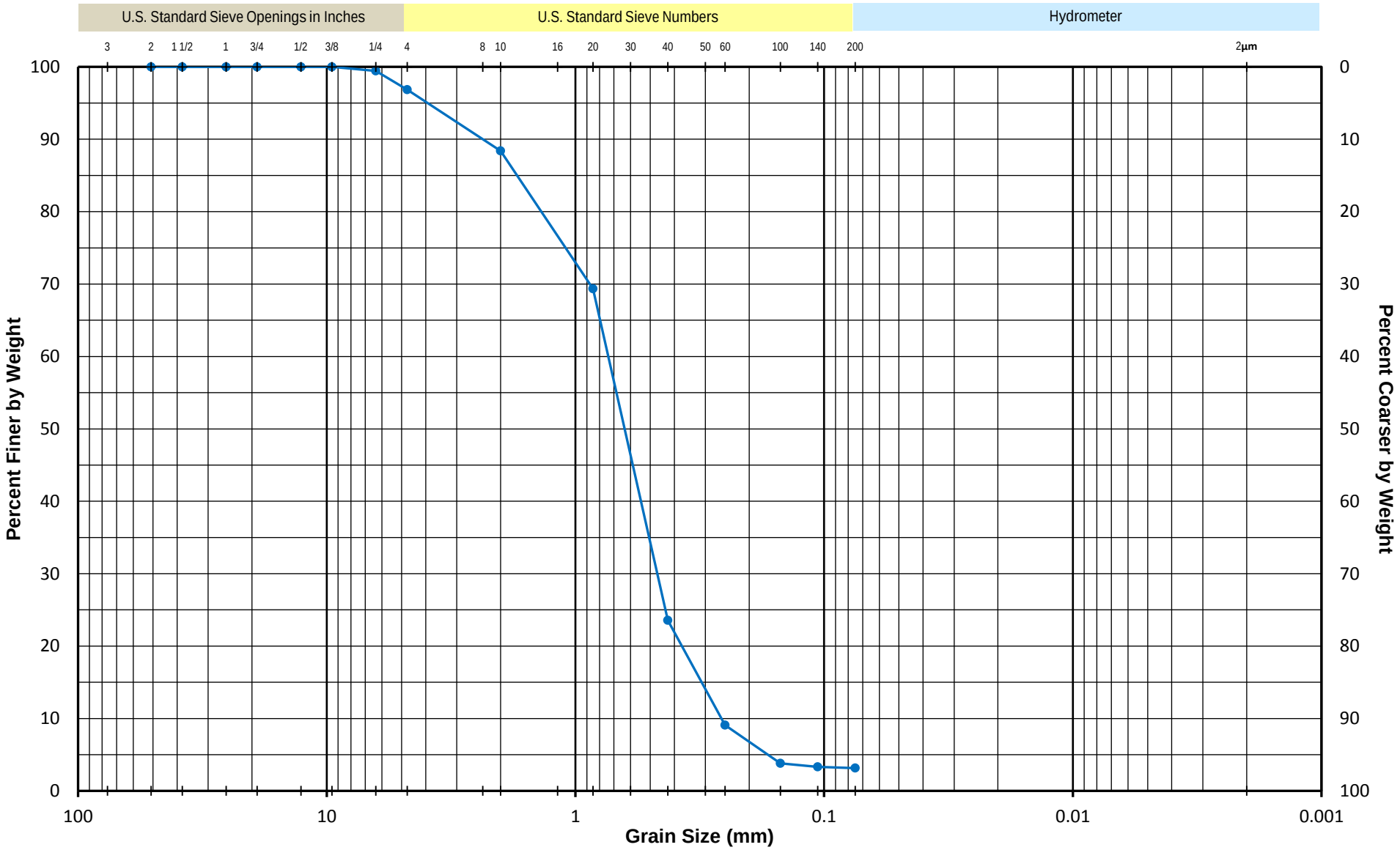


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC28 6-8	Dark Gray Fine to Coarse Sand with silt

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

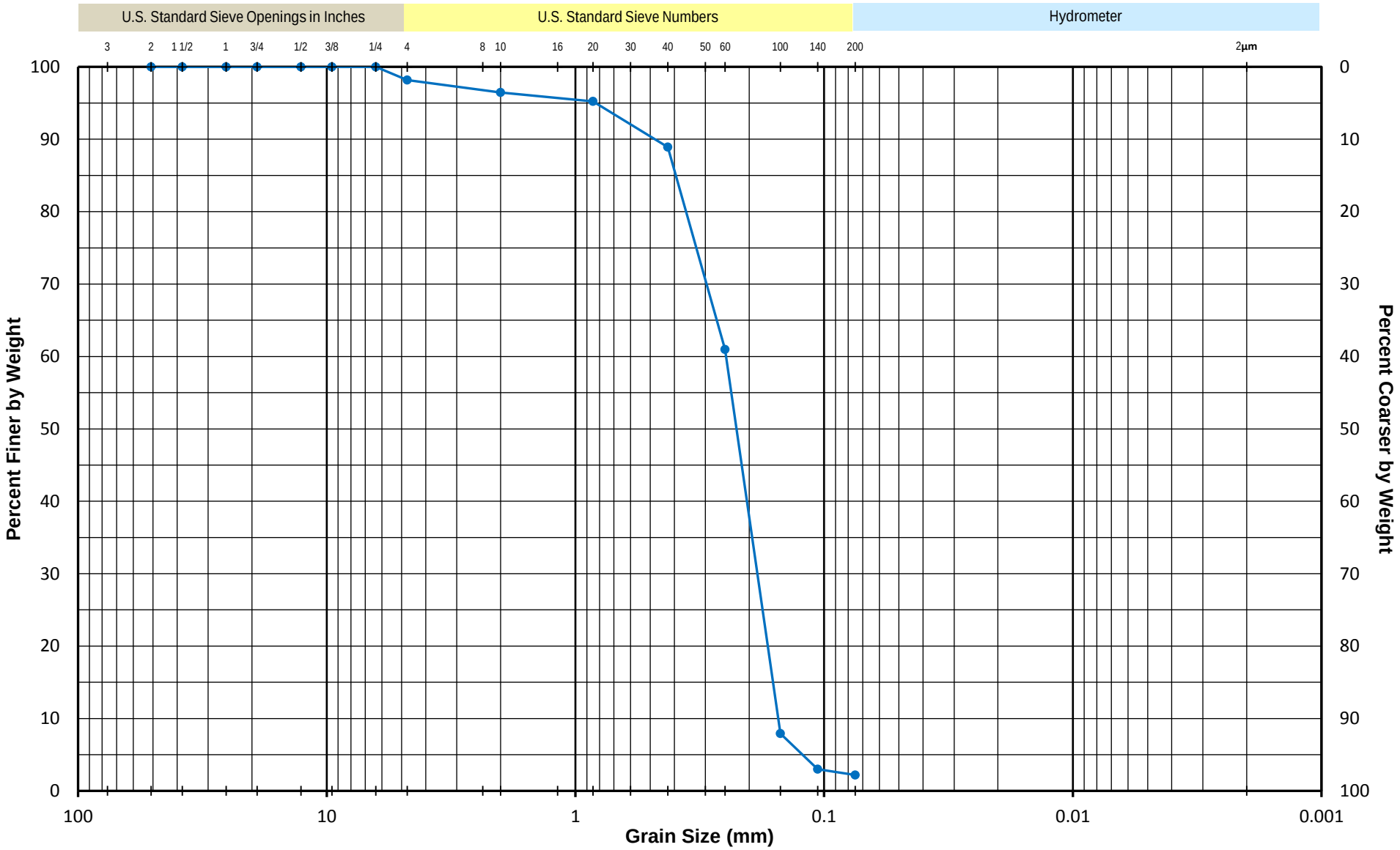


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC28 8-10	Dark Brown Medium to Coarse Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

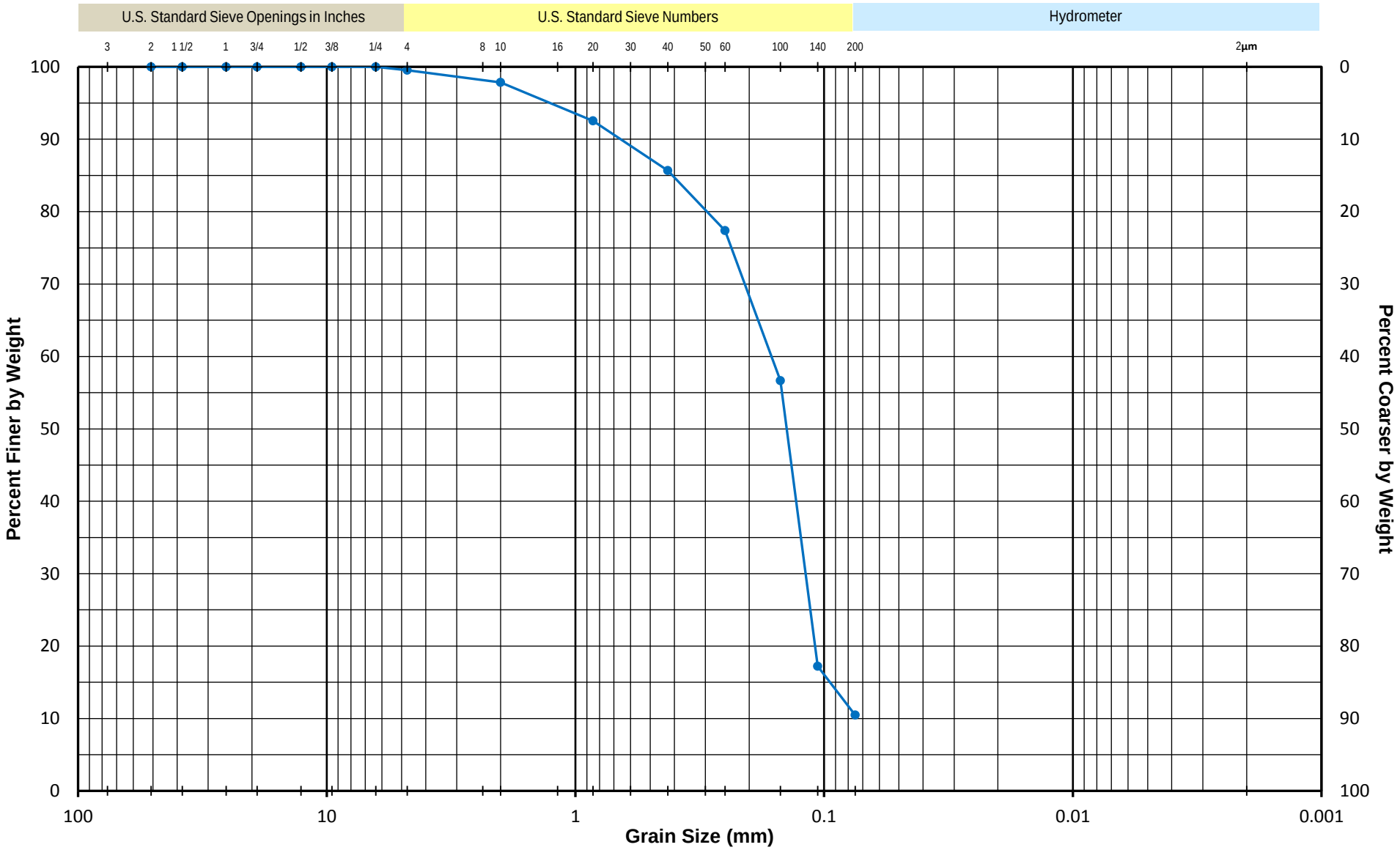


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC28 10-12	Dark Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

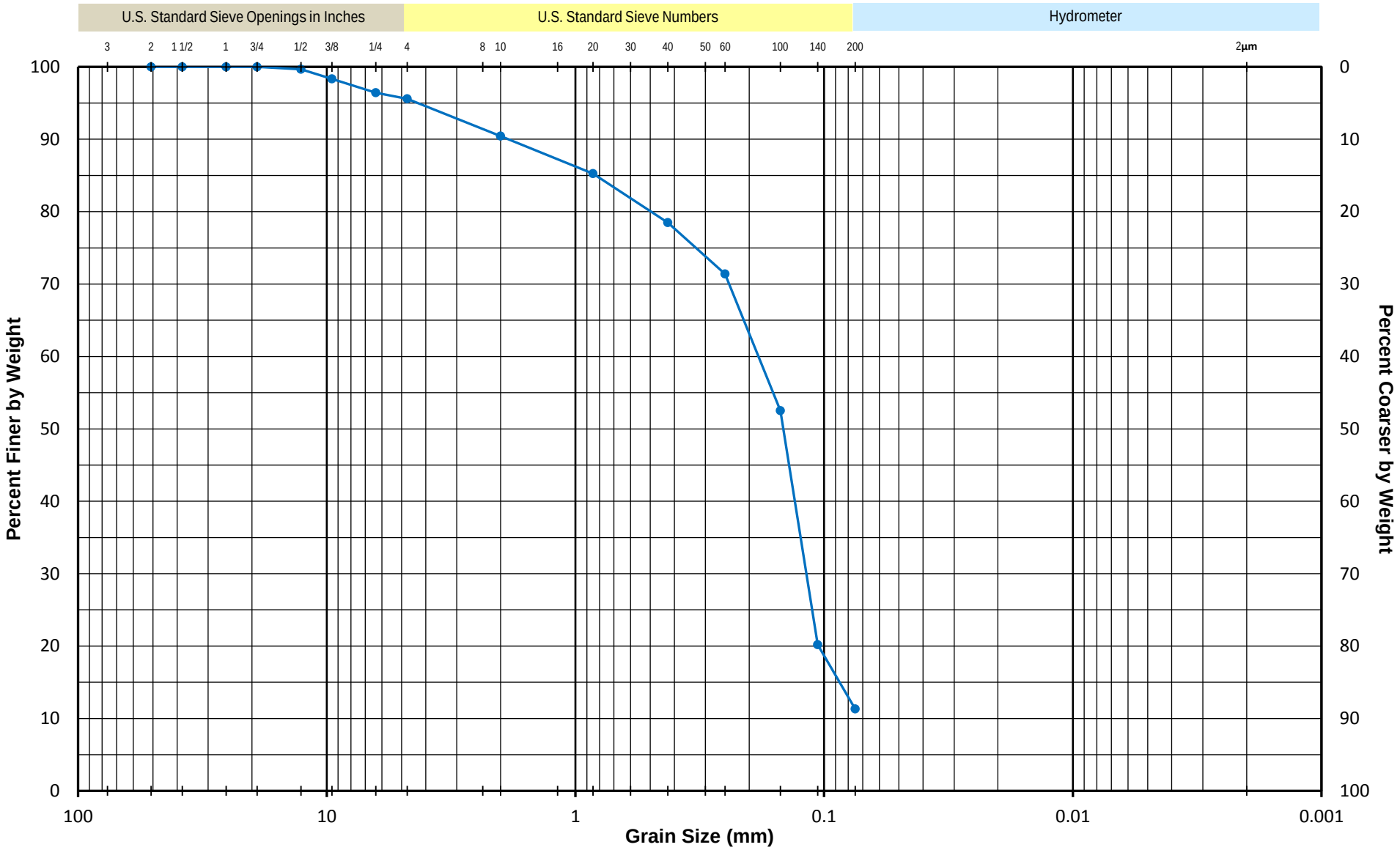


Test Depth	Core ID Section/Sample	Material Description
0.6 ft	VC30 0-2	Very Dark Gray Sand with silt and shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

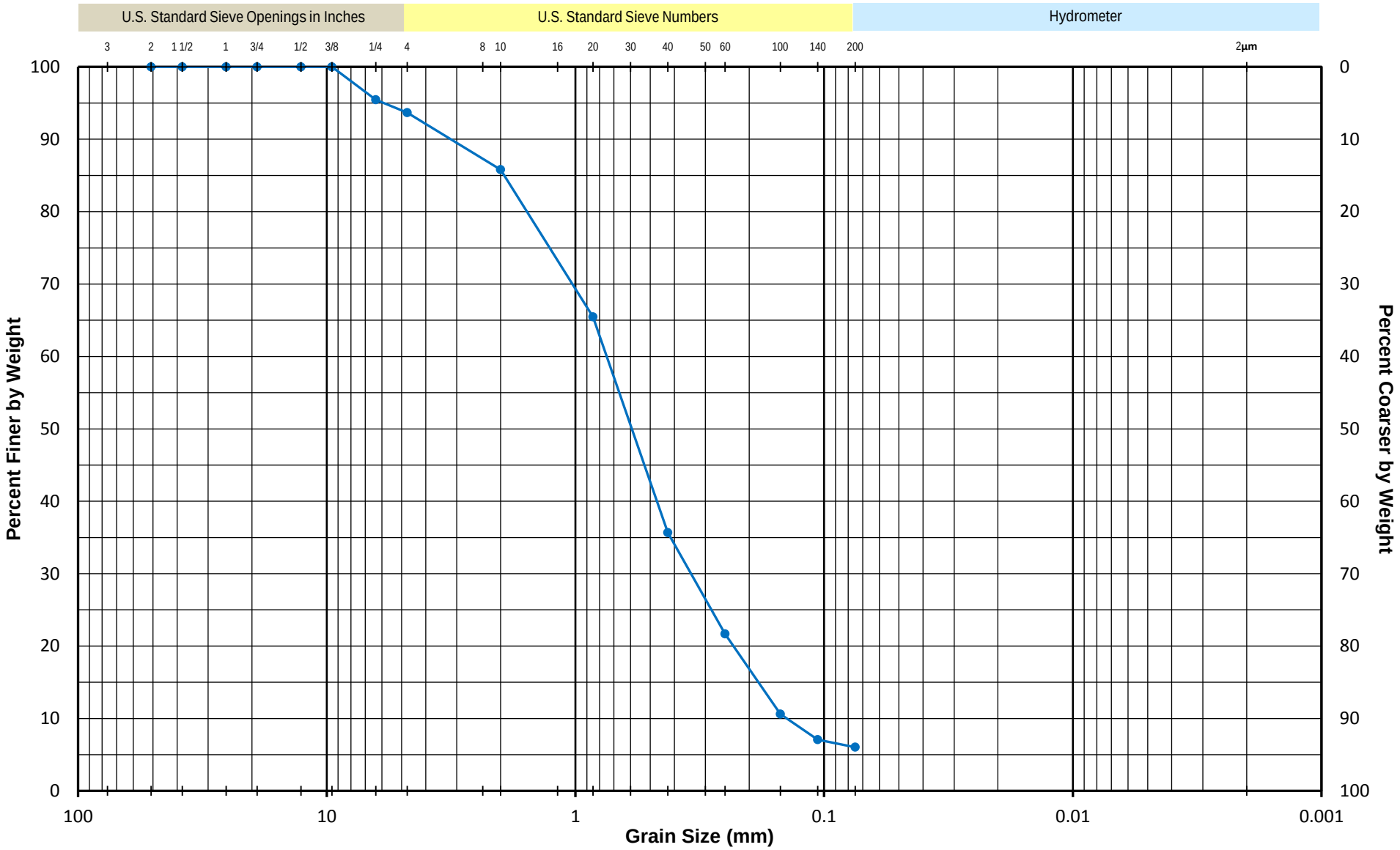


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC30 0-2	Very Dark Gray Fine to Medium Sand with silt and shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

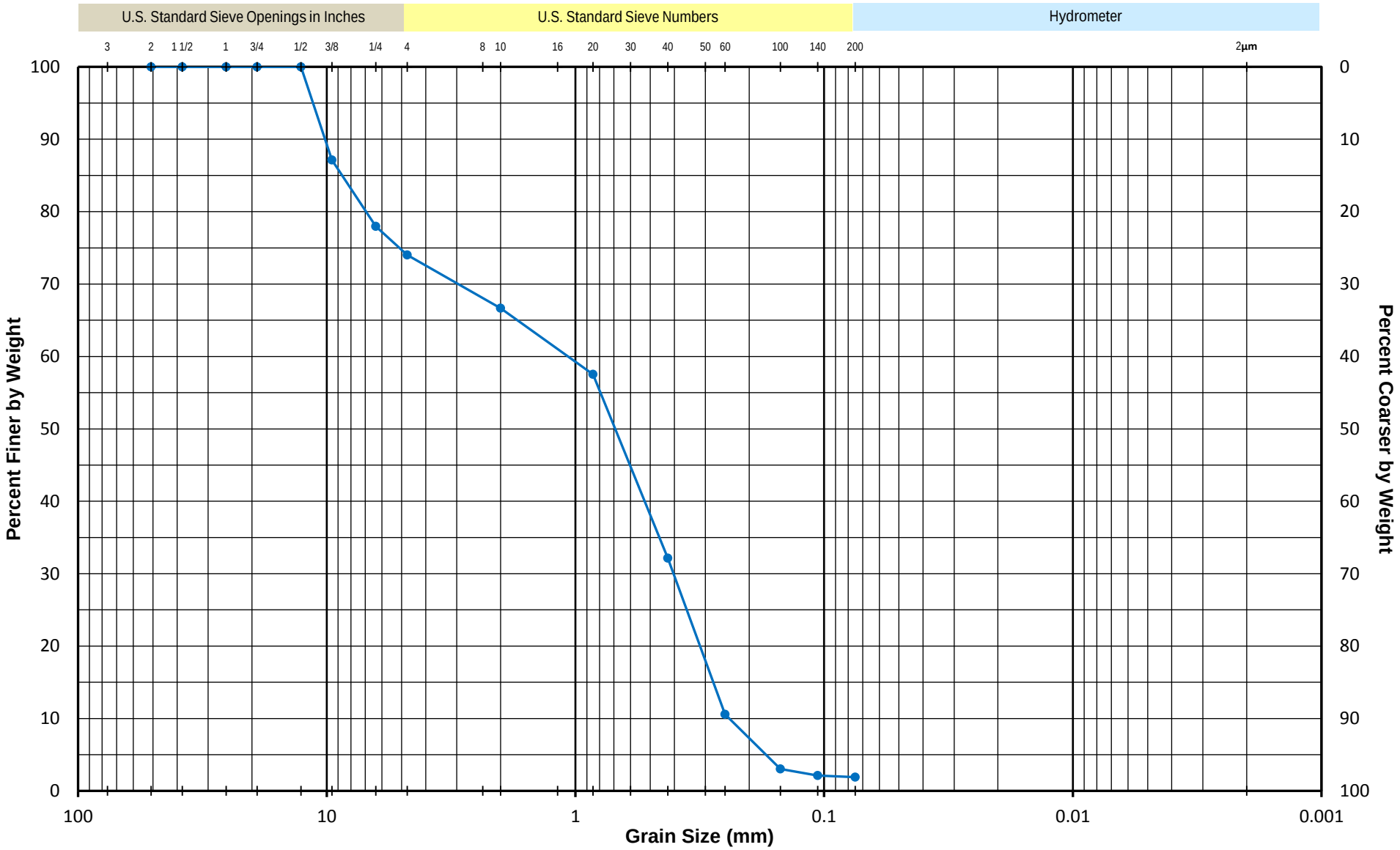


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC30 2-4	Very Dark Gray Medium to Fine Sand with silt

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

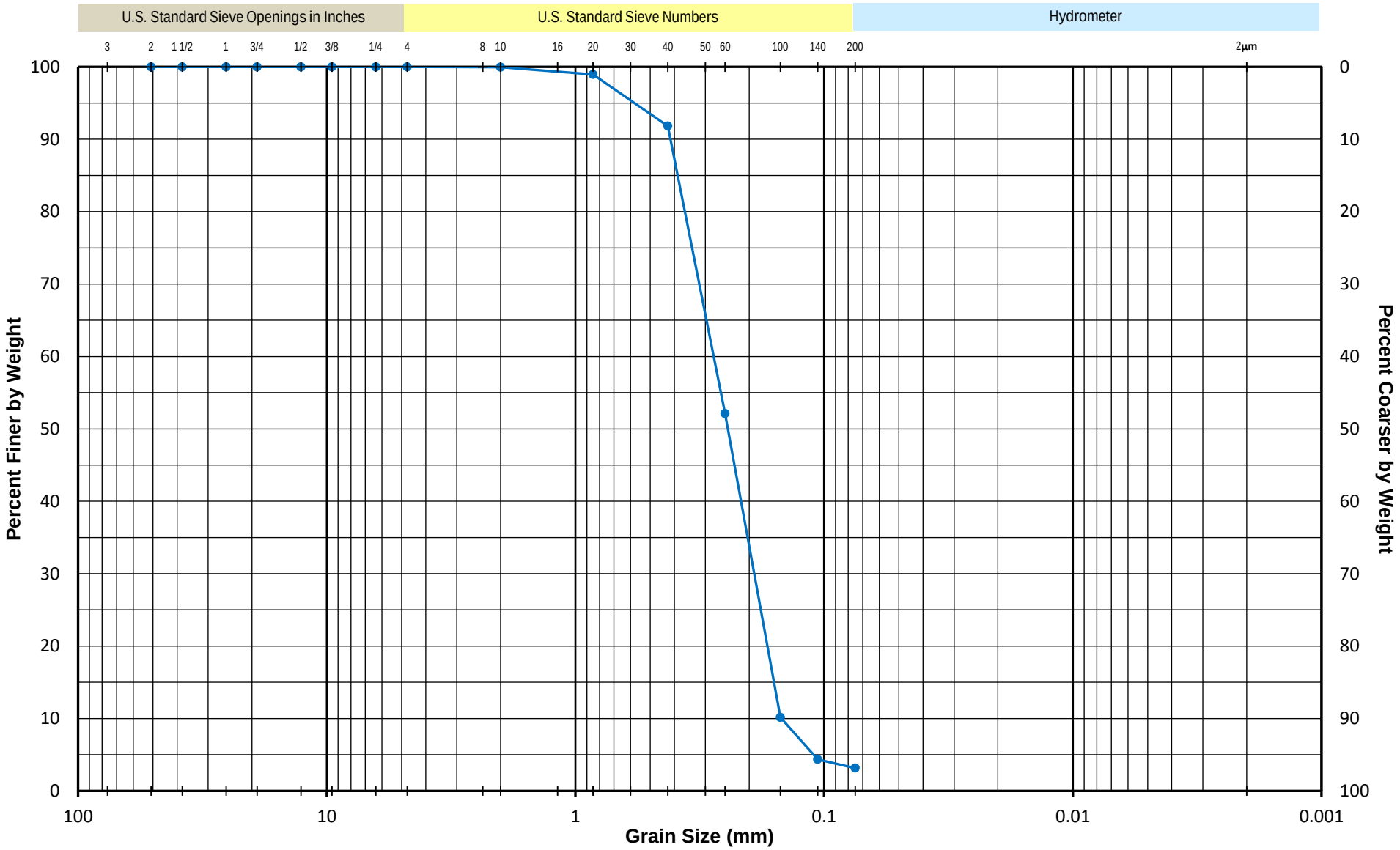


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC30 4-6	Brown Fine to Coarse Sand with gravel

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

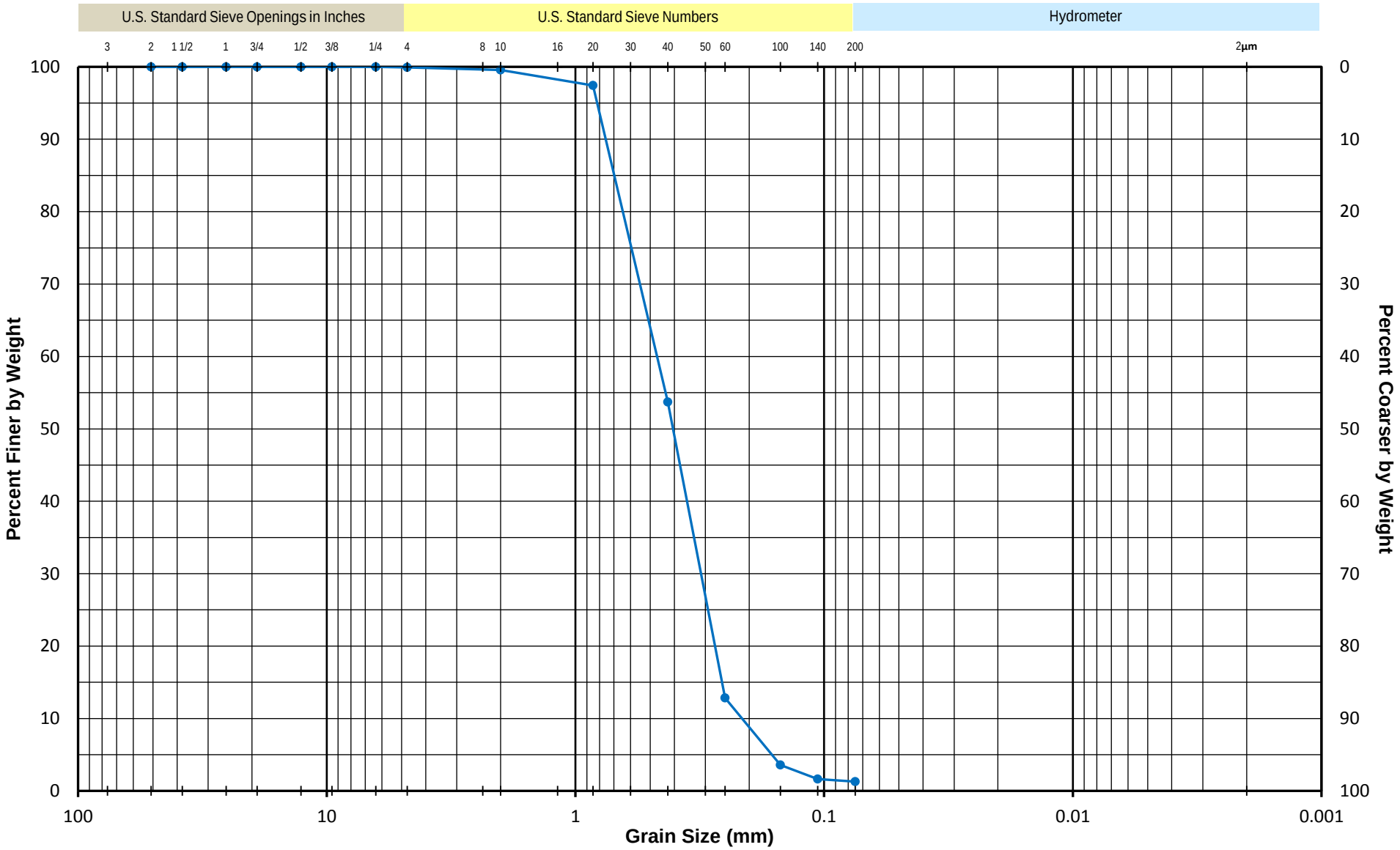


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC30 6-8	Strong Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

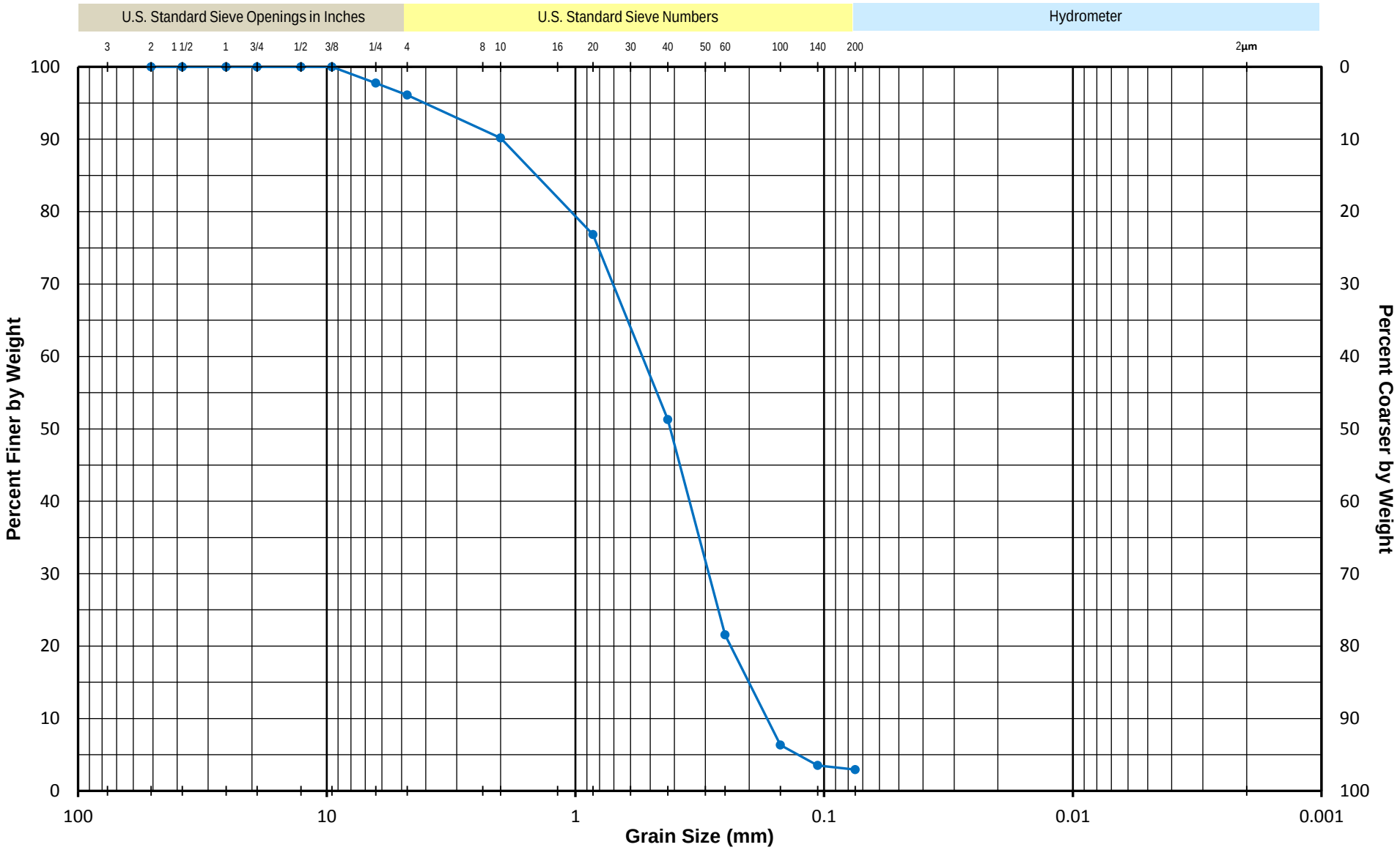


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC30 8-10	Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

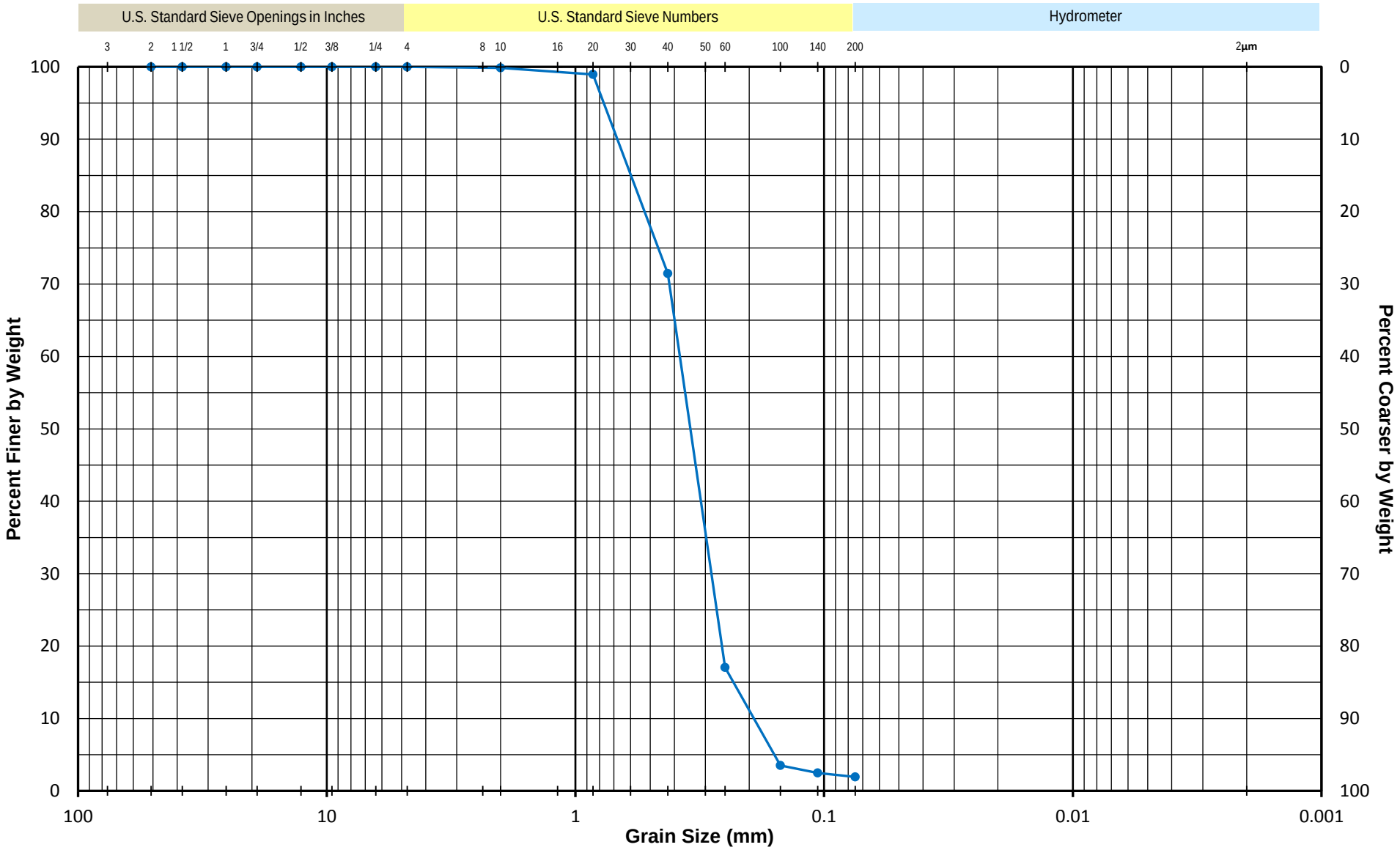


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC30 10-12	Dark Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

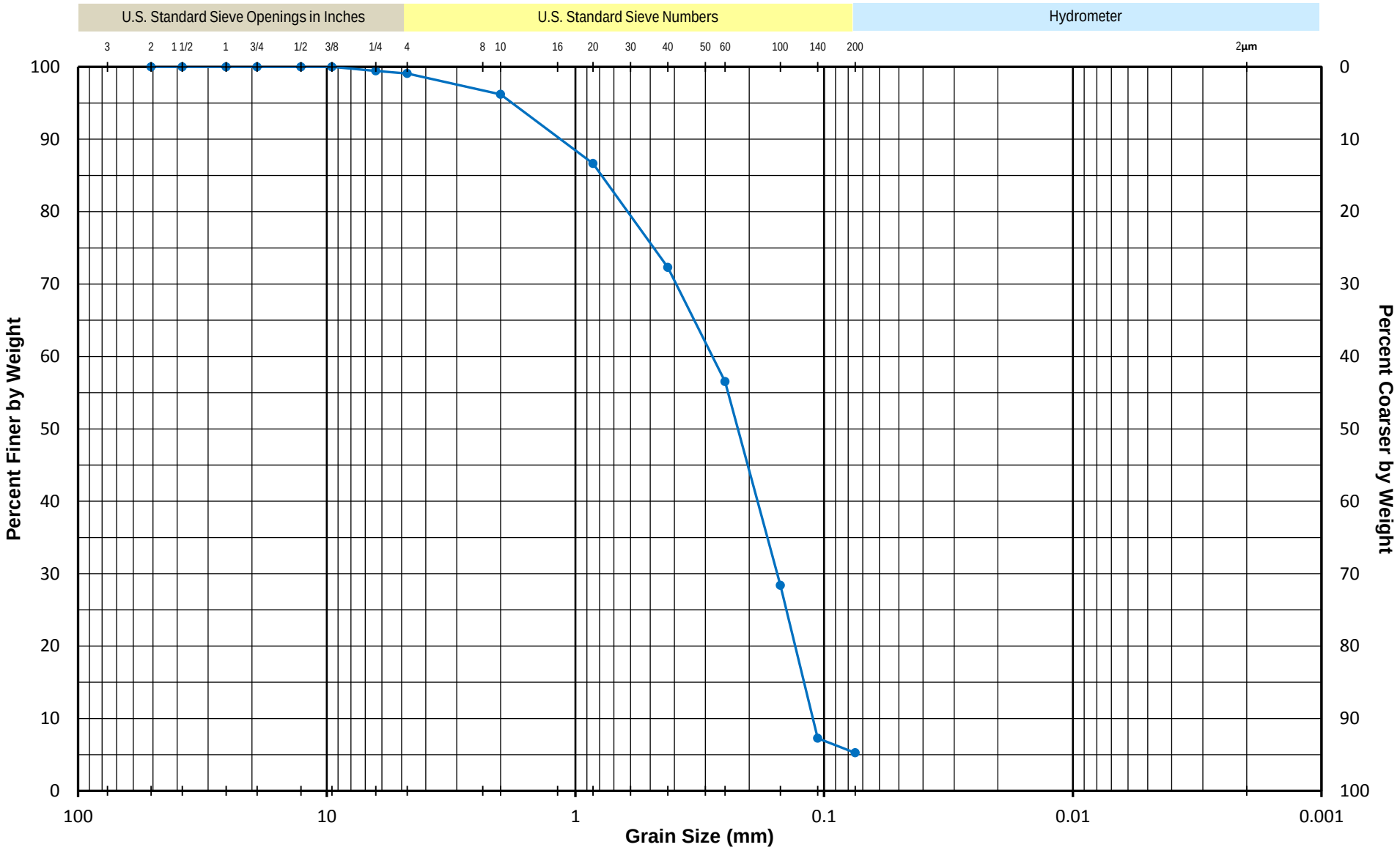


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC33 0-2	Very Dark Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

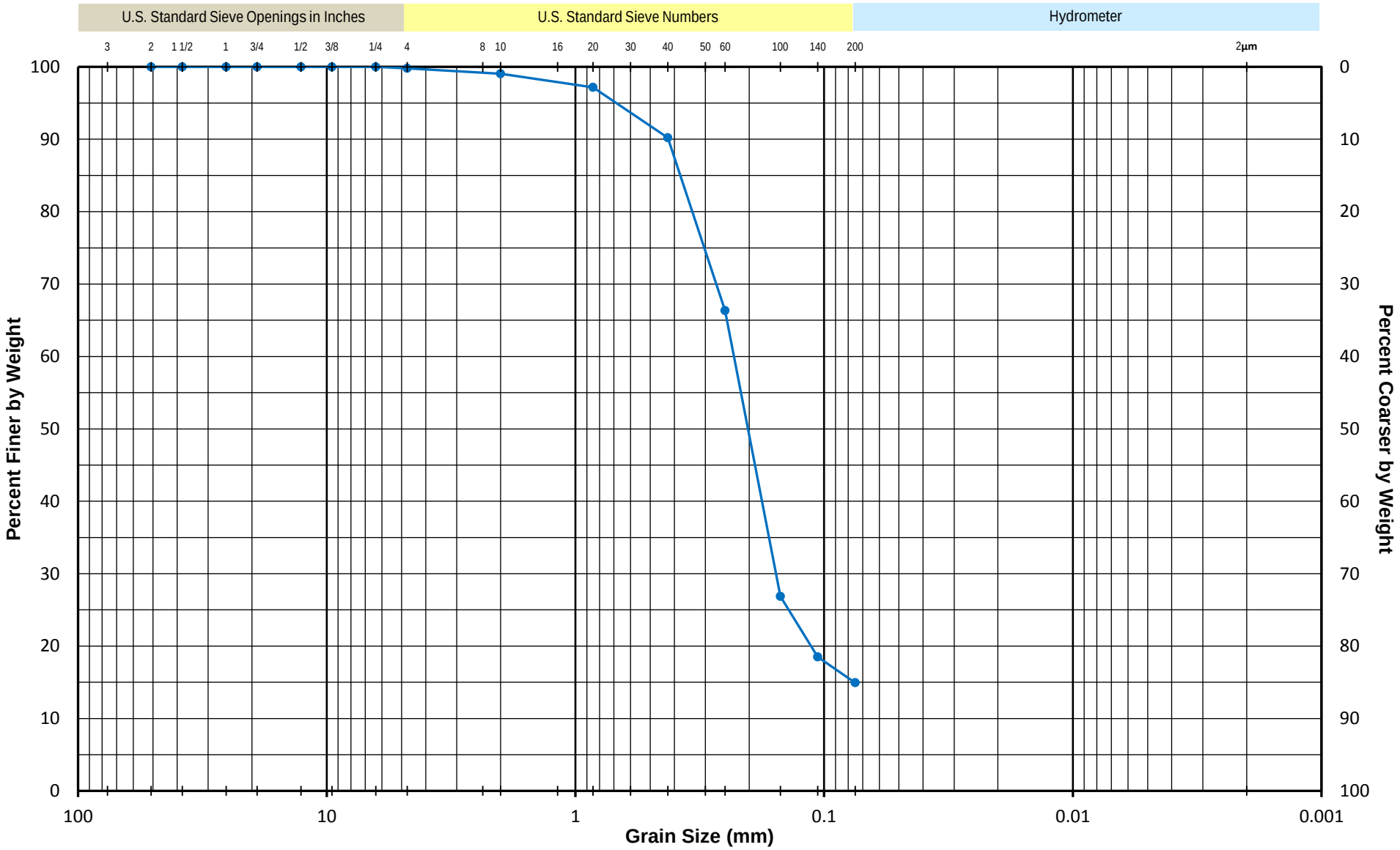


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC33 0-2	Very Dark Gray Medium to Fine Sand with silt and shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

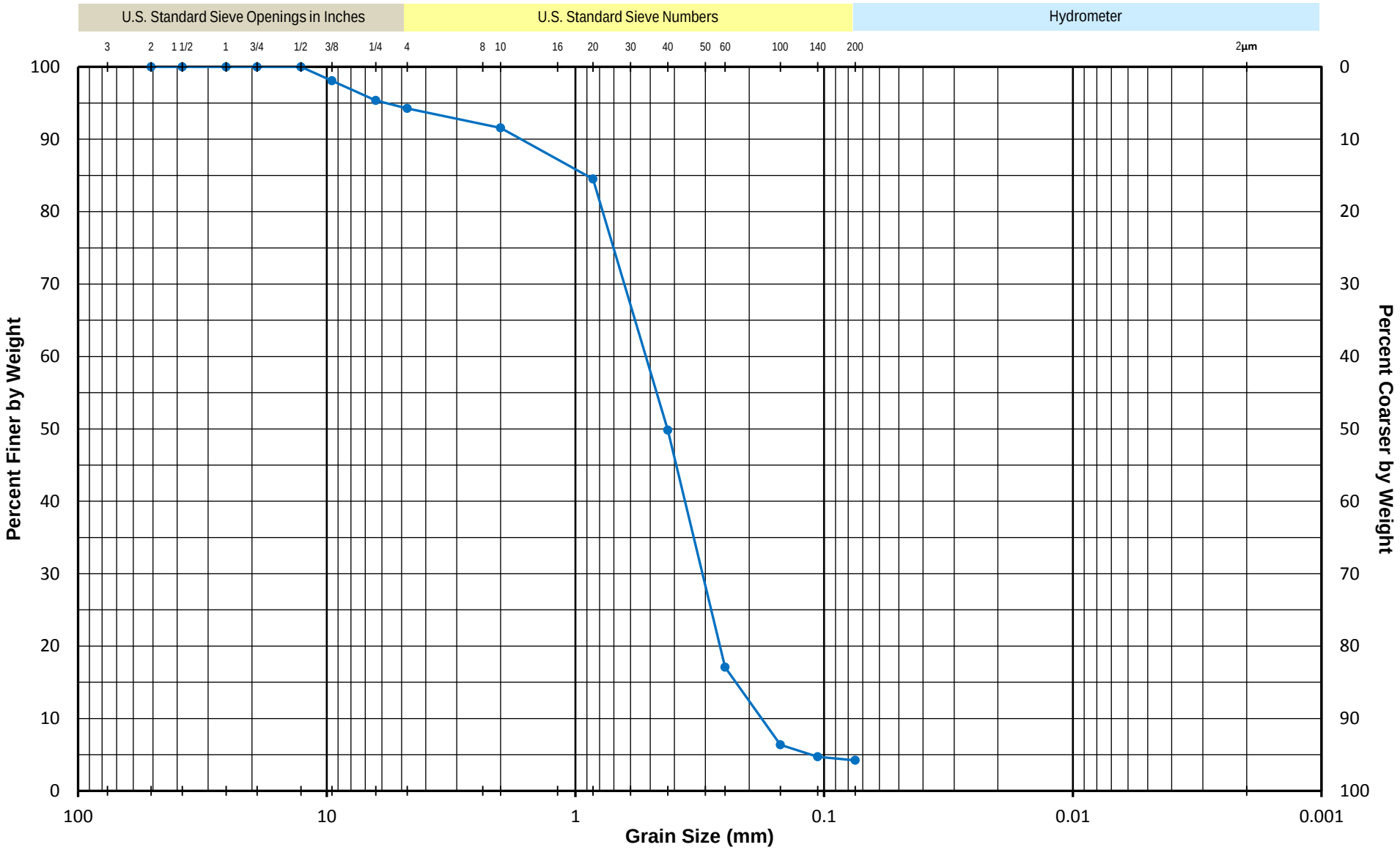


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC33 2-4	Brown and Gray Silty Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

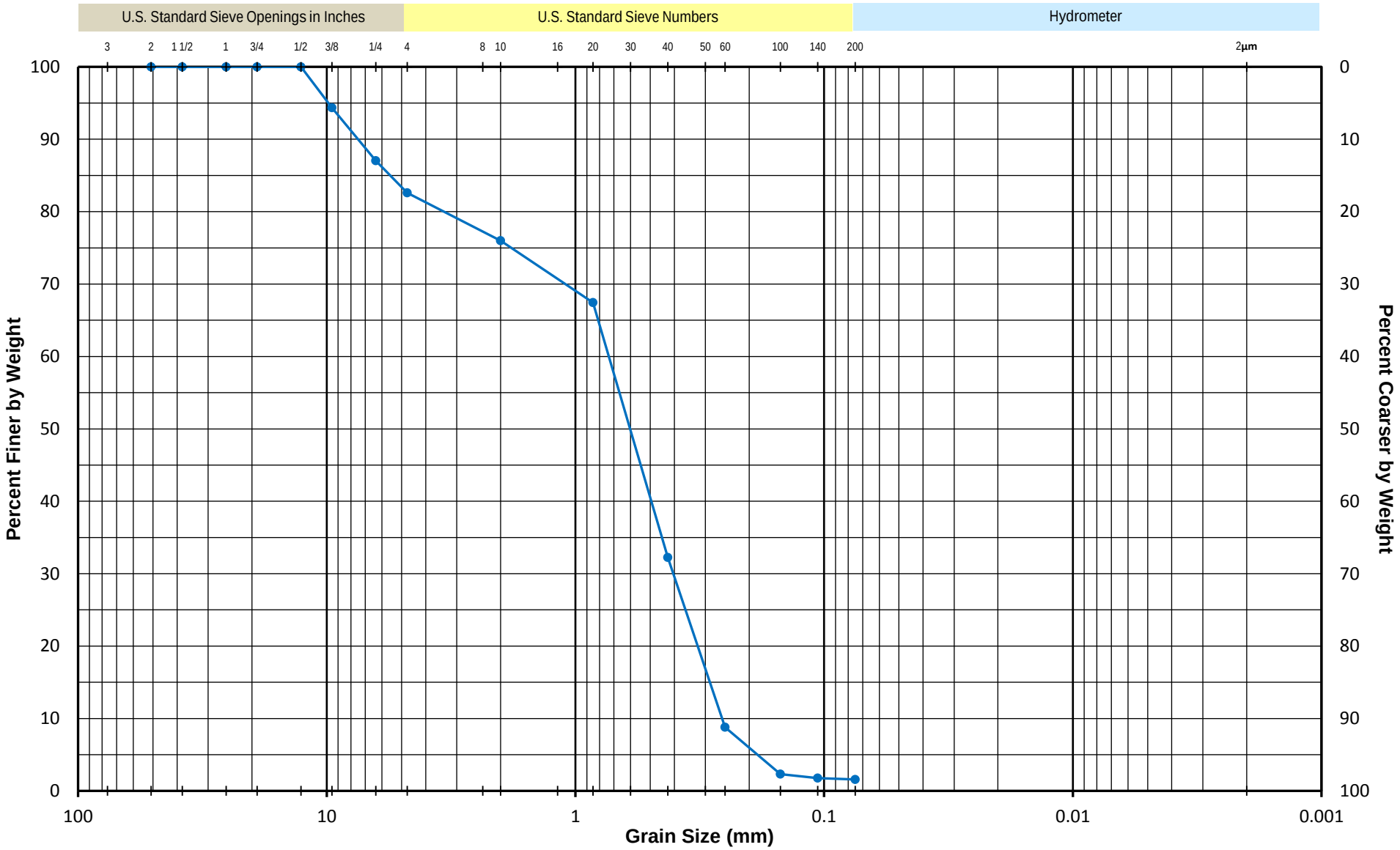


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC33 4-6	Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

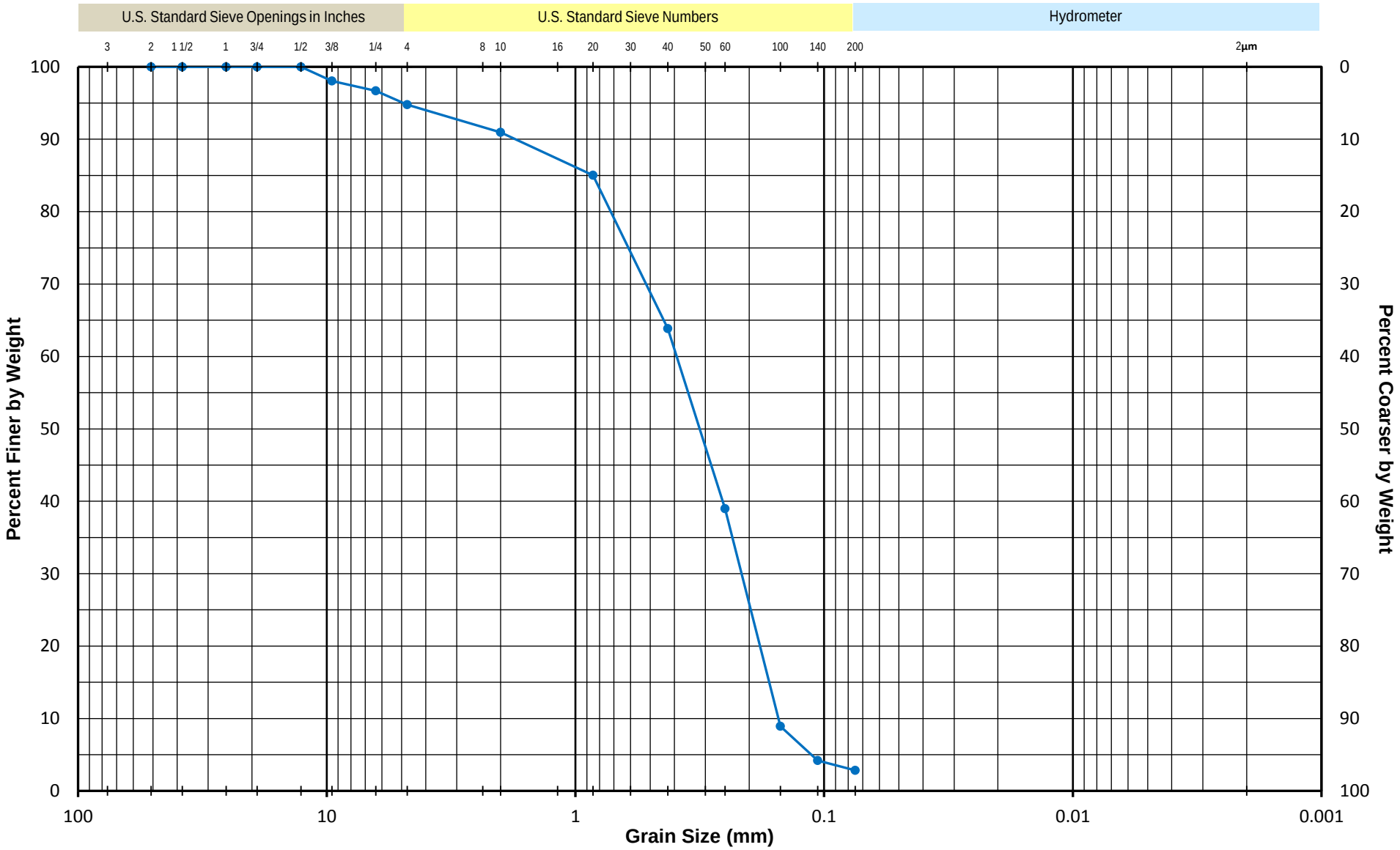


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC33 6-8	Brown Medium to Coarse Sand with gravel

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

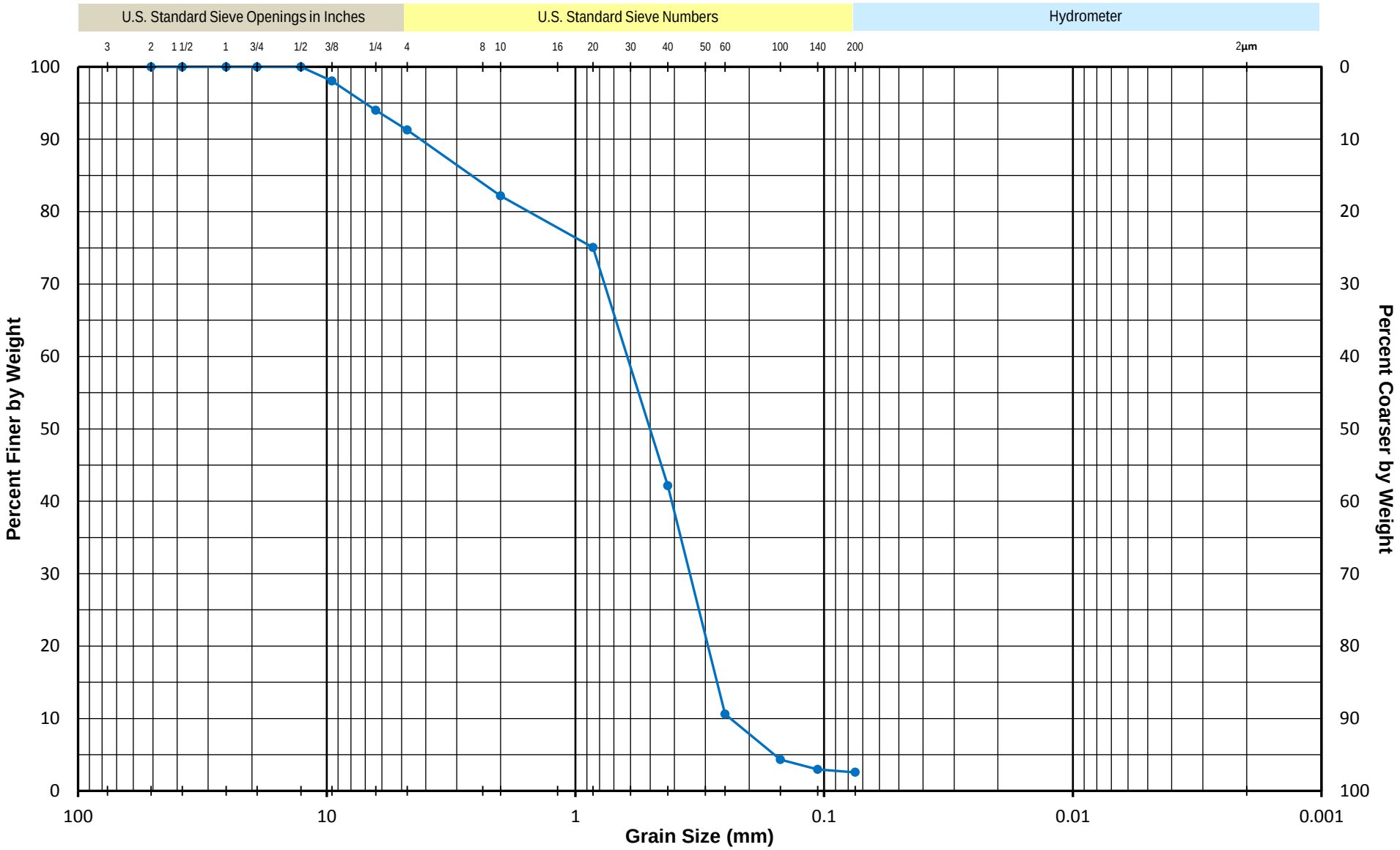


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC33 8-10	Very Dark Grayish Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

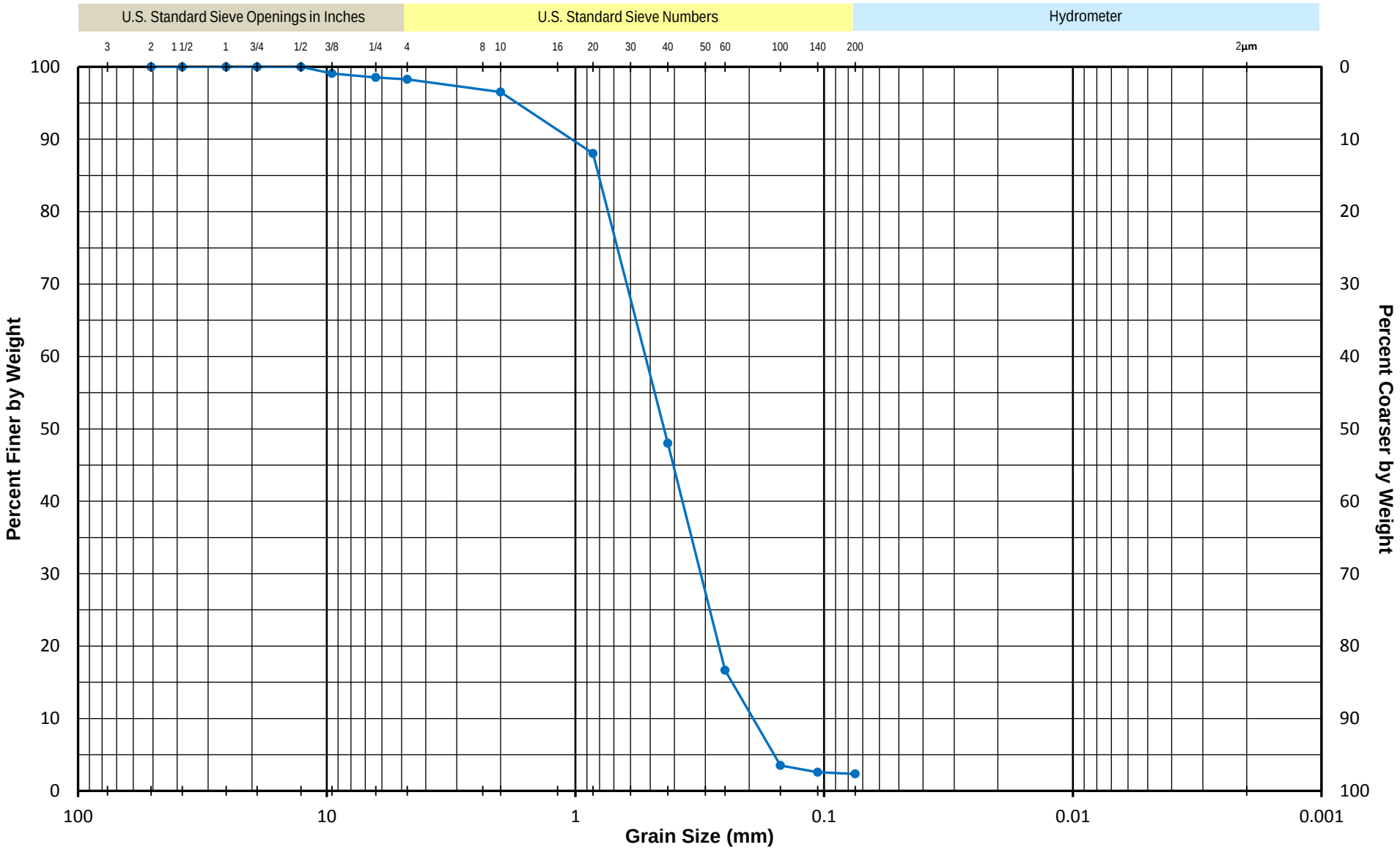


Test Depth	Core ID Section/Sample	Material Description
11.7 ft	VC33 10-12	Very Dark Grayish Brown Fine to Coarse Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

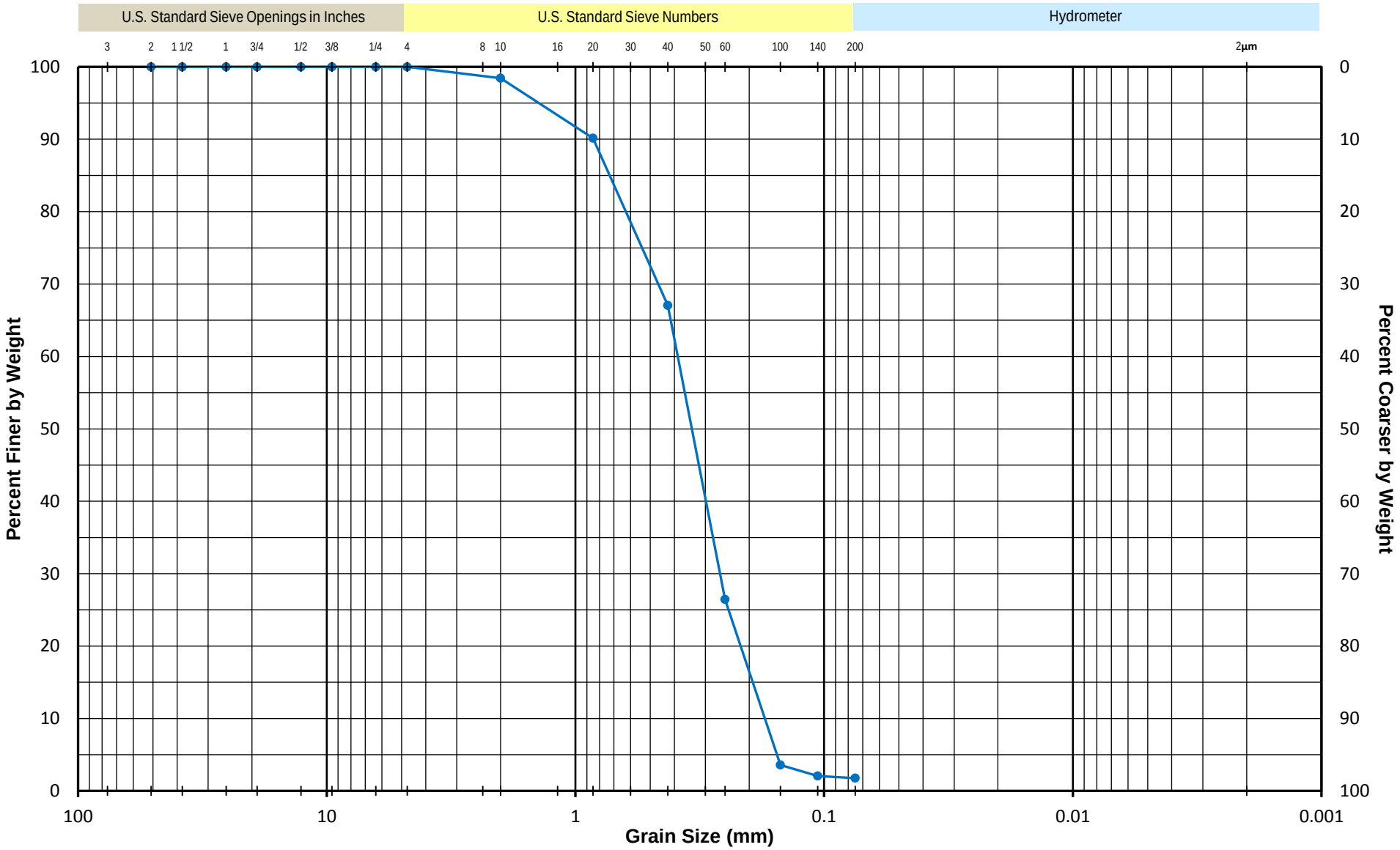


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC34 0-2	Gray Fine to Medium Sand with shells

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

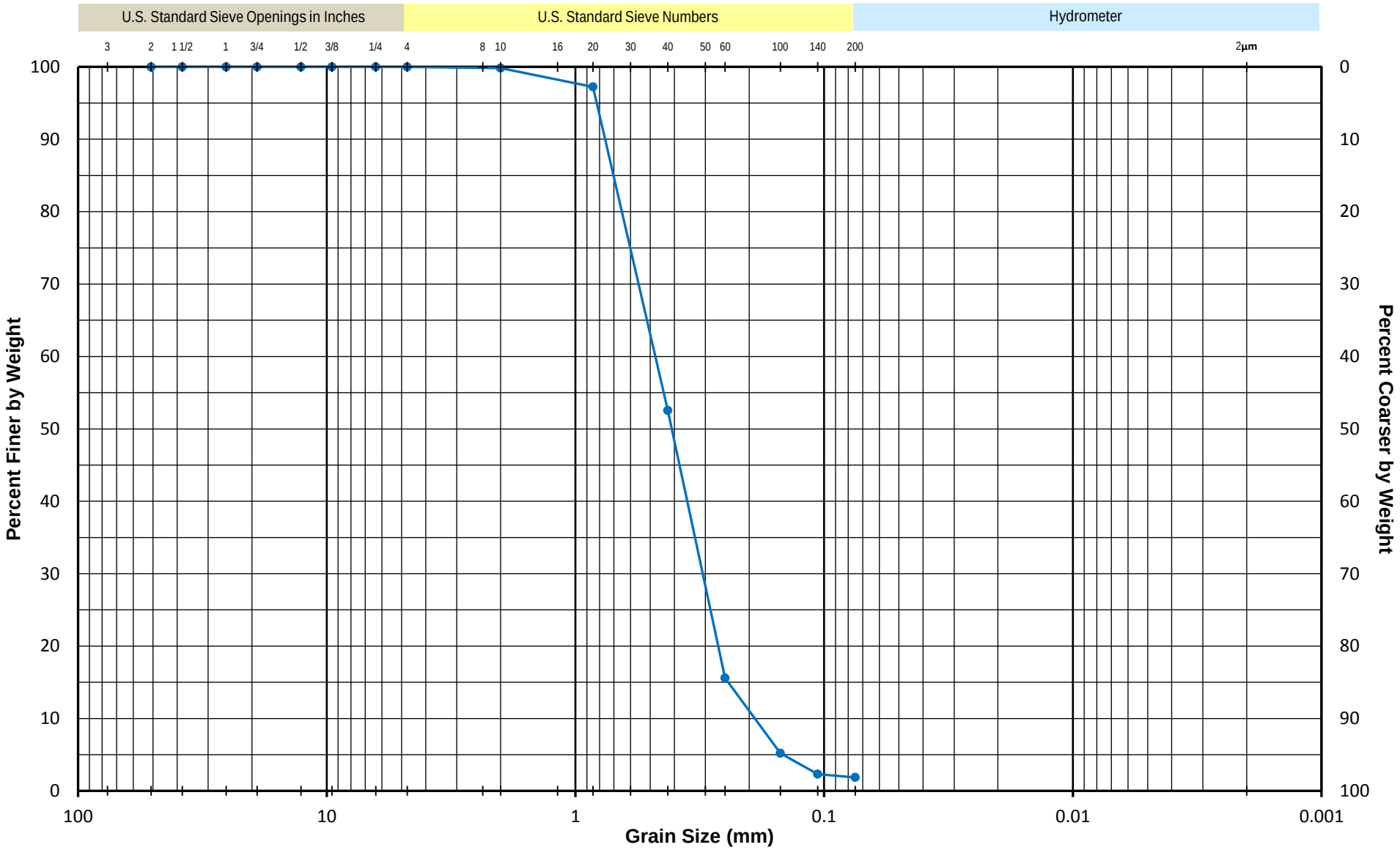


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC34 0-2	Very Dark Grayish Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

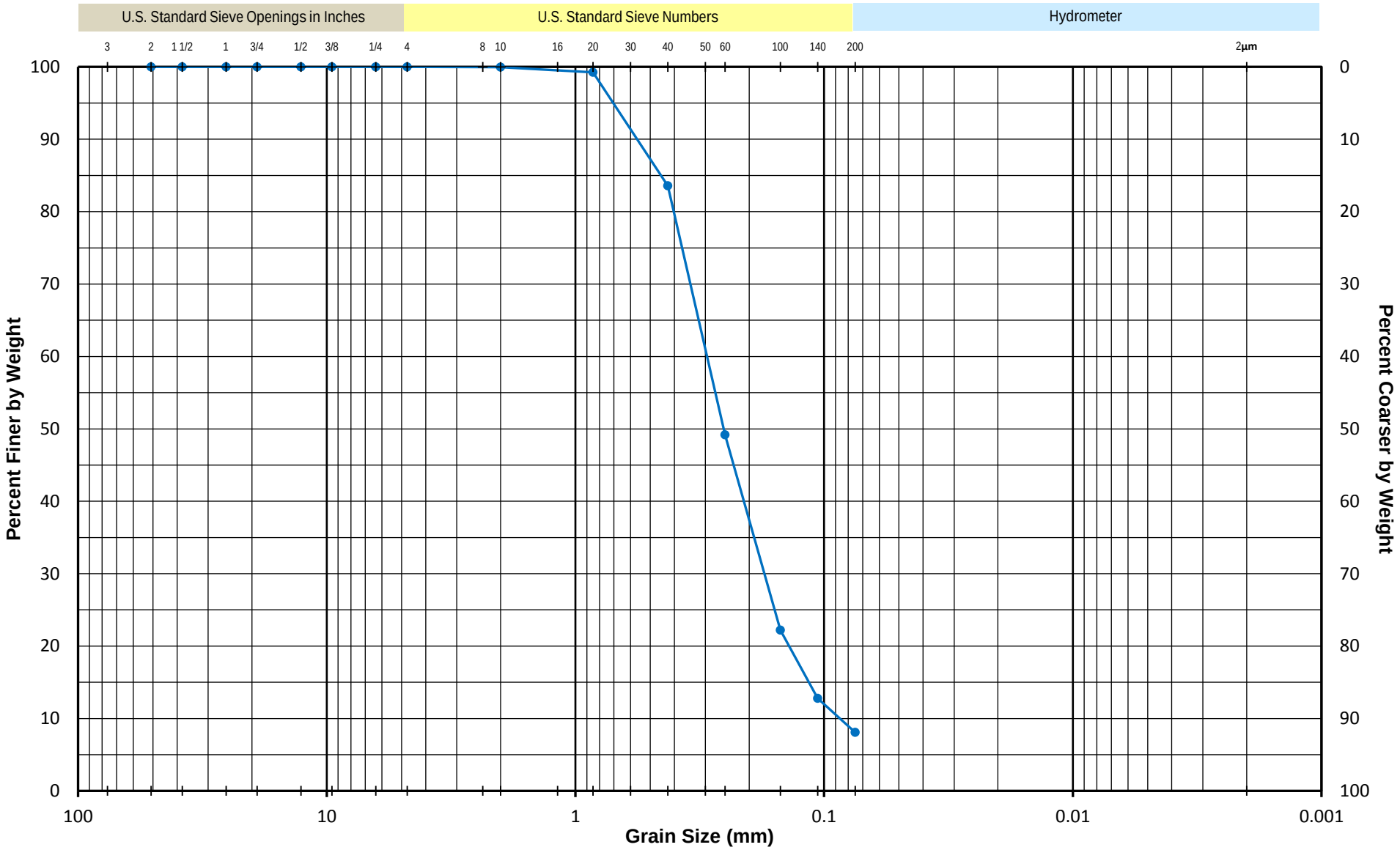


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC34 2-4	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

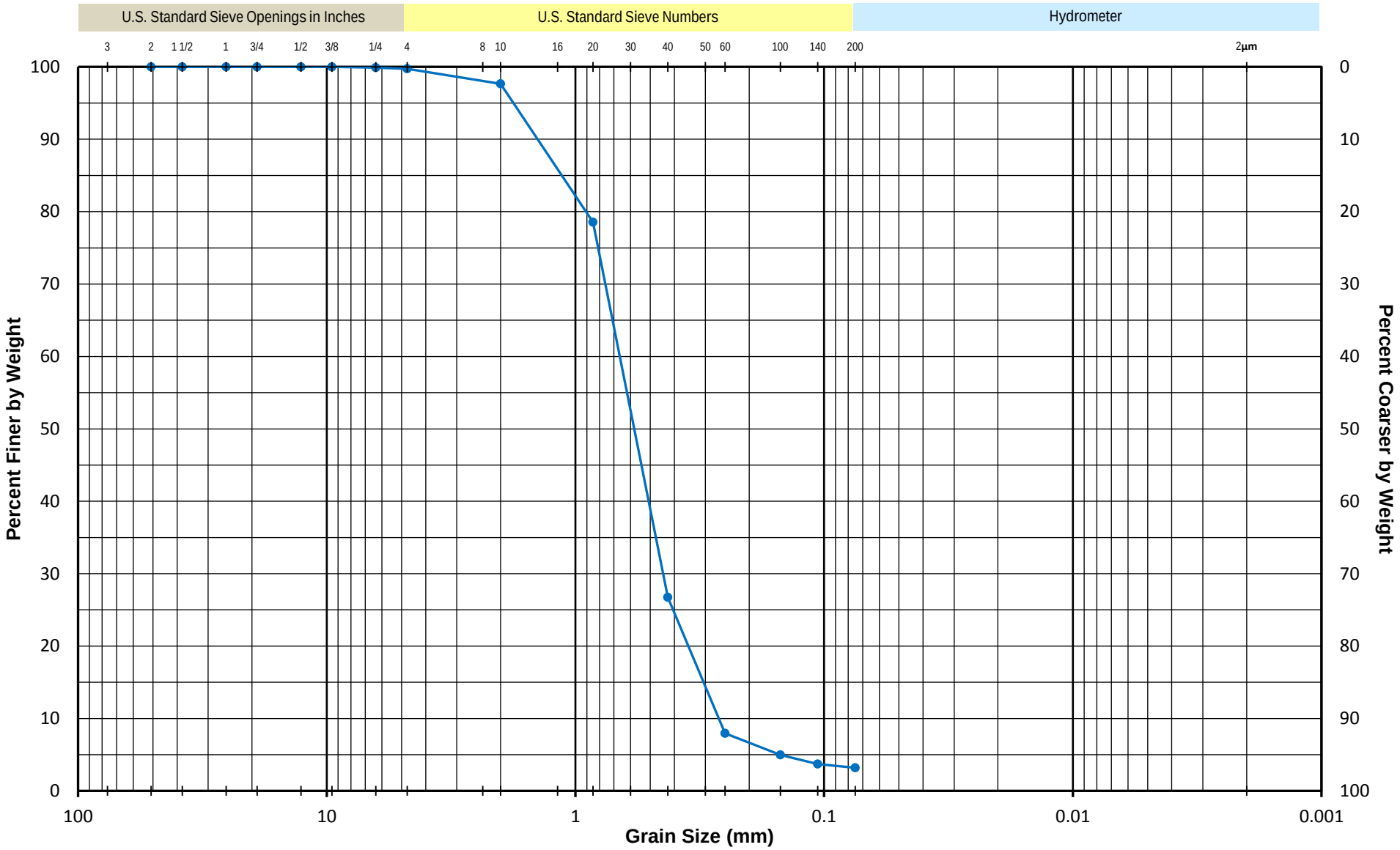


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC34 4-6	Dark Gray Fine to Medium Sand with silt

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

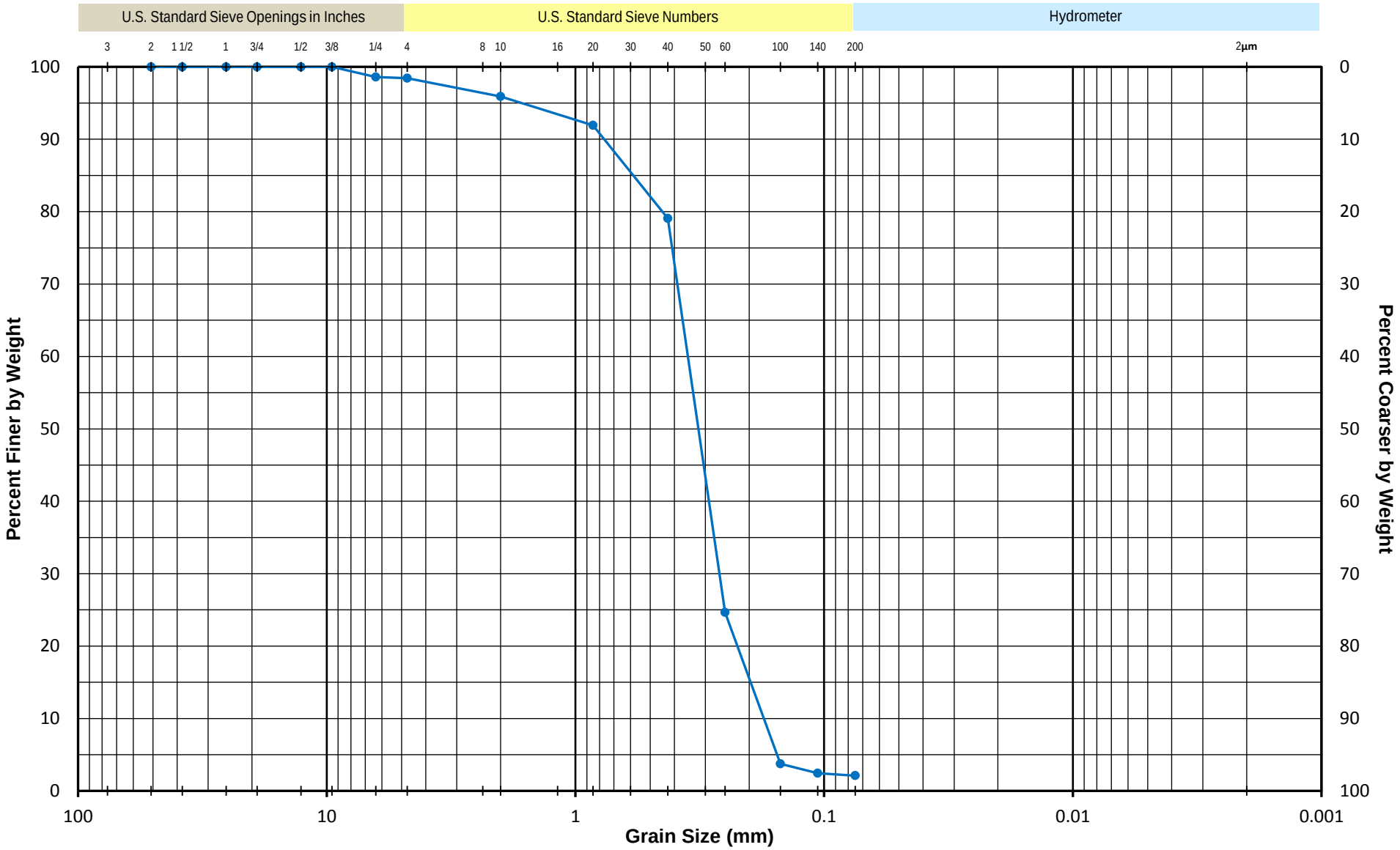


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC34 6-8	Very Dark Gray Medium to Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

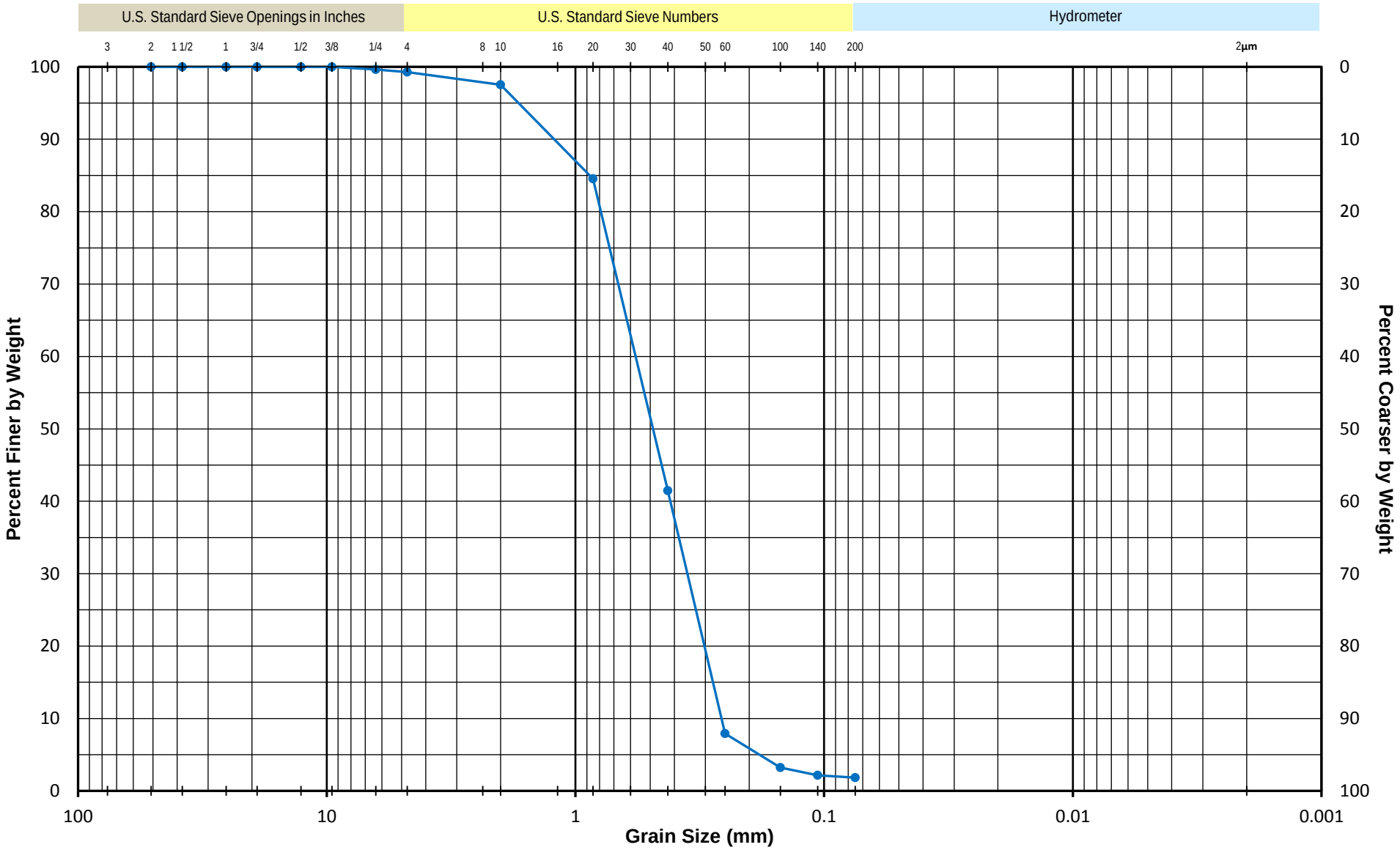


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC34 8-10	Black Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

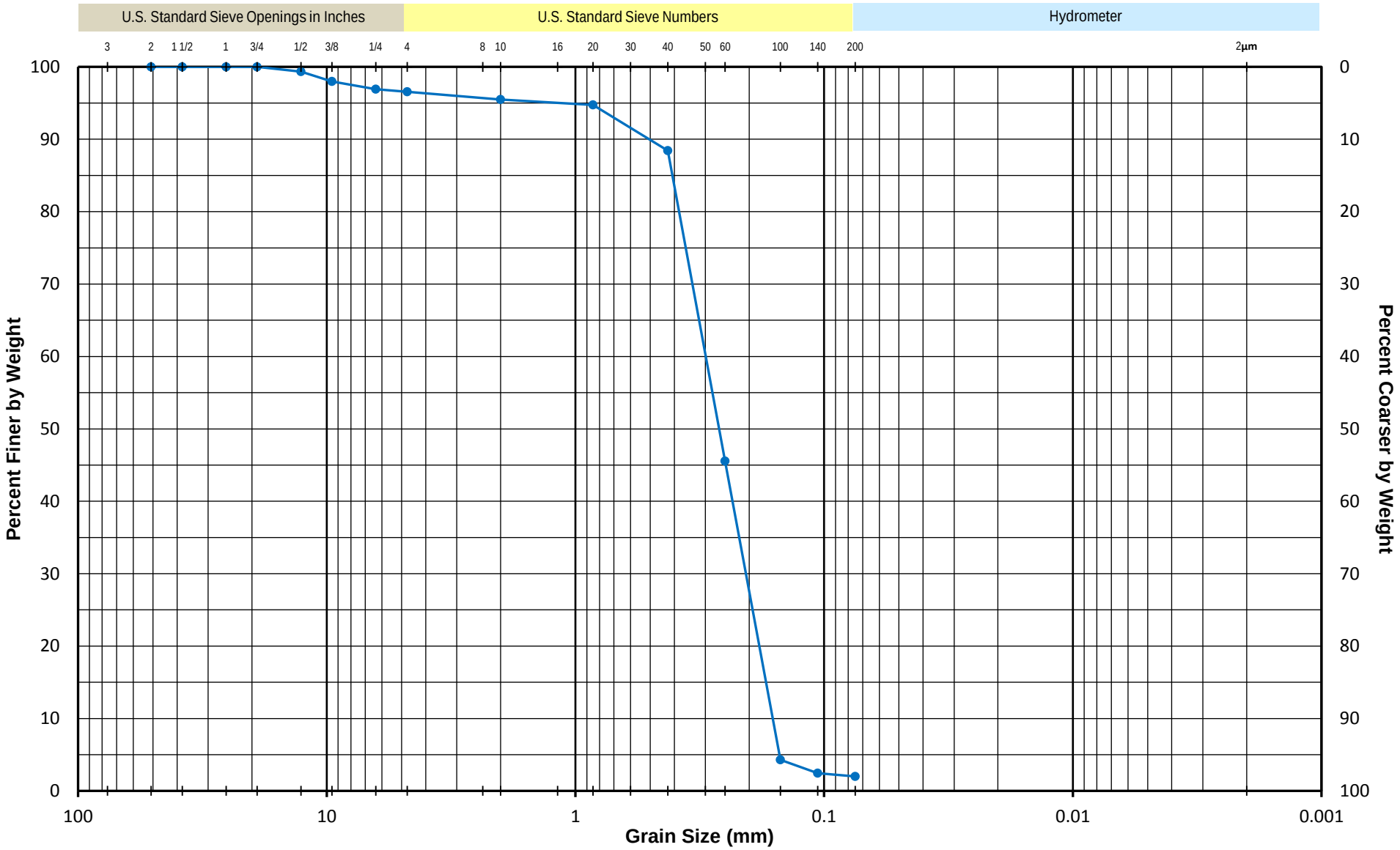


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC34 10-12	Dark Grayish Brown Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

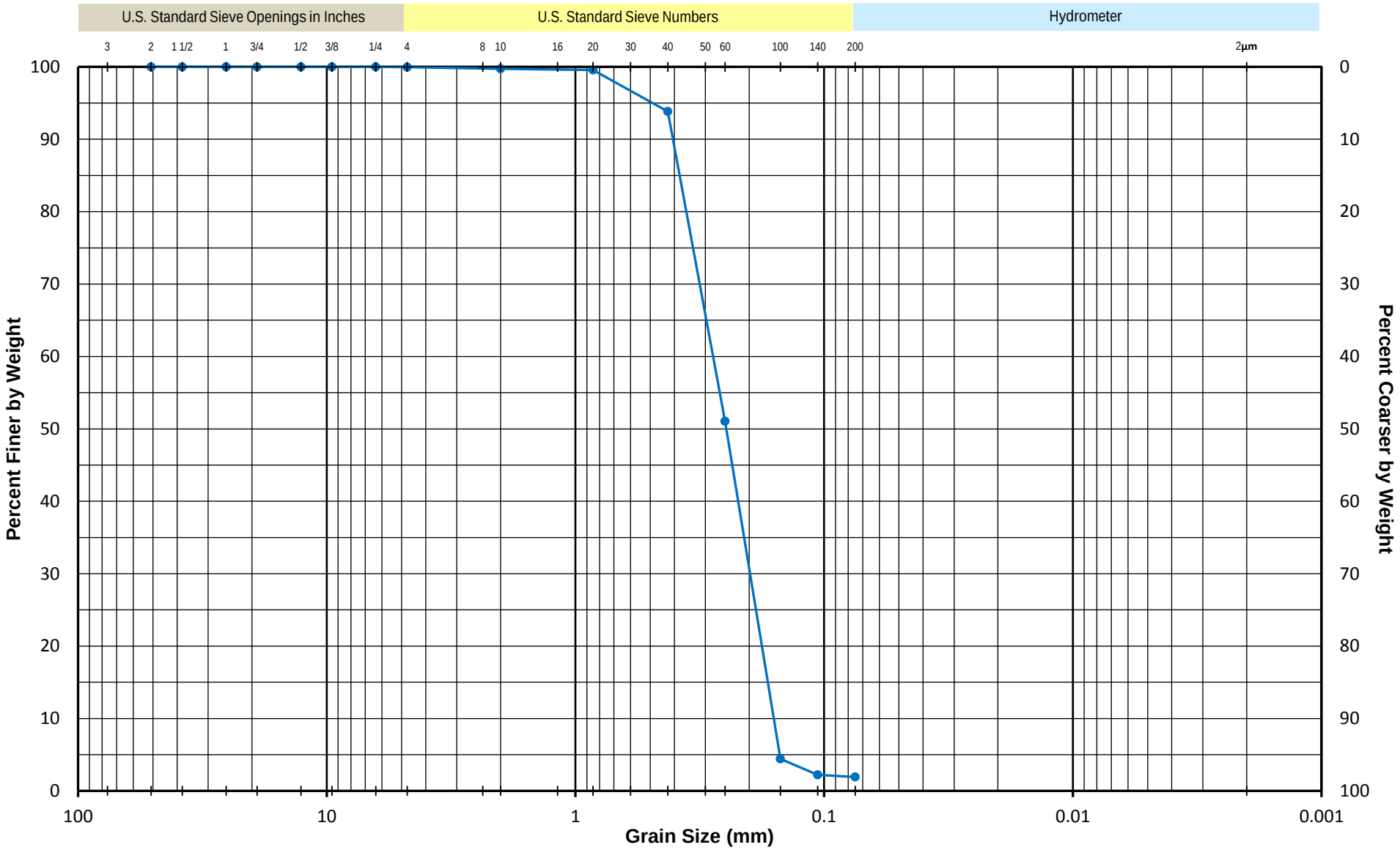


Test Depth	Core ID Section/Sample	Material Description
0.5 ft	VC42 0-2	Dark Gray Fine to Medium Sand with shells

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

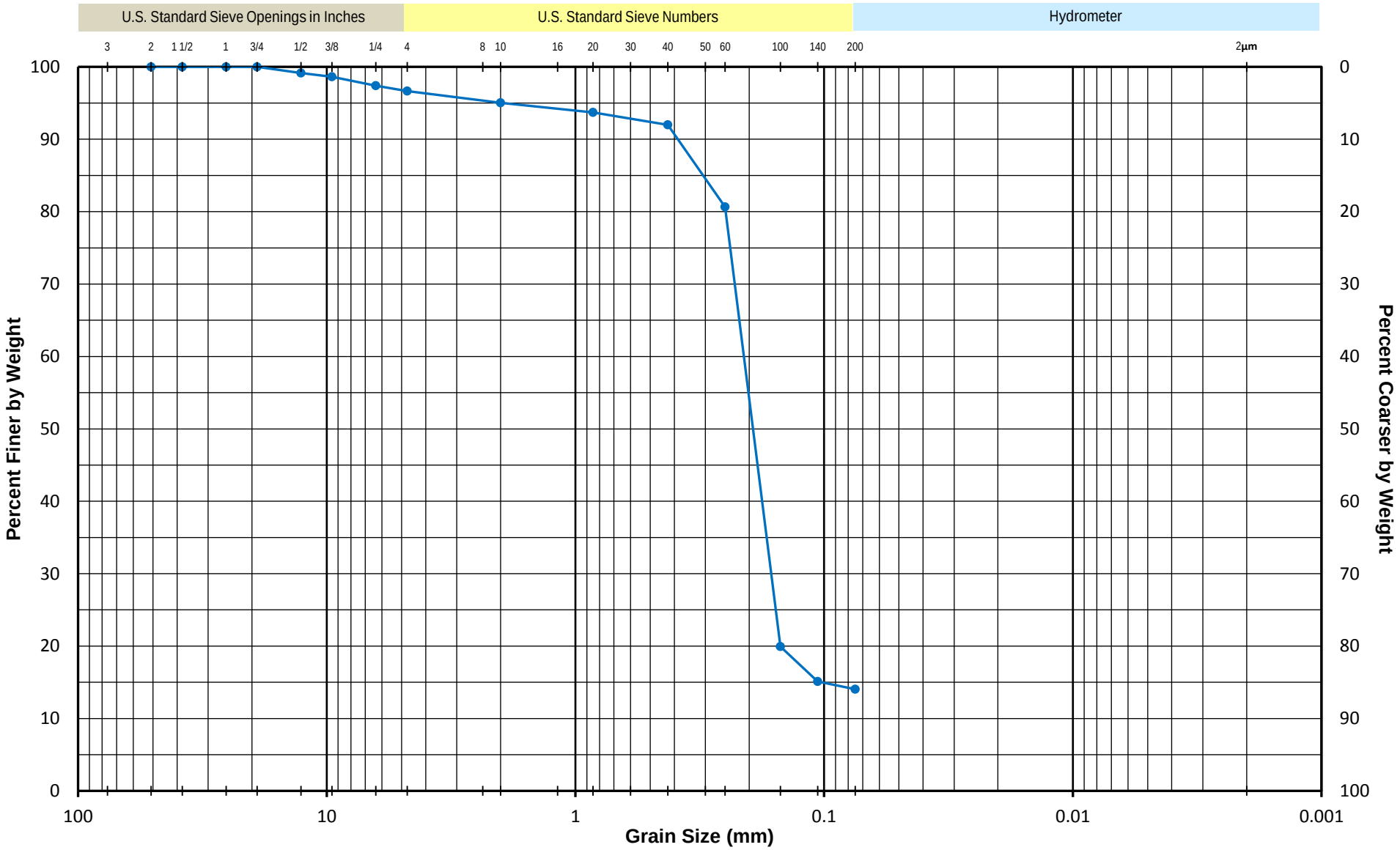


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC42 0-2	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

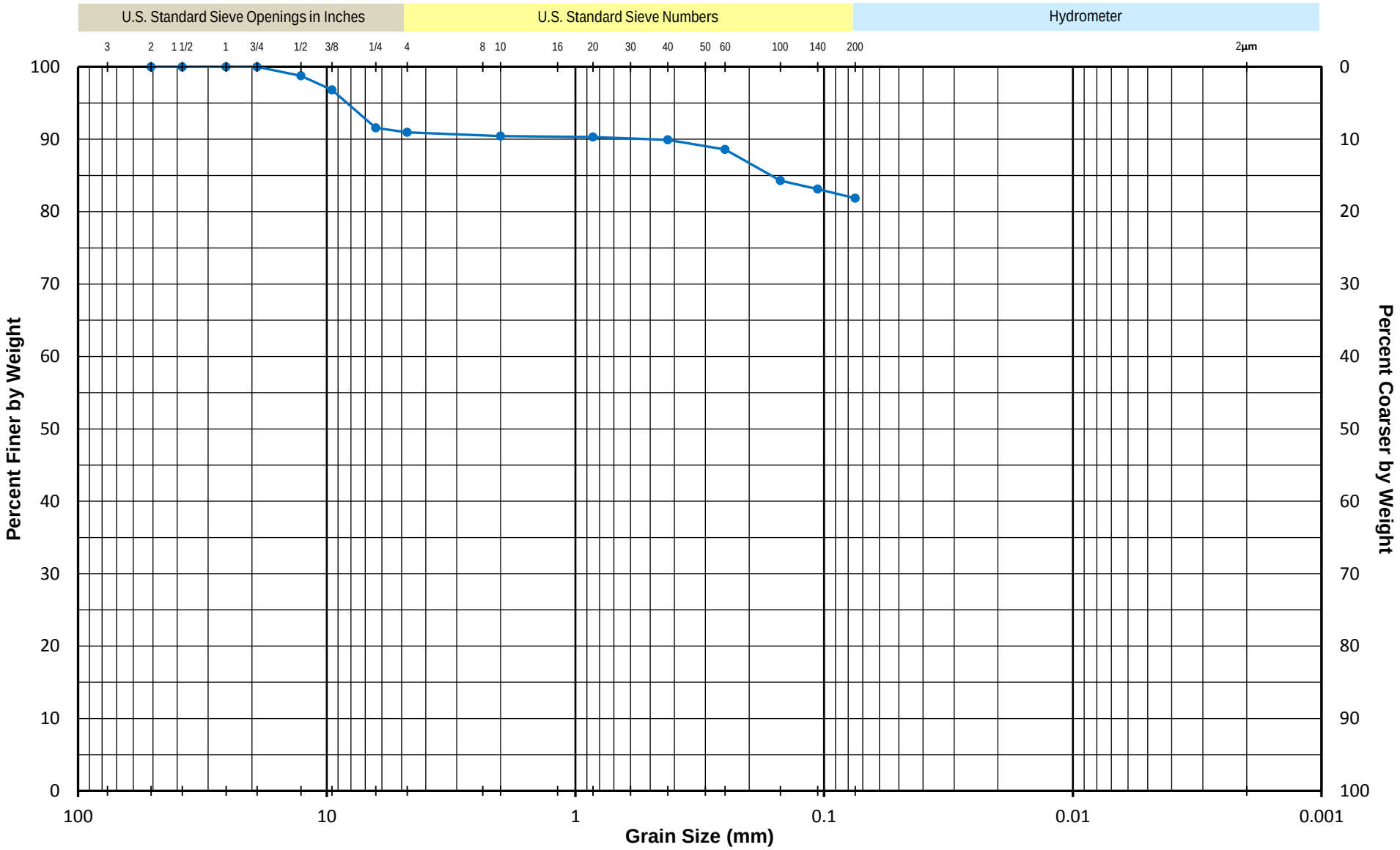


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC42 2-4	Black Fine to Medium Sand with clay seams and shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

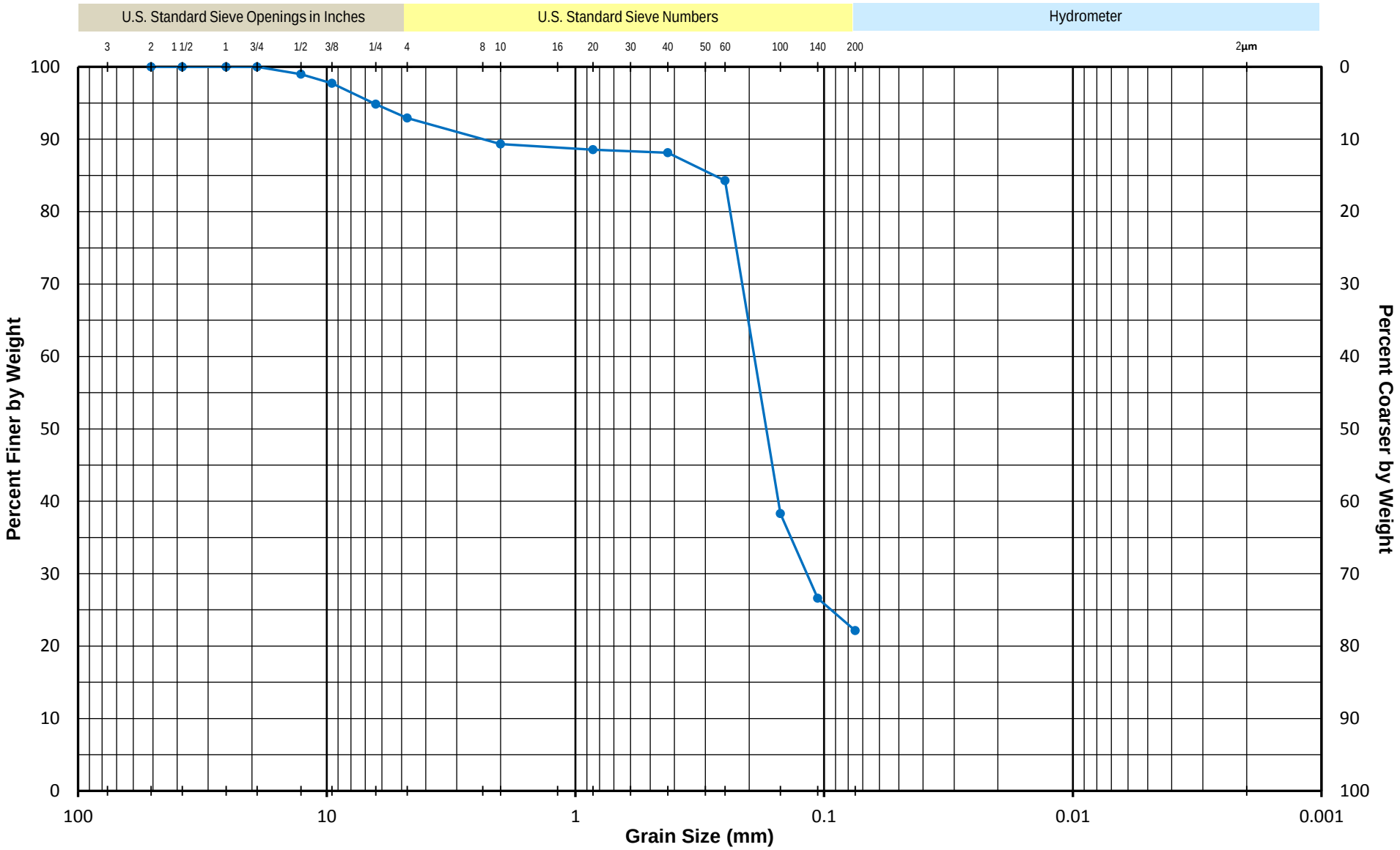


Test Depth	Core ID Section/Sample	Material Description
5.9 ft	VC42 4-6	Black Clay with sand and shells

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

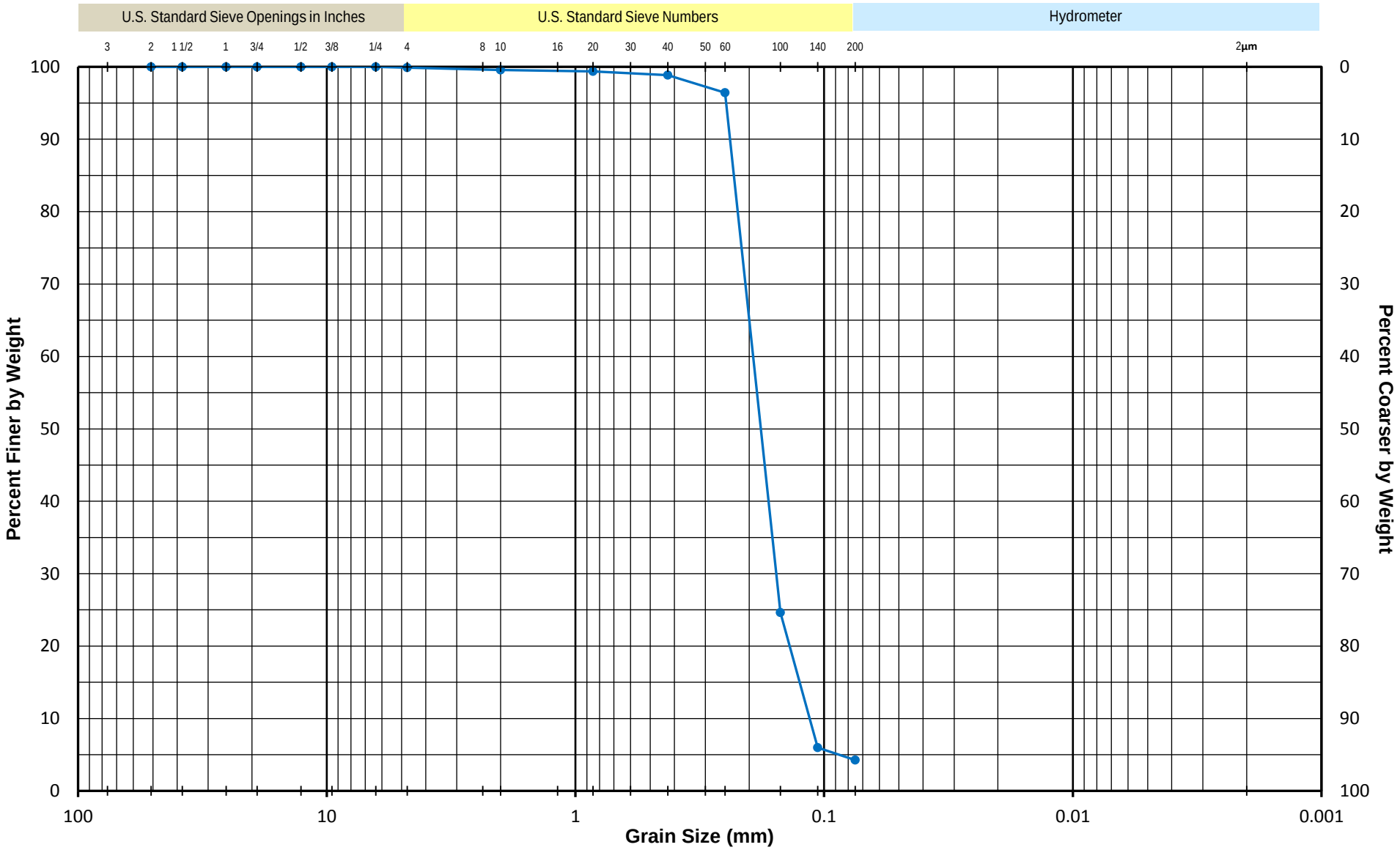


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC42 6-8	Black Fine Sand with clay pockets and shells

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

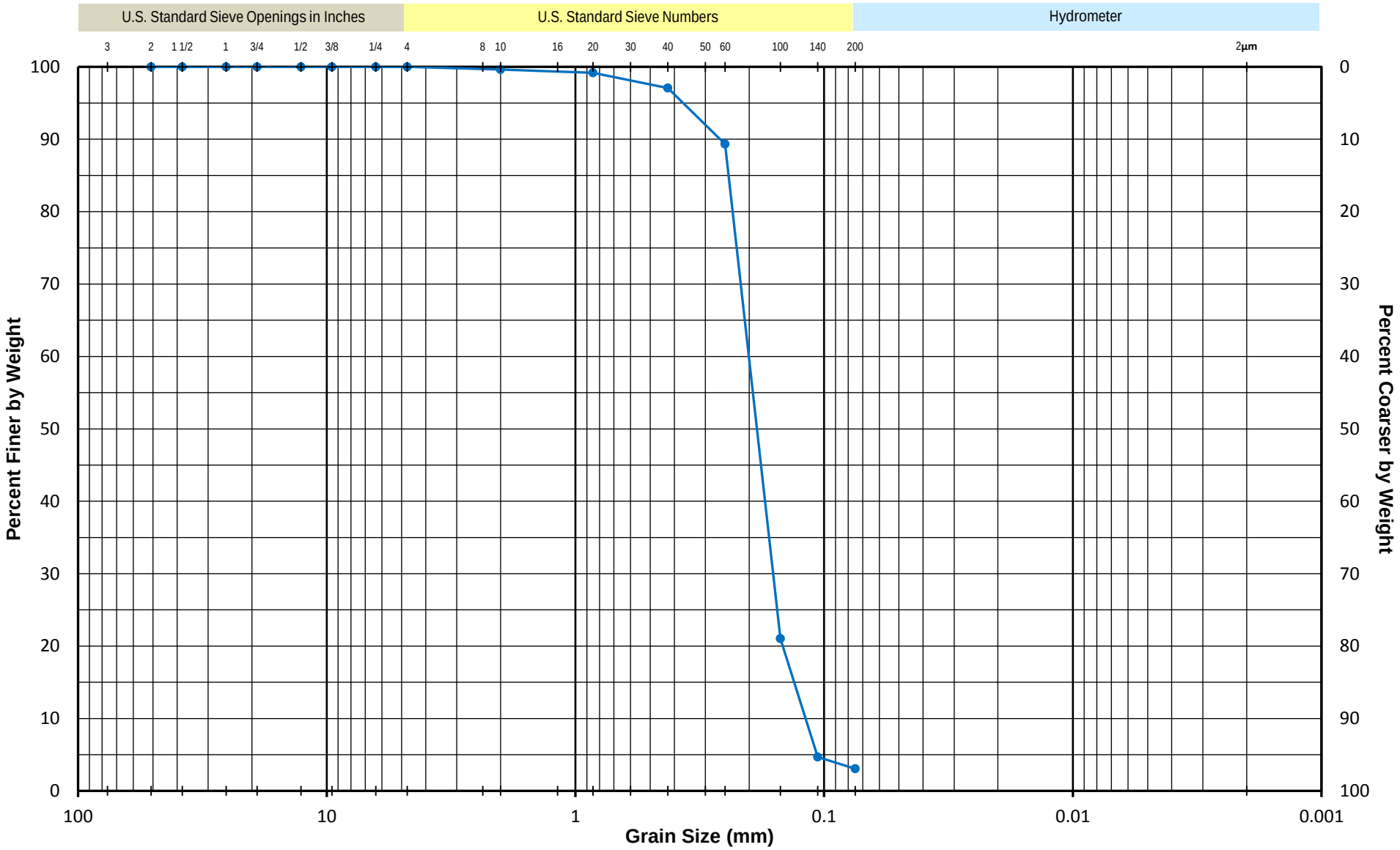


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC42 8-10	Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

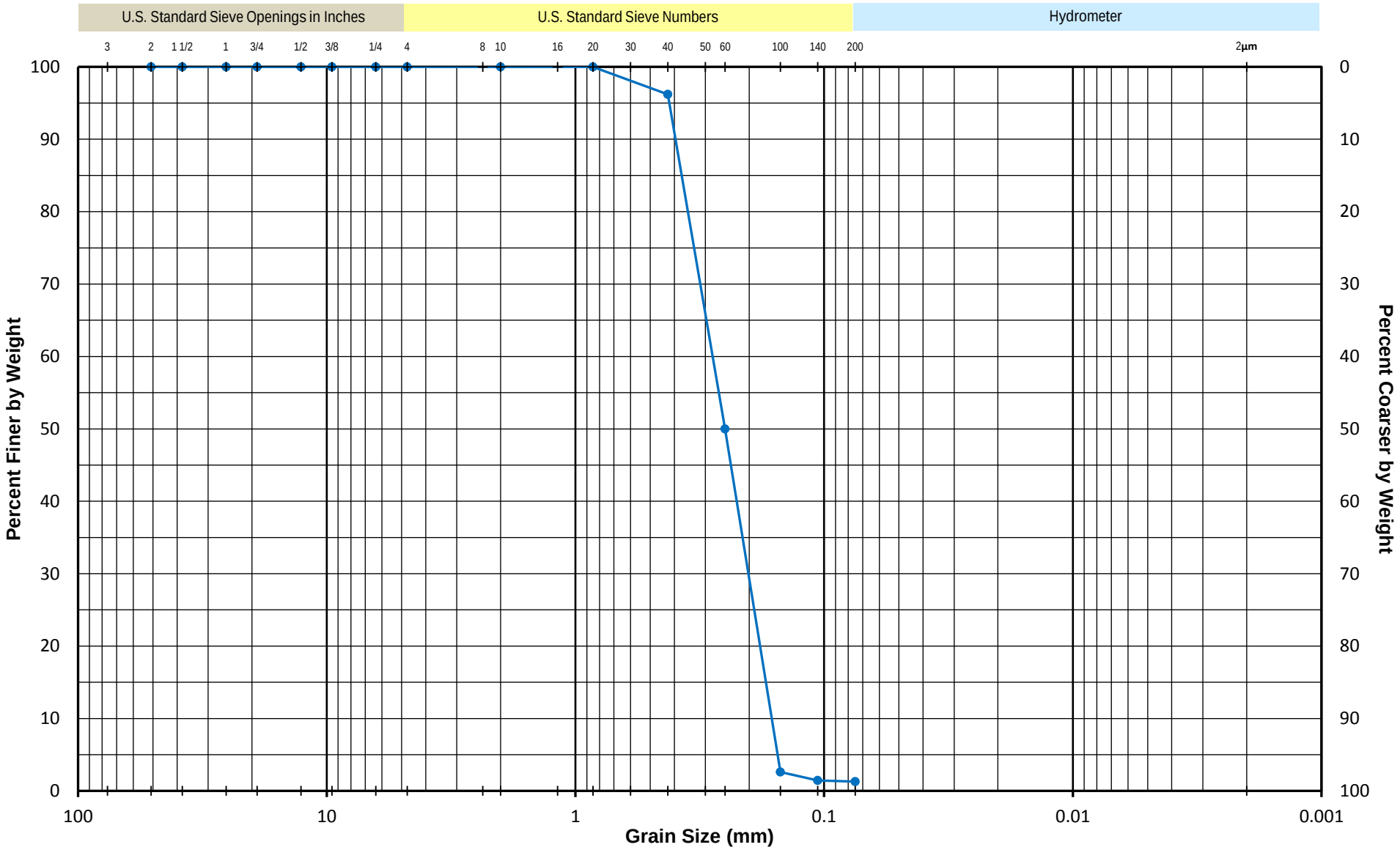


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC42 10-12	Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

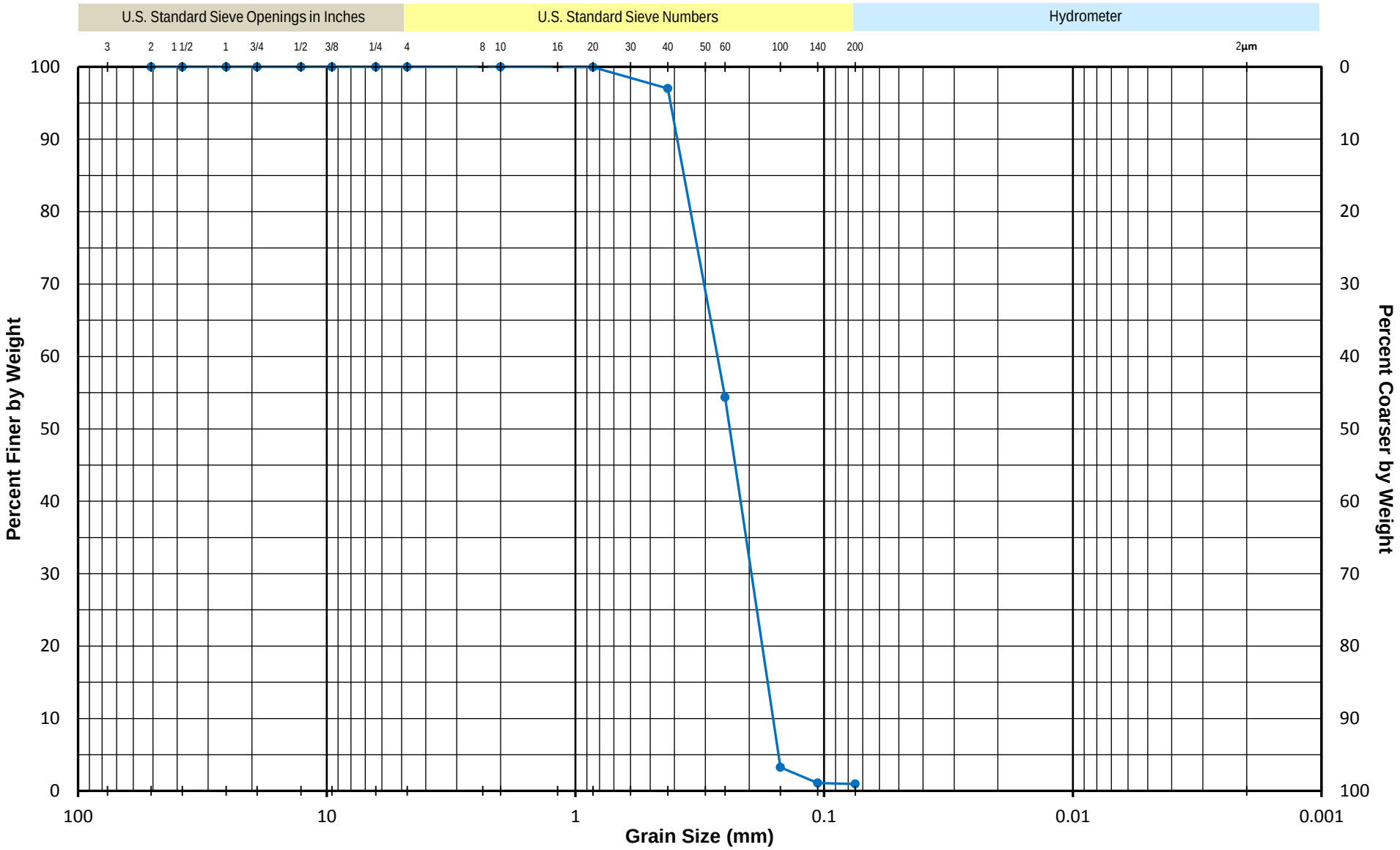


Test Depth	Core ID Section/Sample	Material Description
0.5 ft	VC43 0-2	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

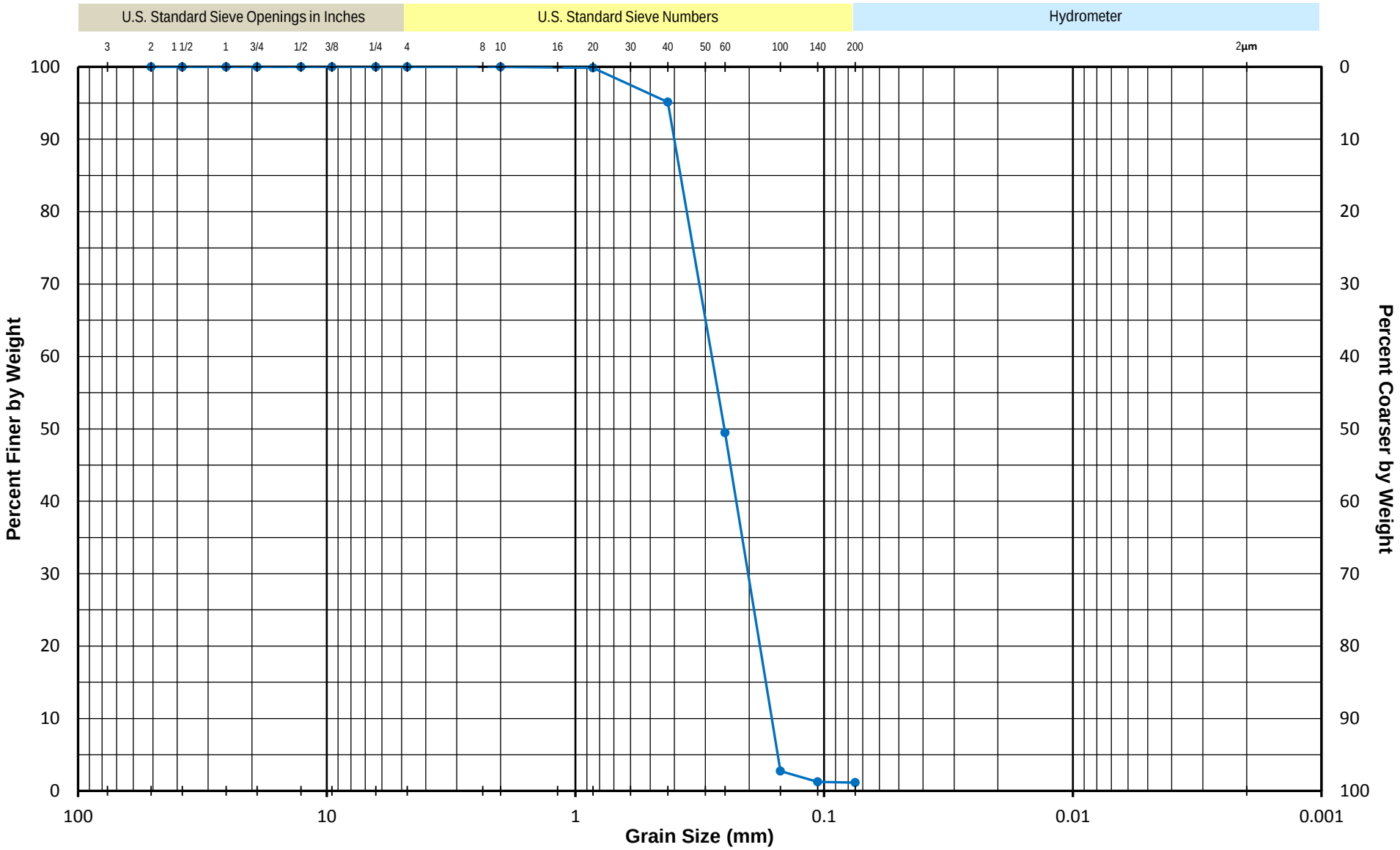


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC43 0-2	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

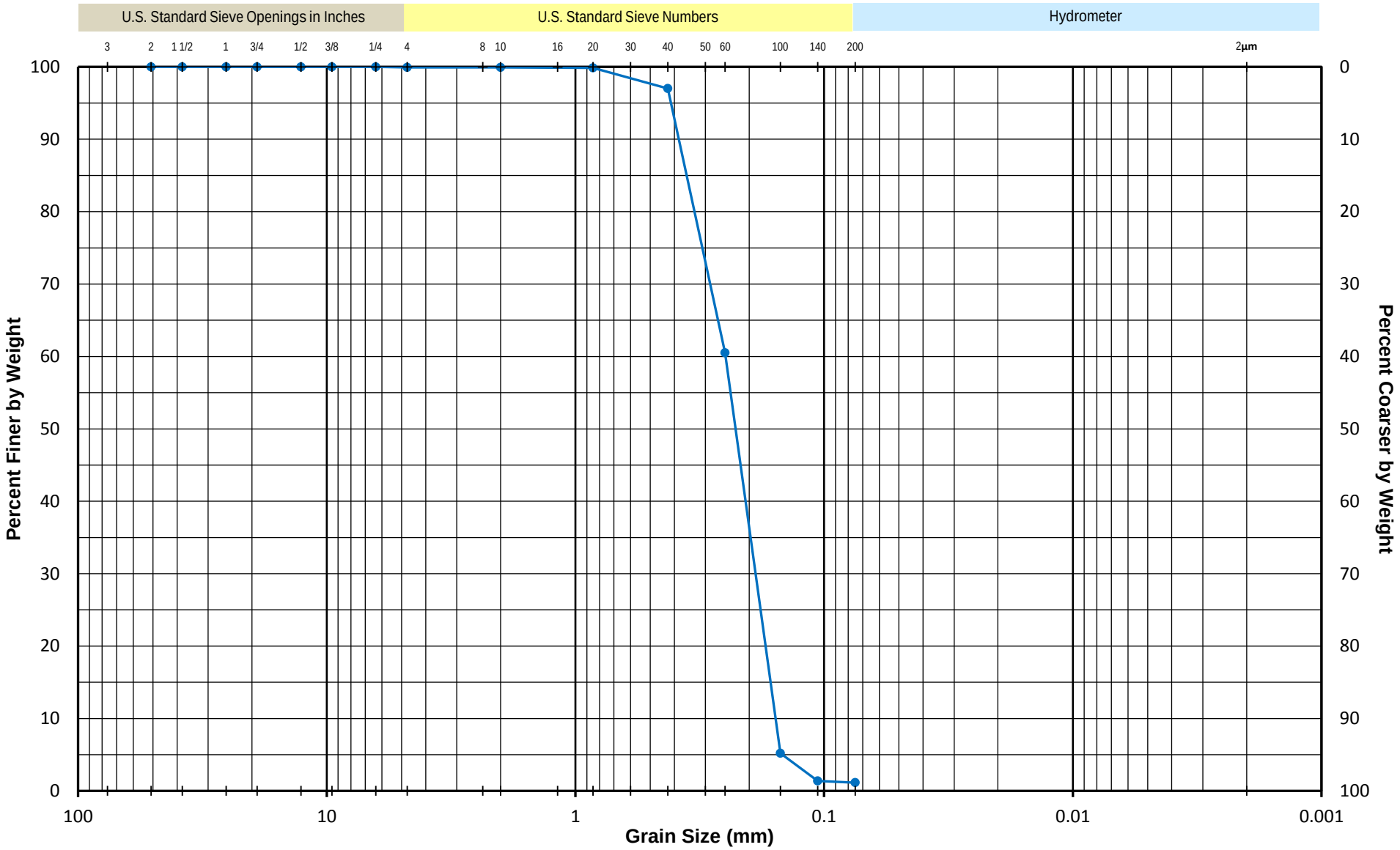


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC43 2-4	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

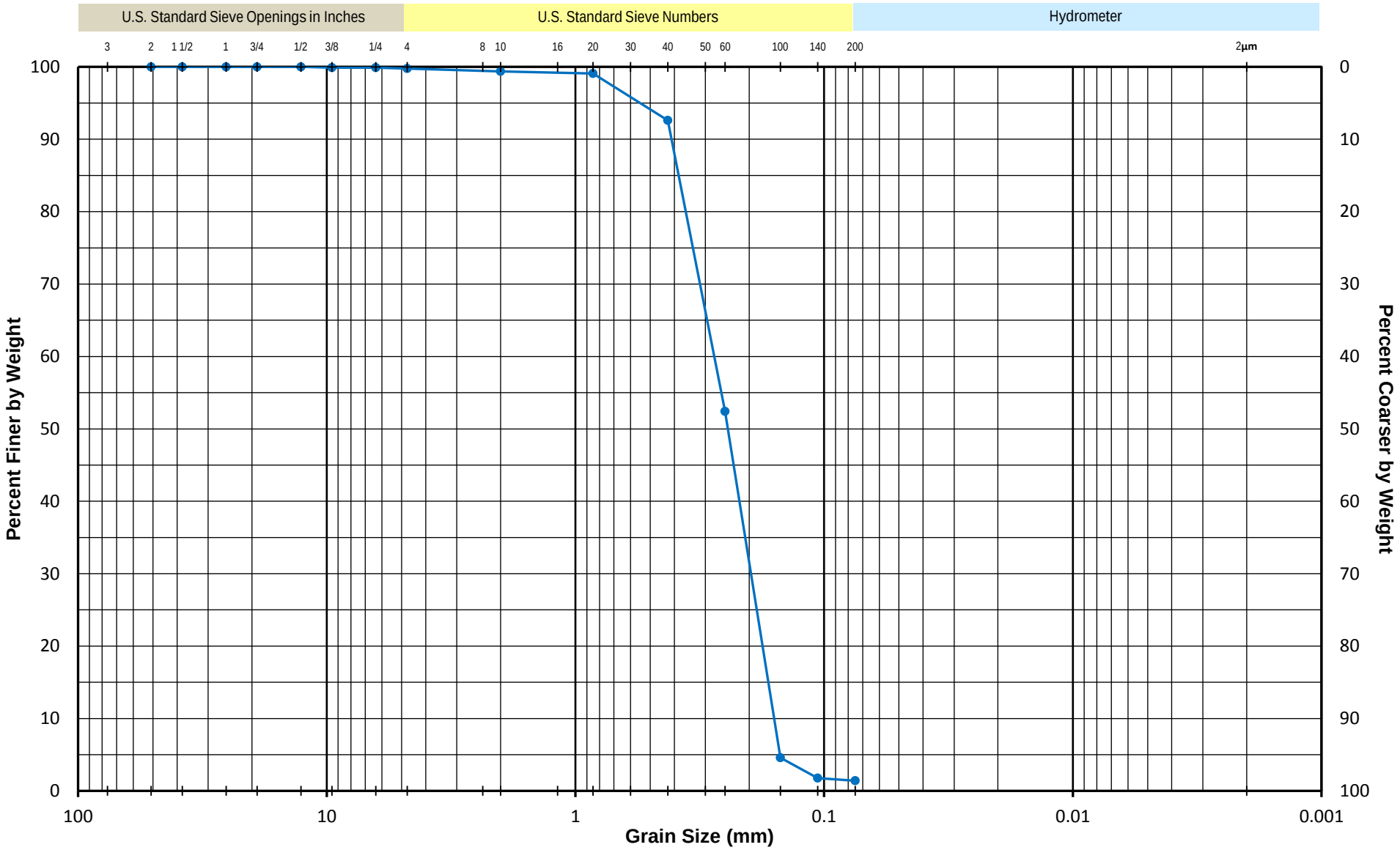


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC43 4-6	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

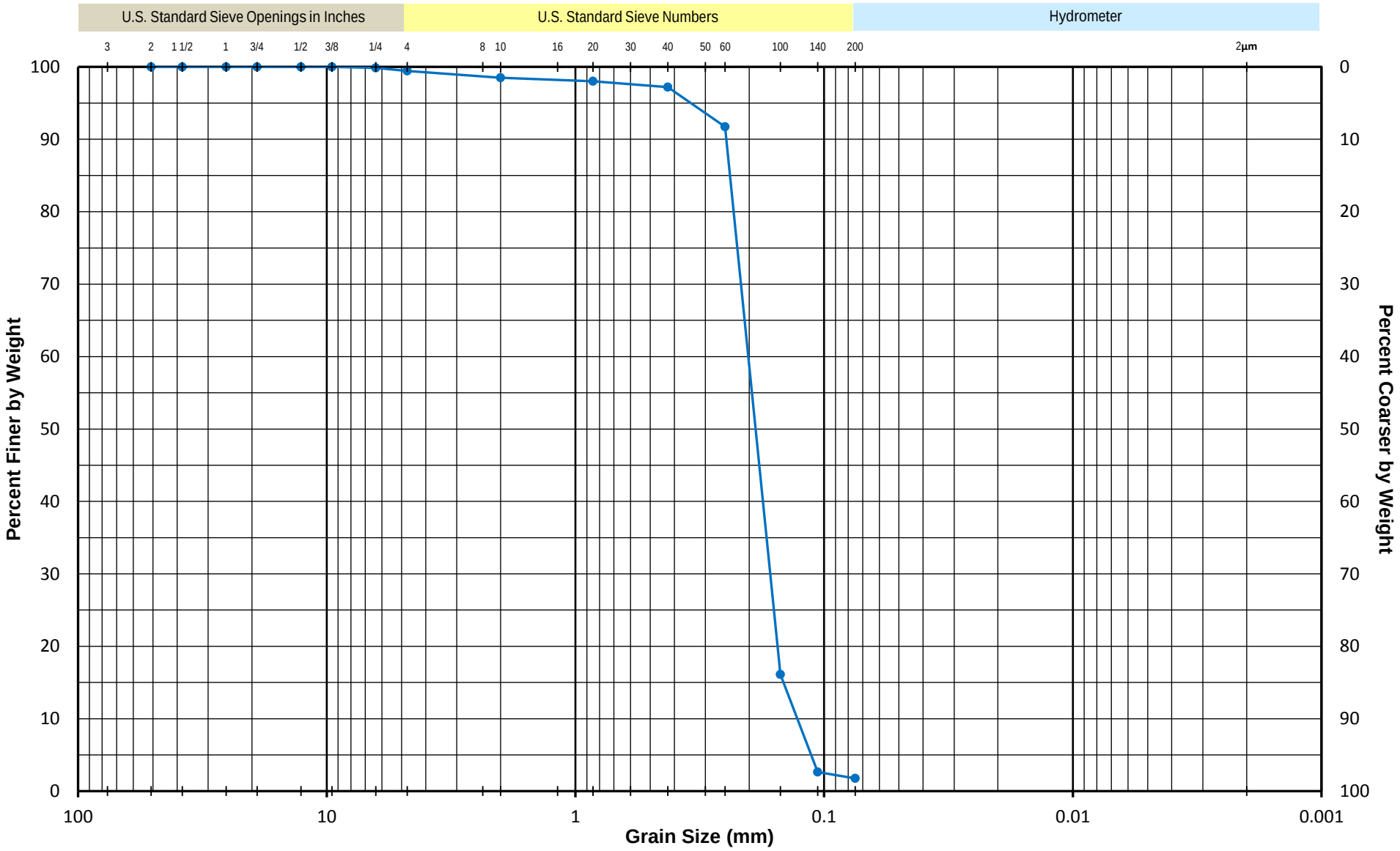


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC43 6-8	Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

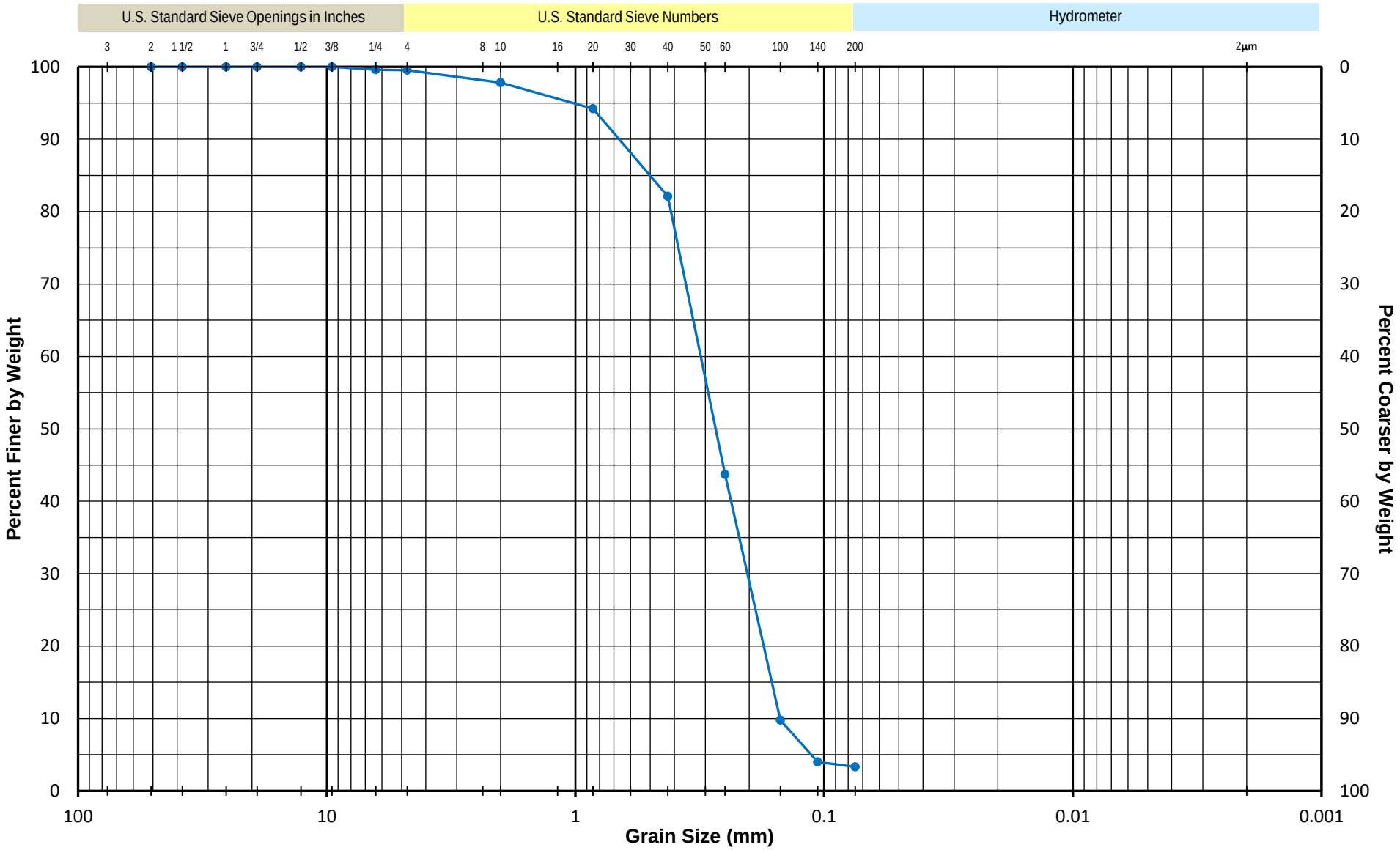


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC43 8-10	Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

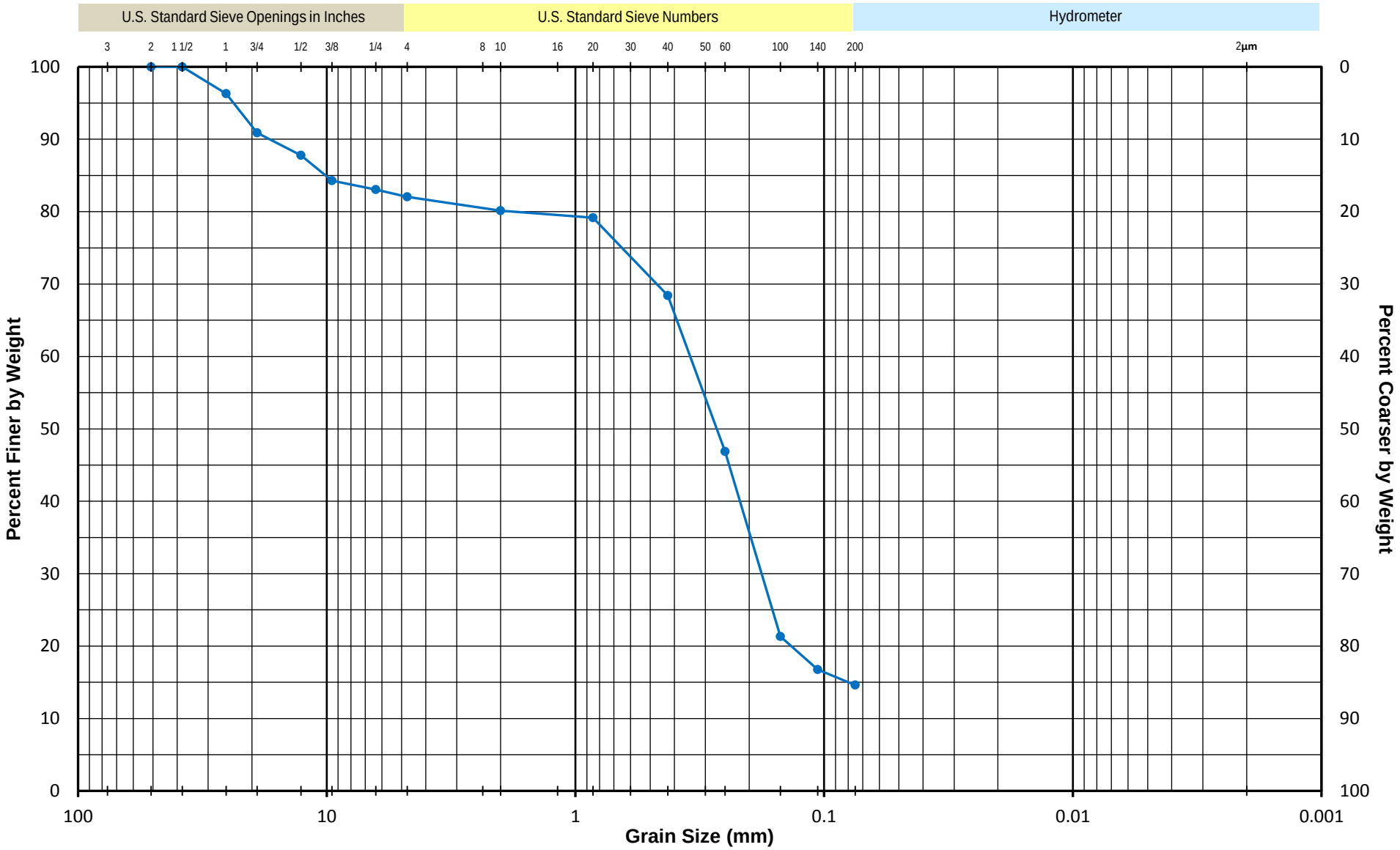


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC43 10-12	Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

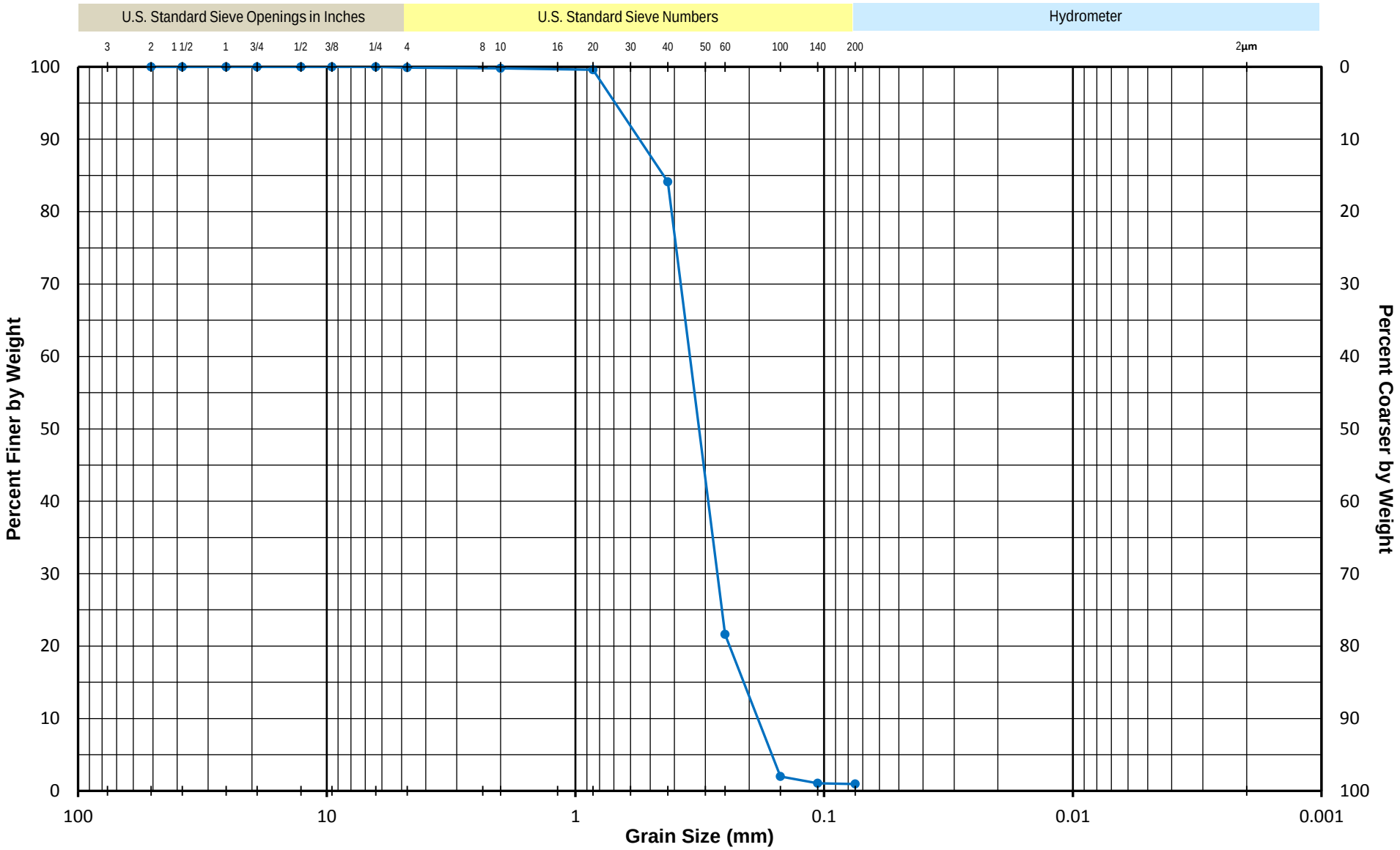


Test Depth	Core ID Section/Sample	Material Description
0.5 ft	VC46 0-2	Dark Olive Gray Clayey Sand with large shells

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

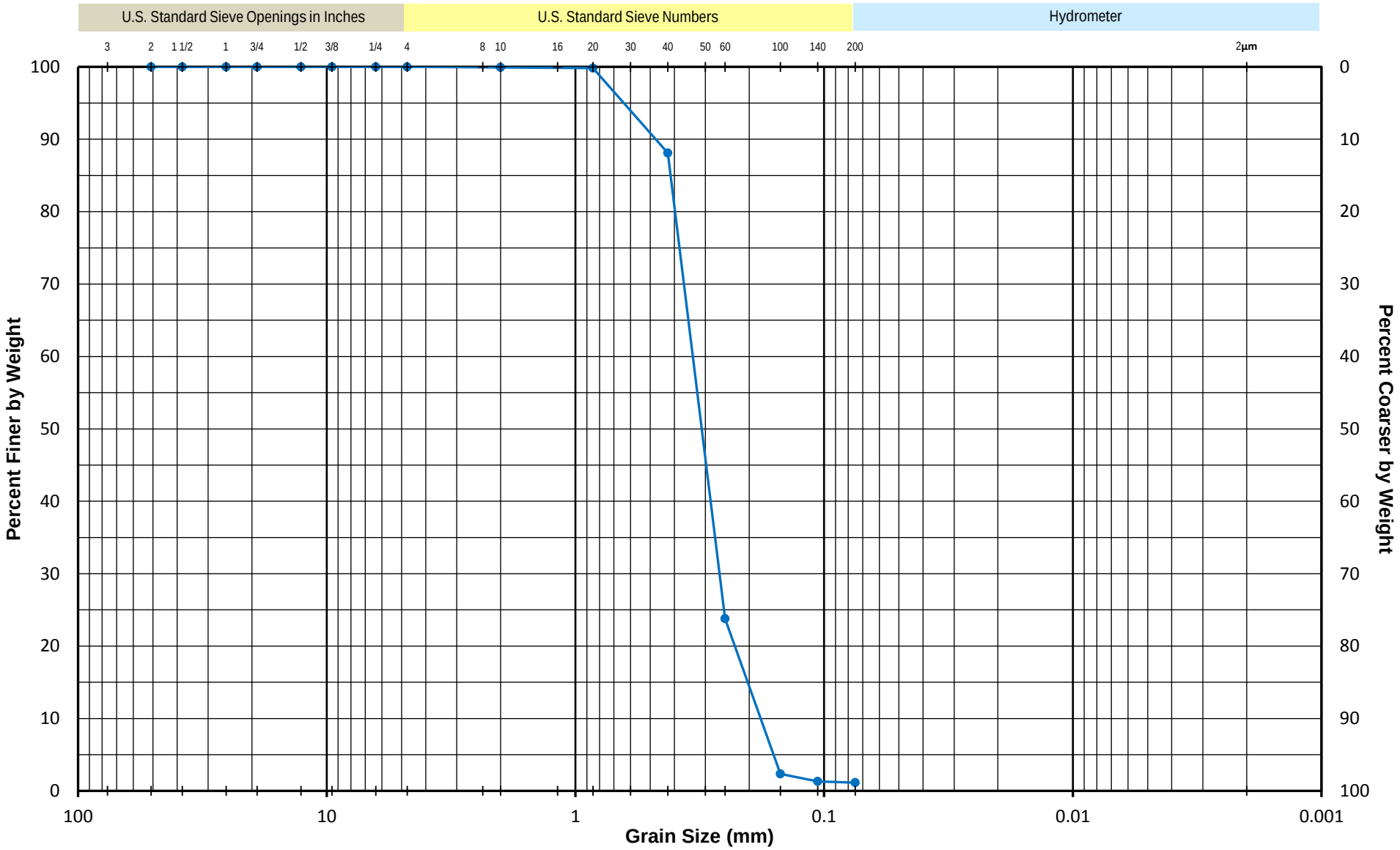


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC46 0-2	Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

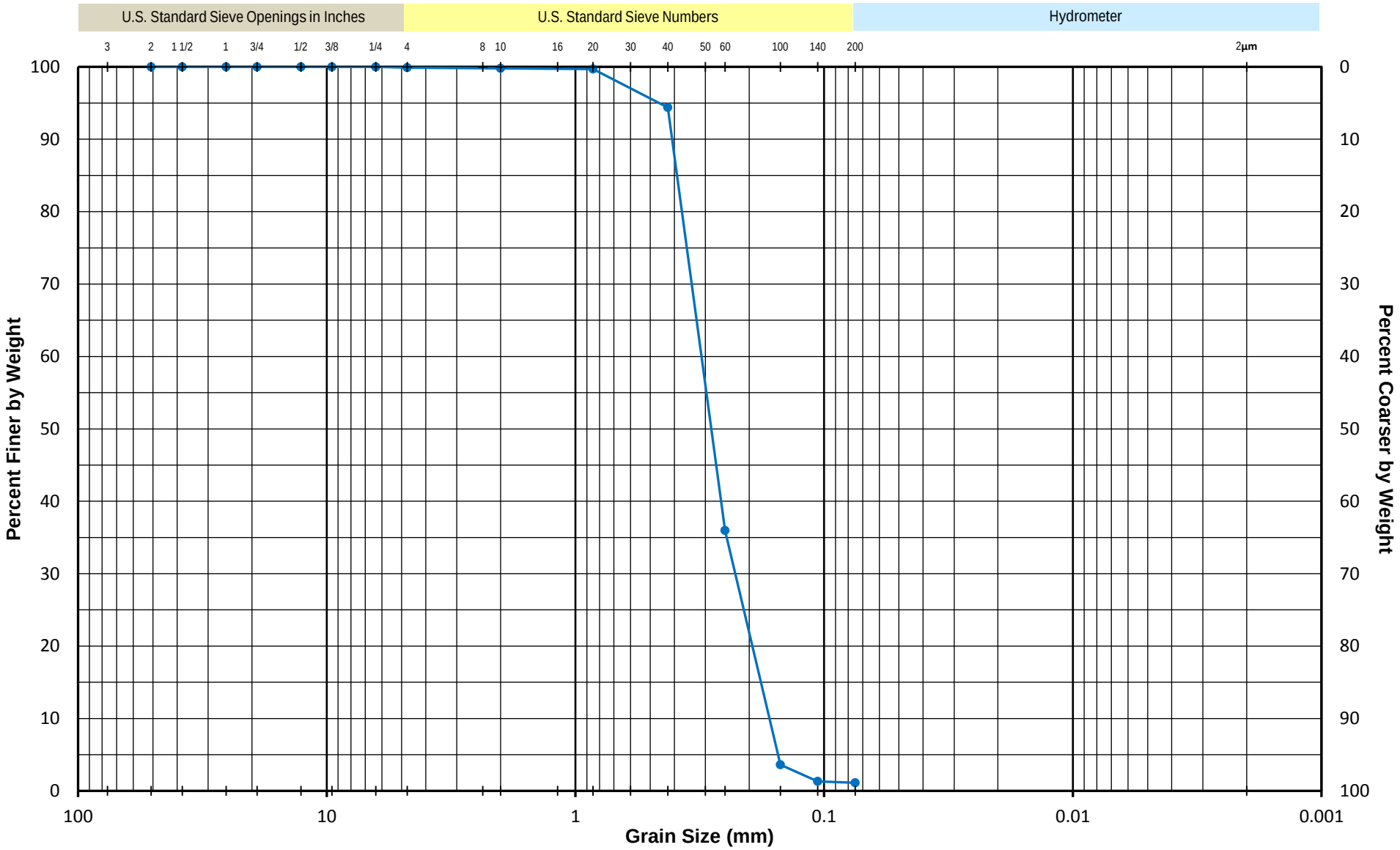


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC46 2-4	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

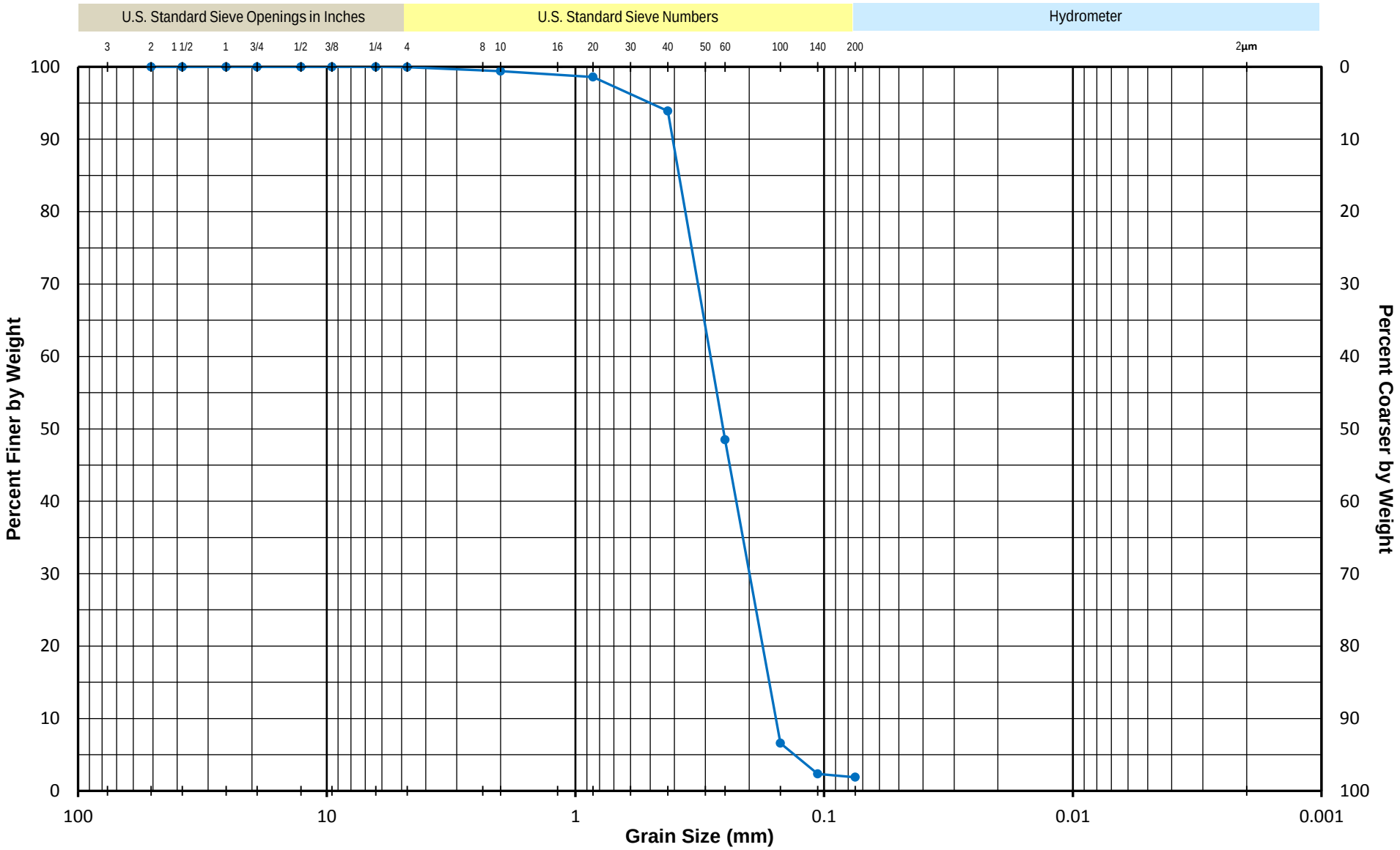


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC46 4-6	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

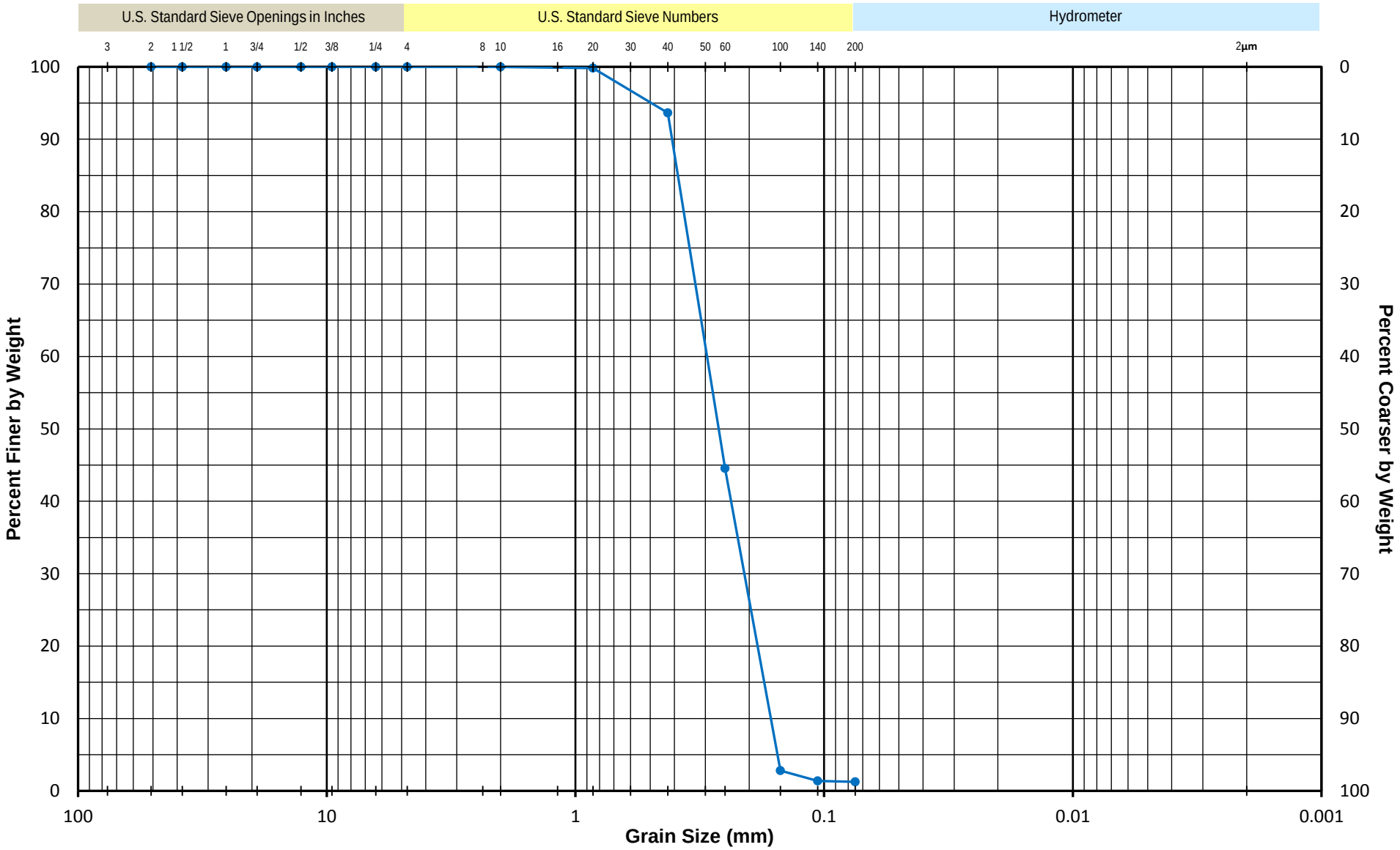


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC46 6-8	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

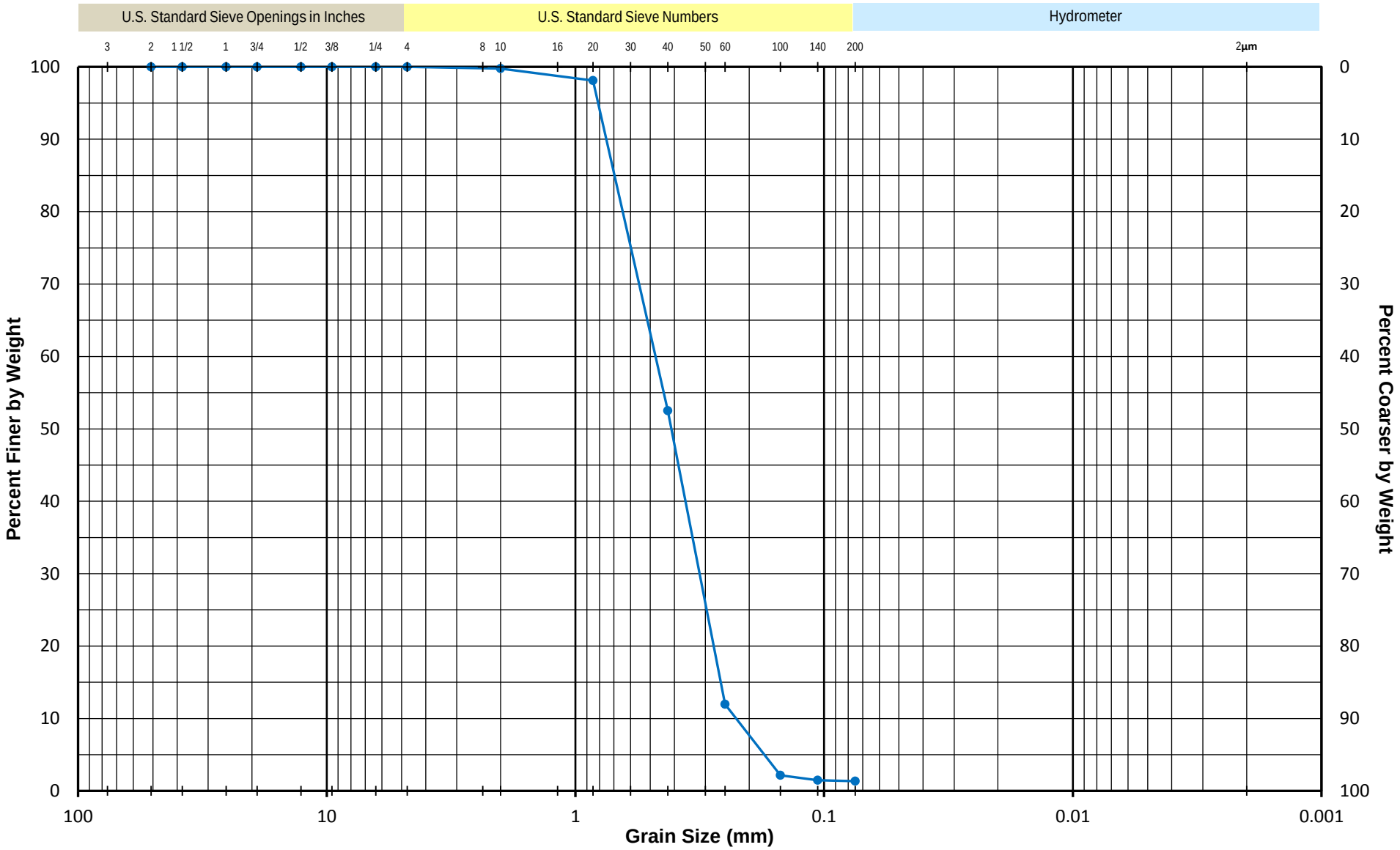


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC46 8-10	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

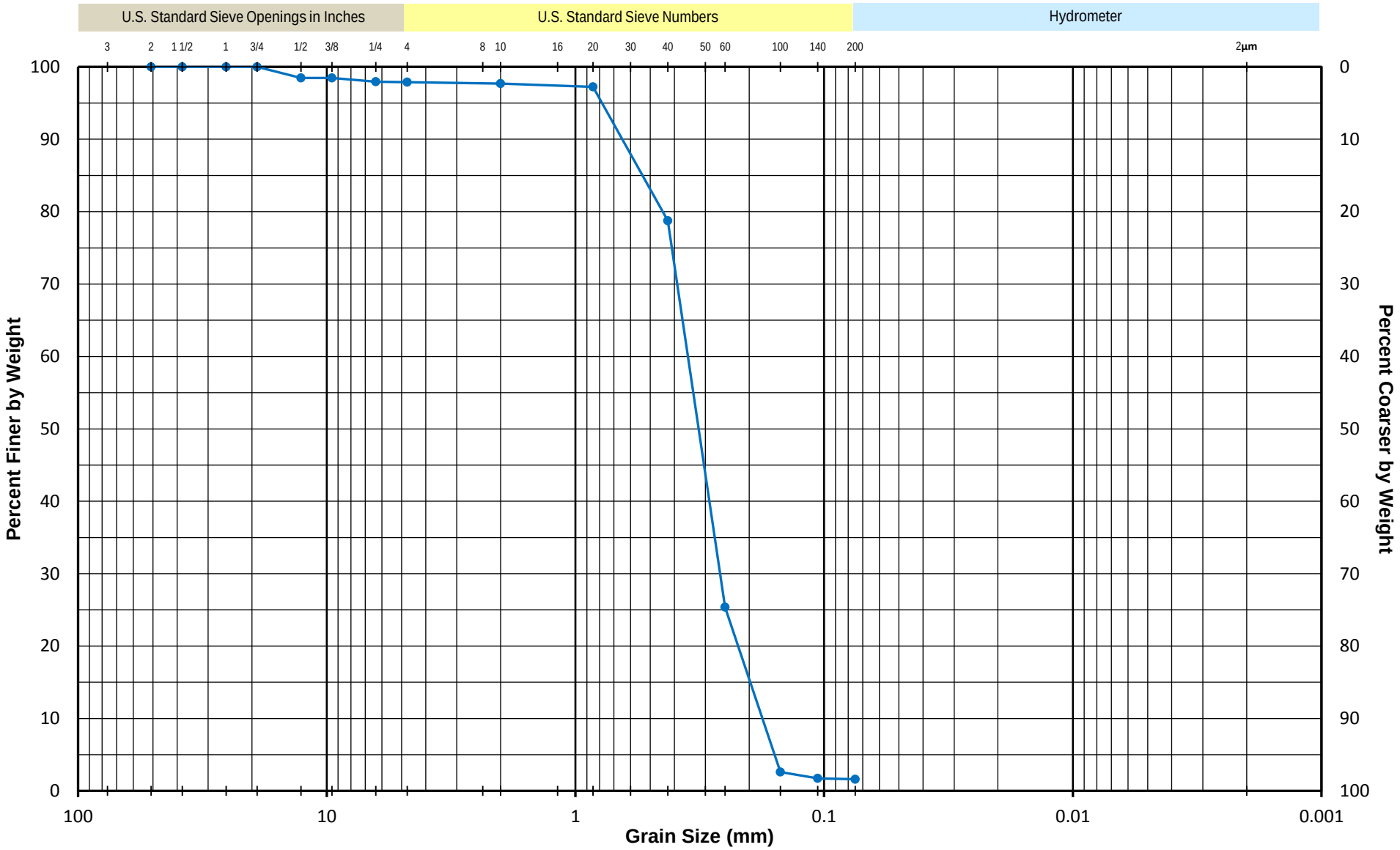


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC46 10-12	Dark Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

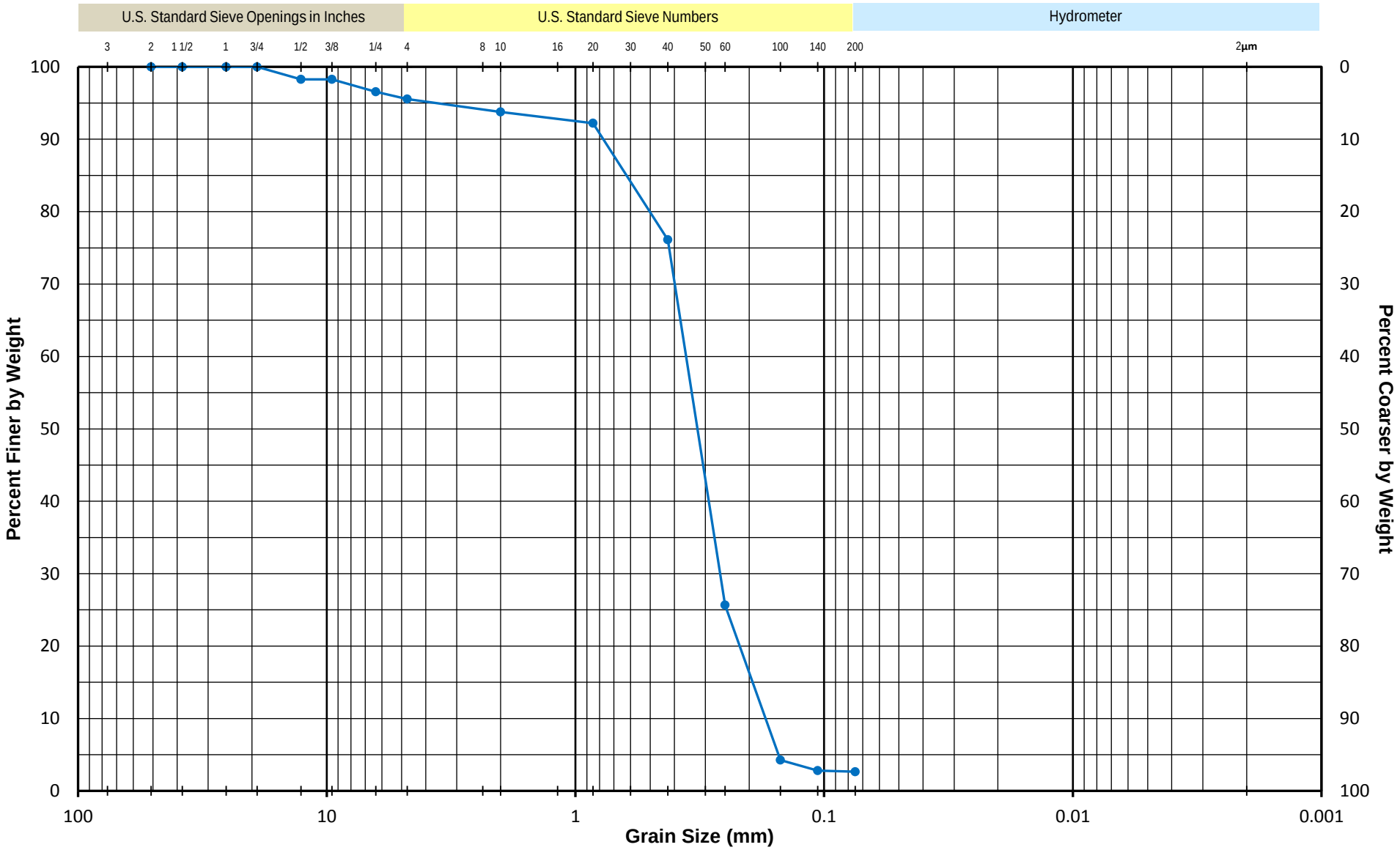


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC47 0-2	Black Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

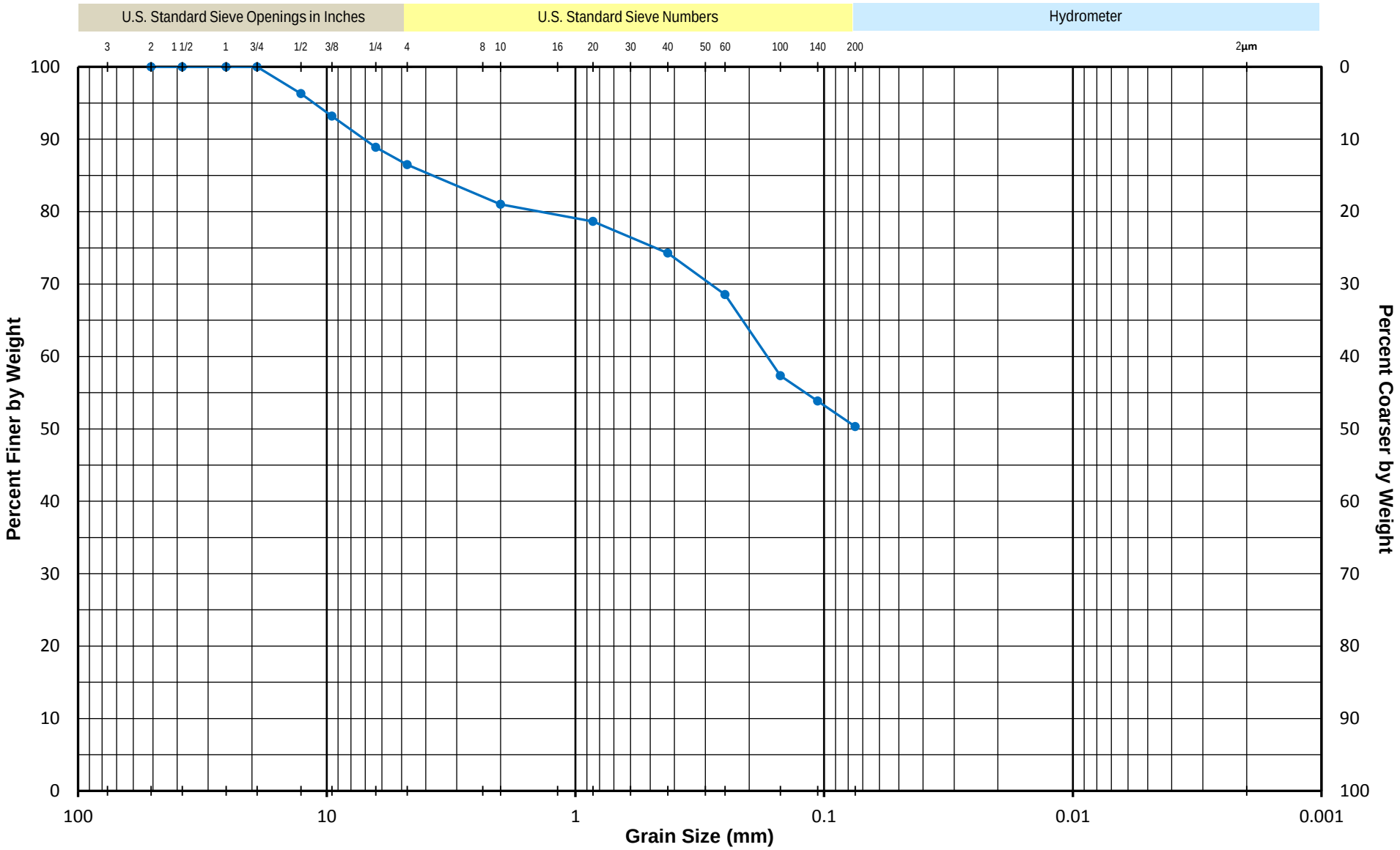


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC47 0-2	Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

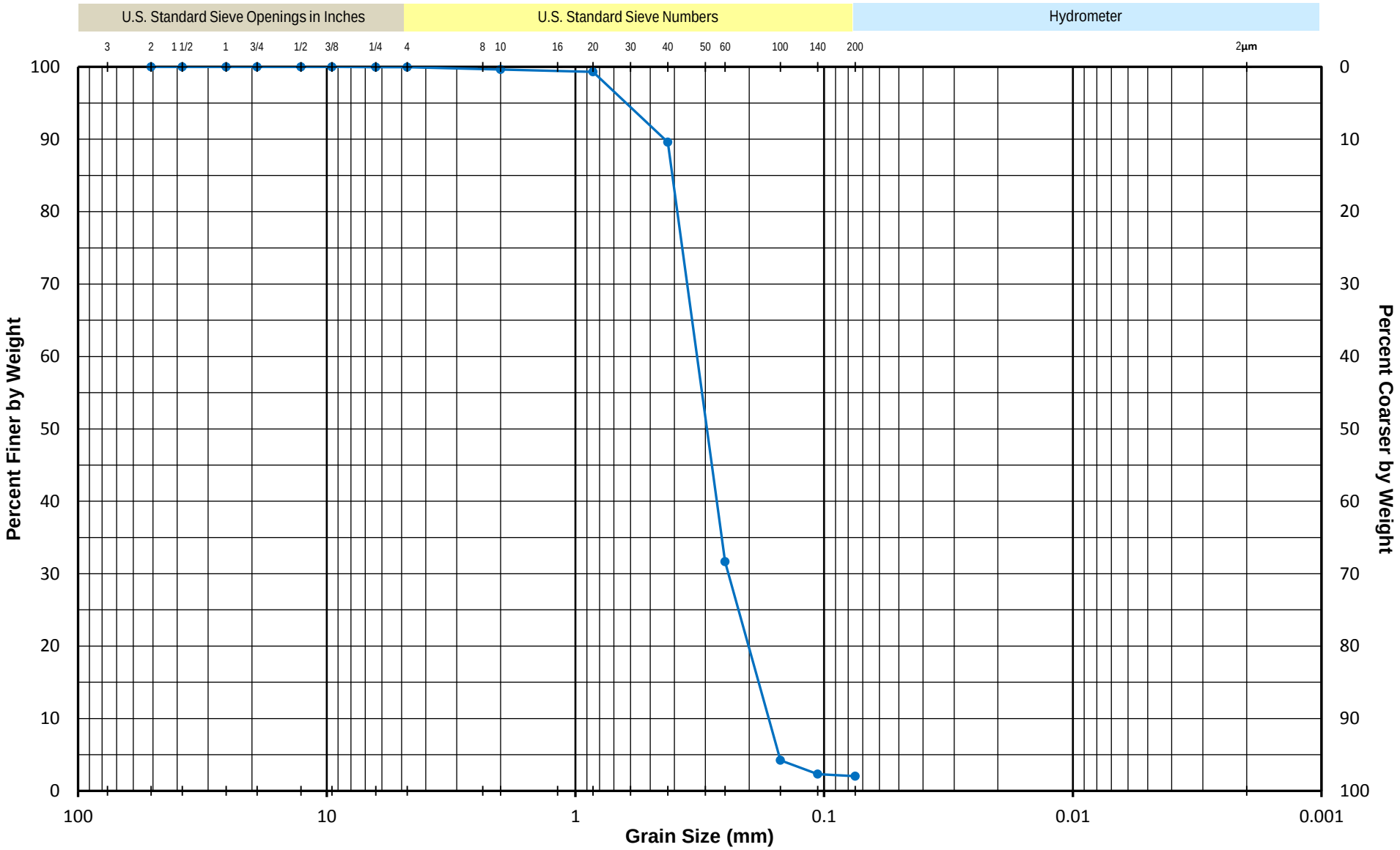


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC47 2-4	Black Sandy Clay with shell, shell fragments, and organics

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

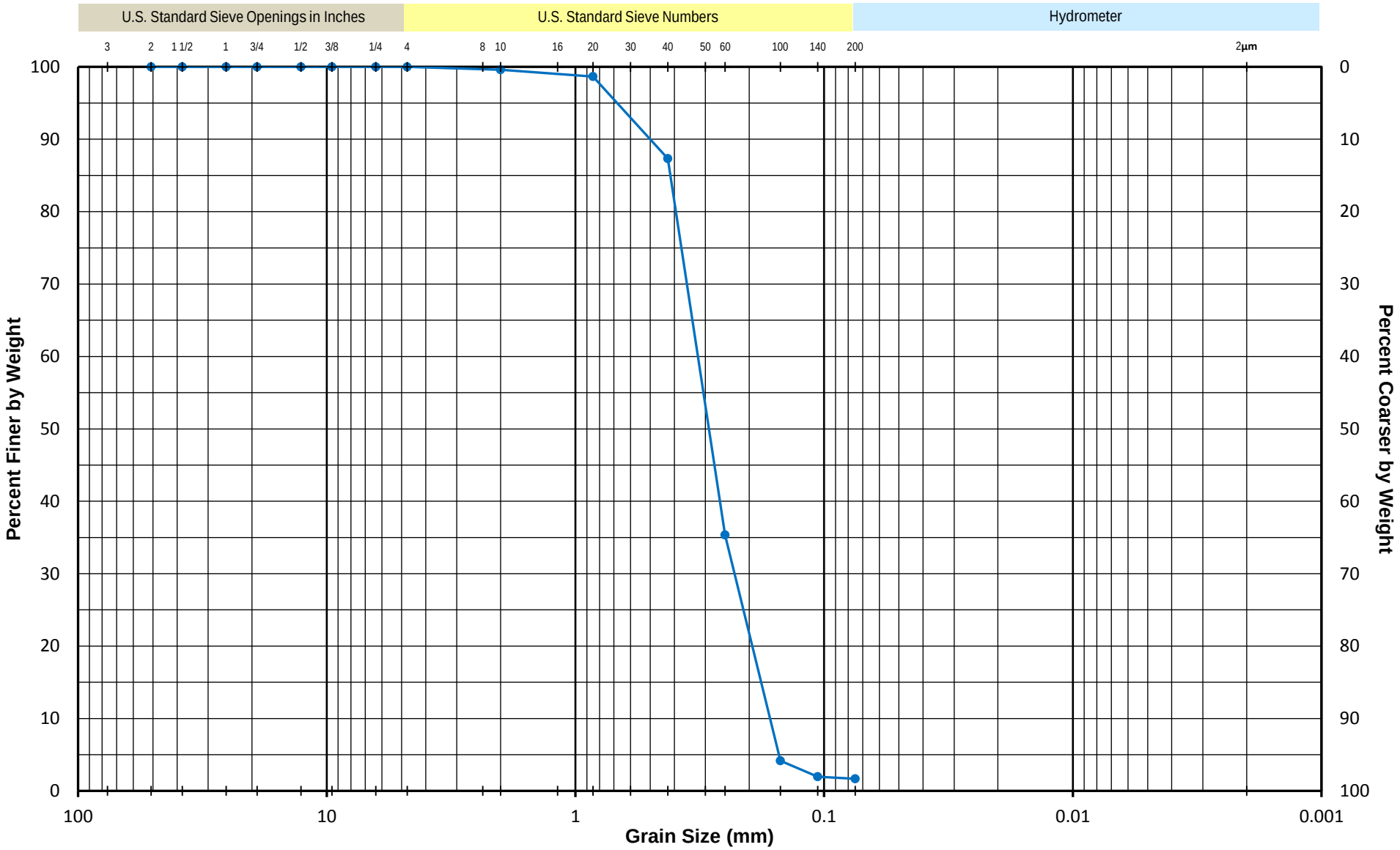


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC47 4-6	Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

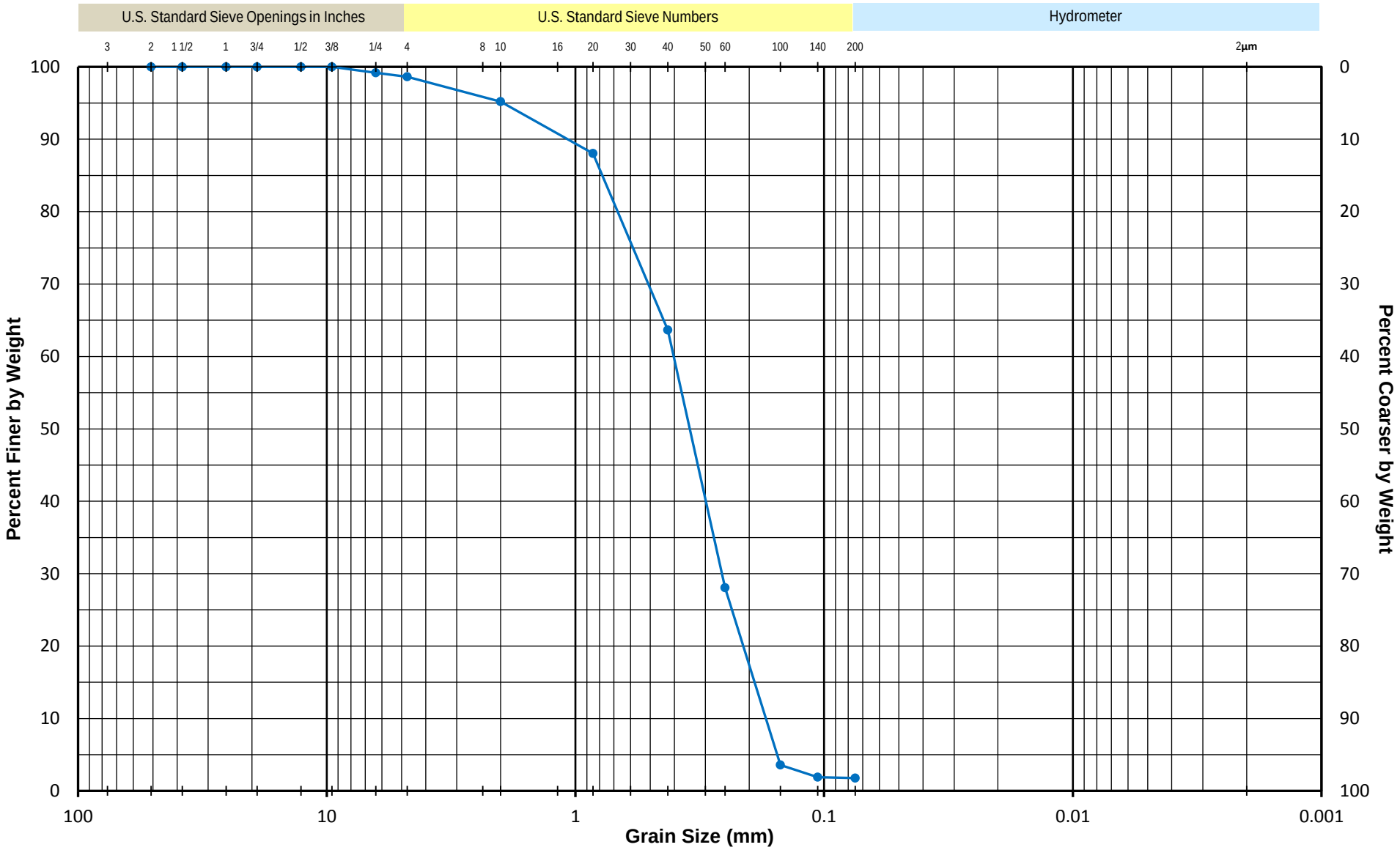


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC47 6-8	Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

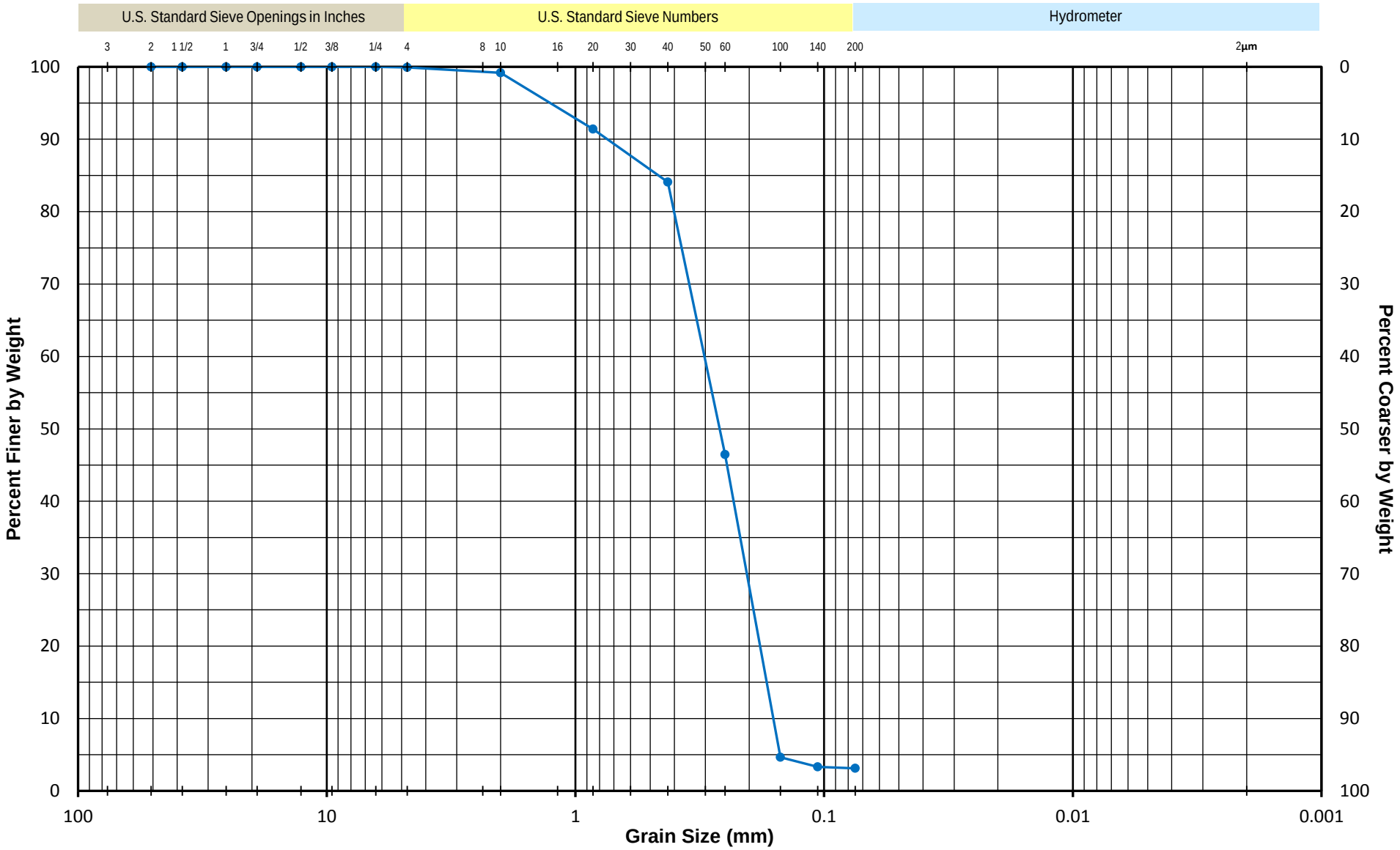


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC47 8-10	Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

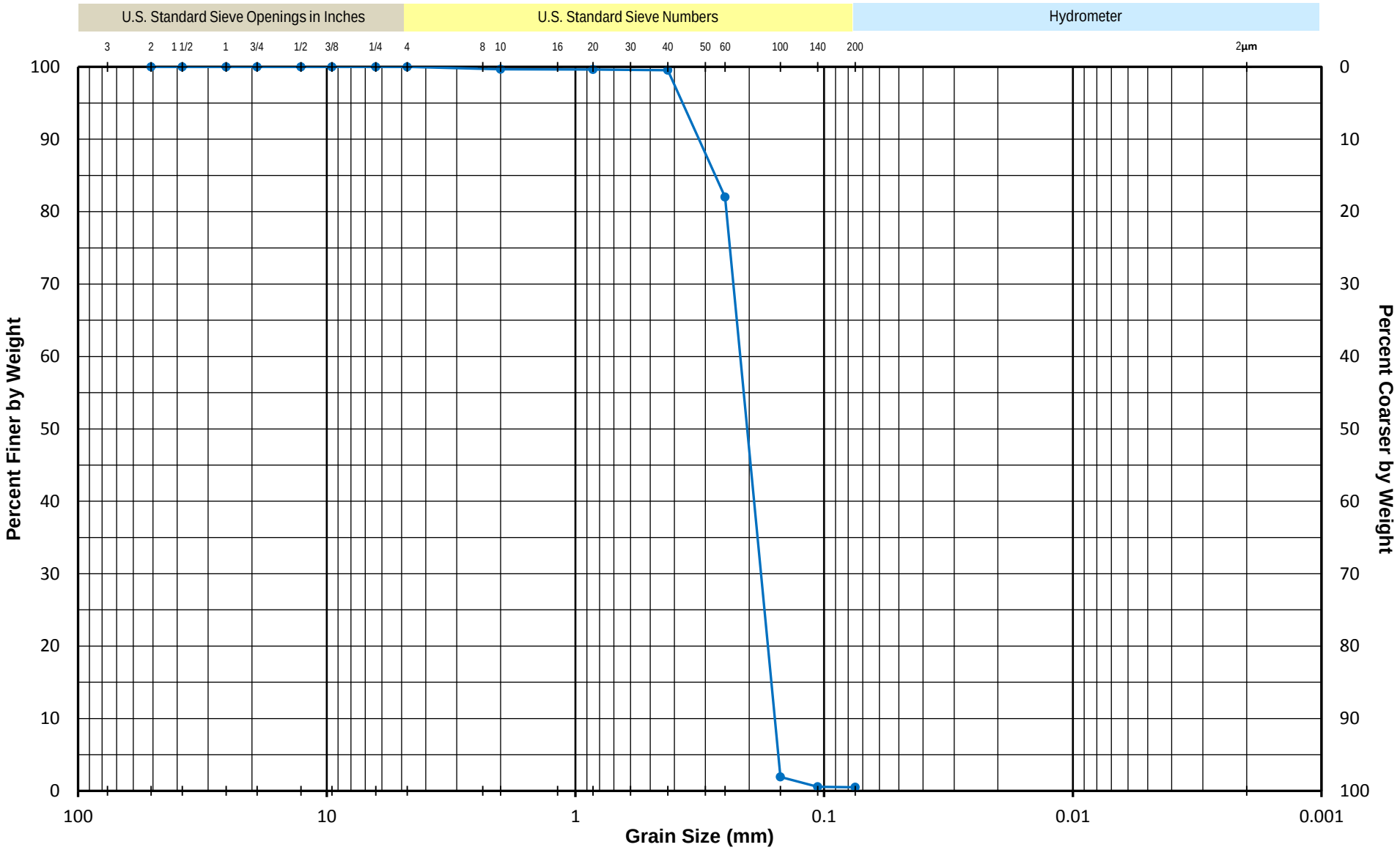


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC47 10-12	Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

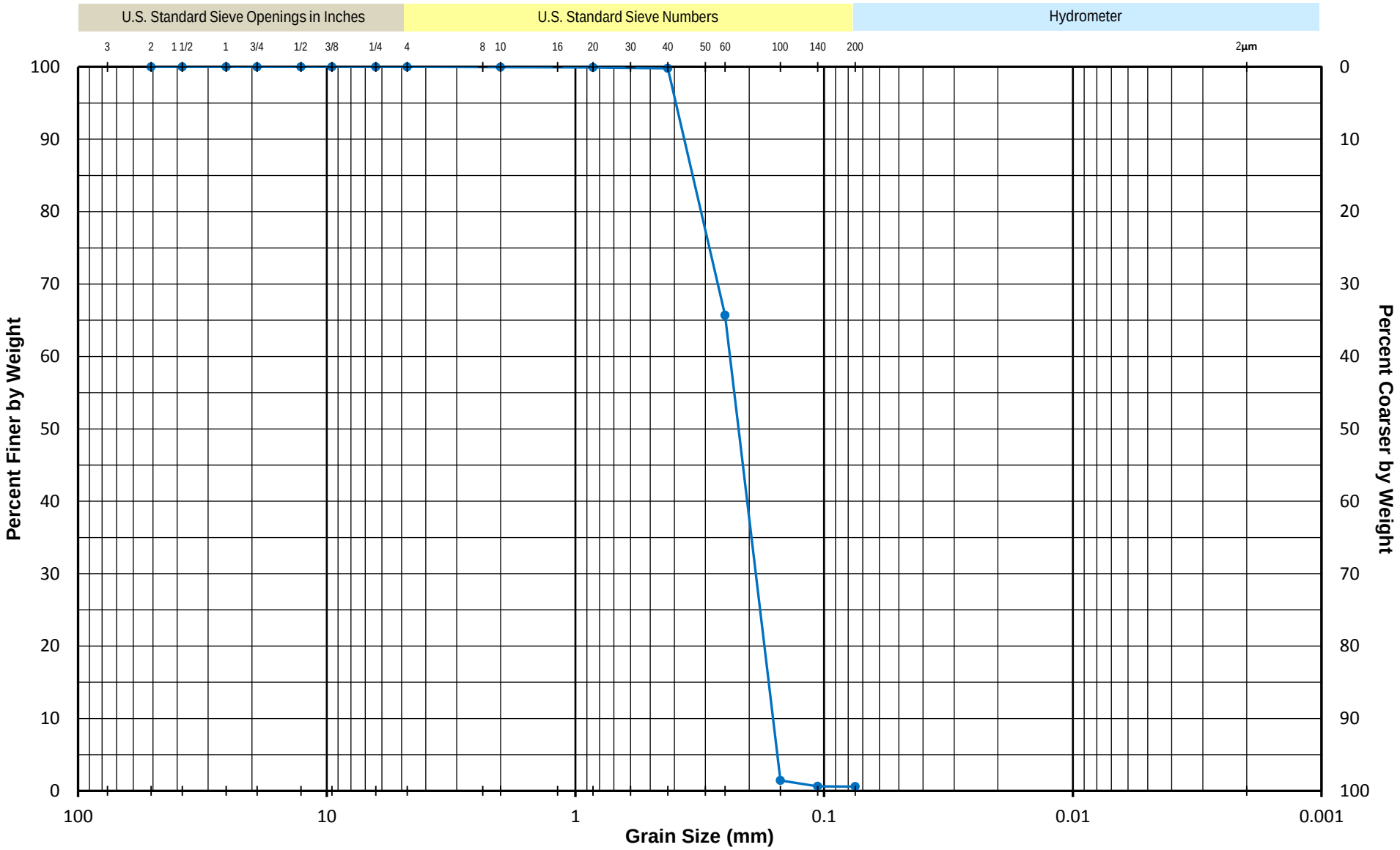


Test Depth	Core ID Section/Sample	Material Description
0.10 ft	VC49 0-2	Light Olive Brown Fine Sand with trace shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

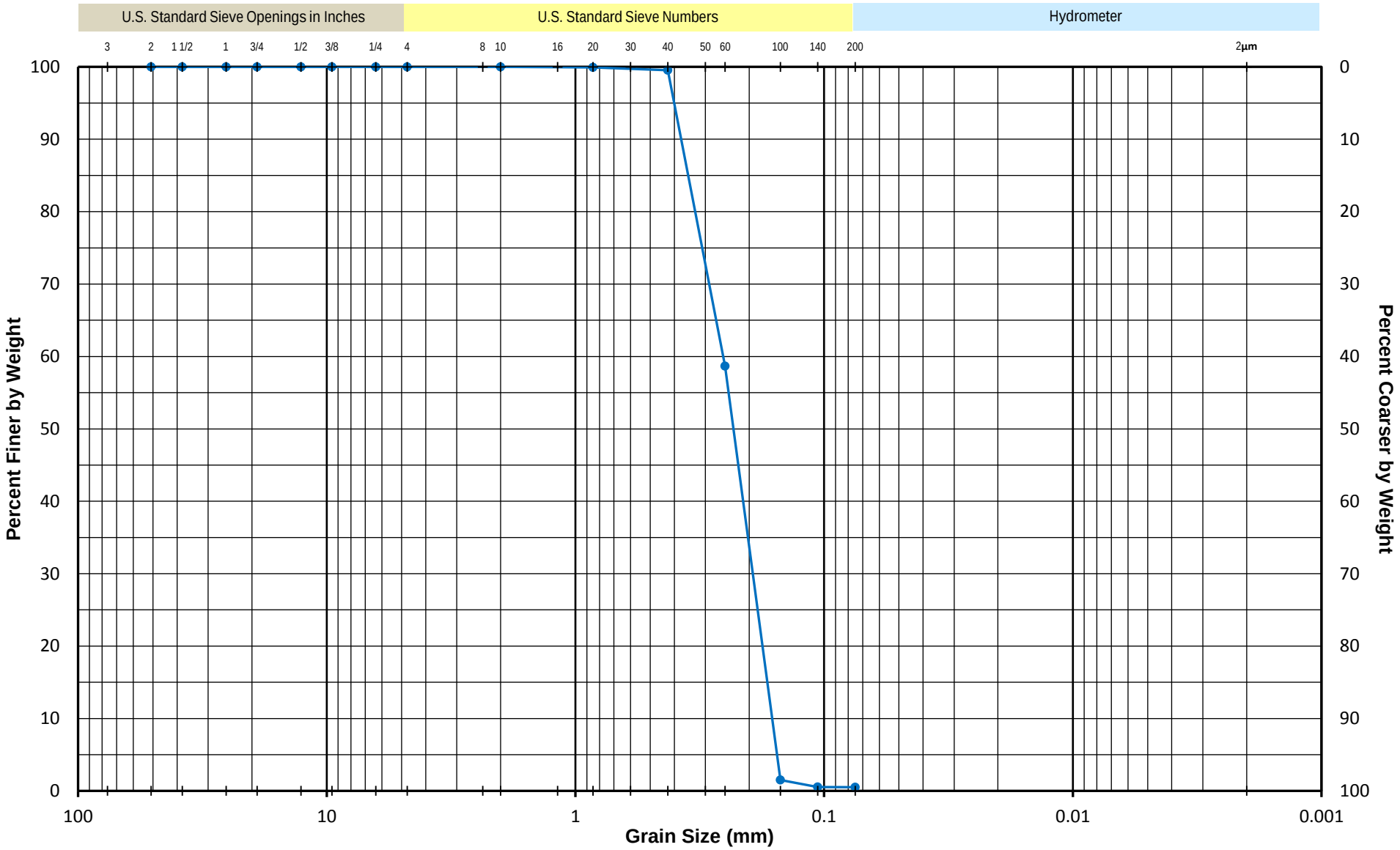


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC49 0-2	Grayish Brown Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

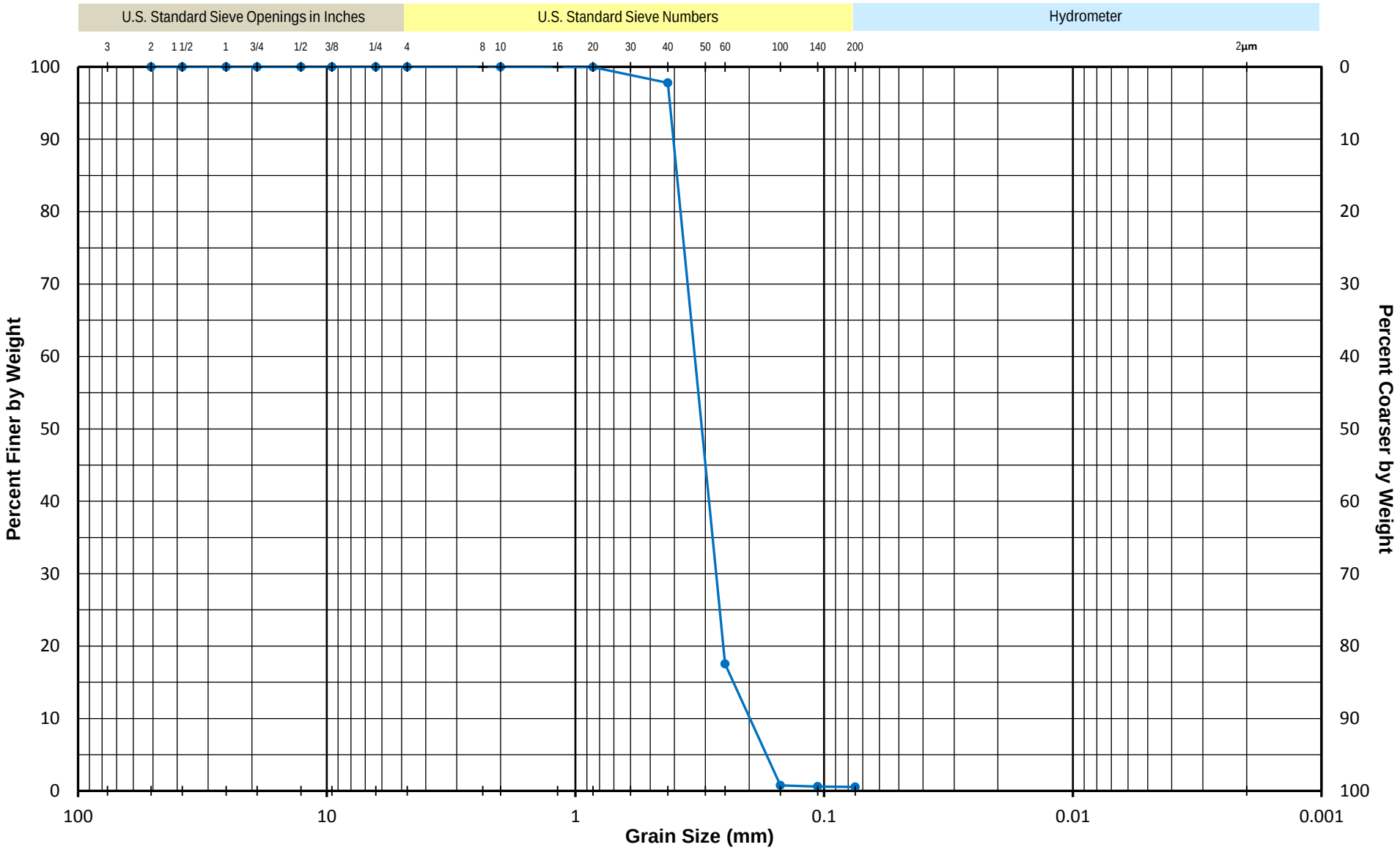


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC49 2-4	Grayish Brown Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

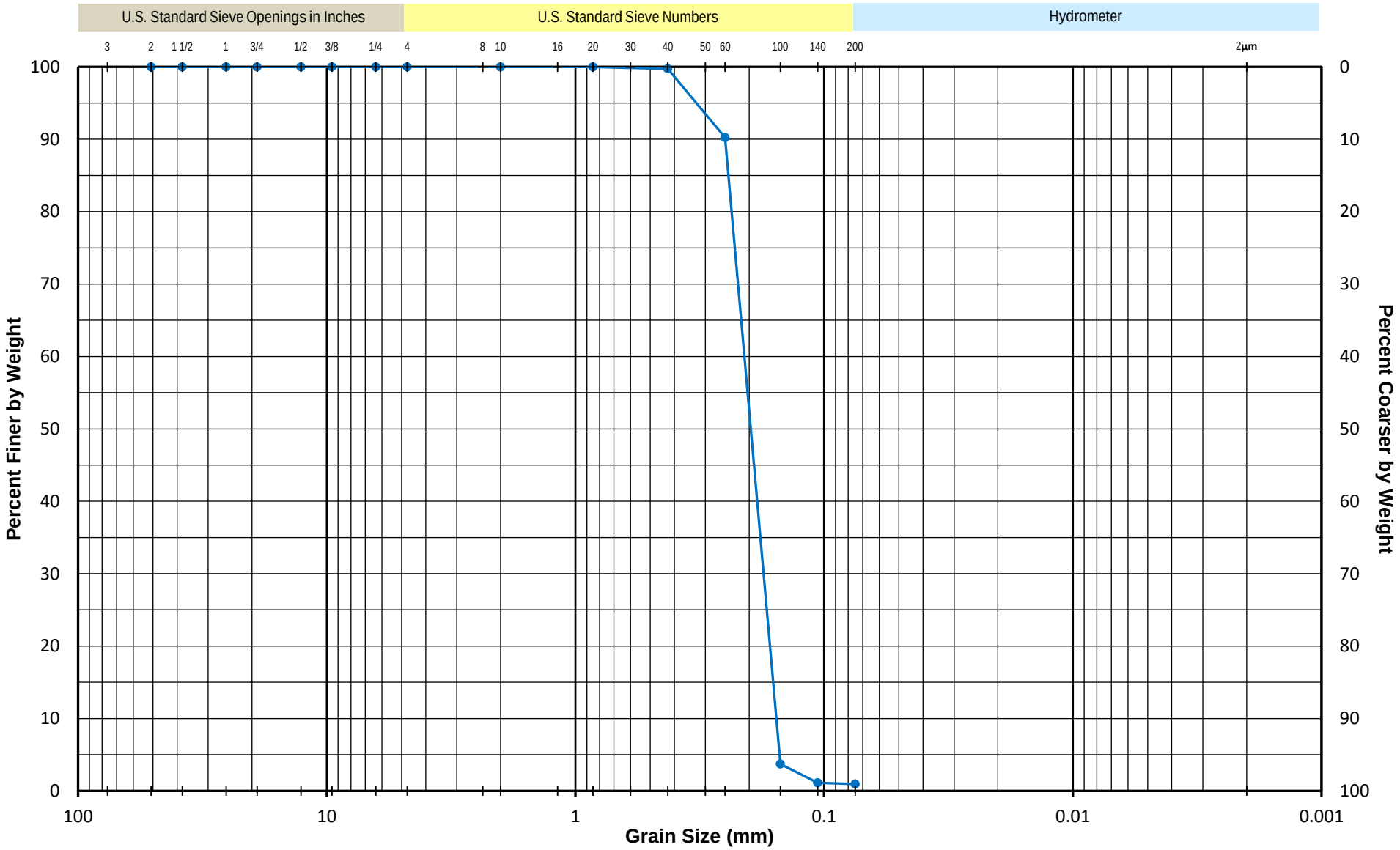


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC49 4-6	Grayish Brown Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

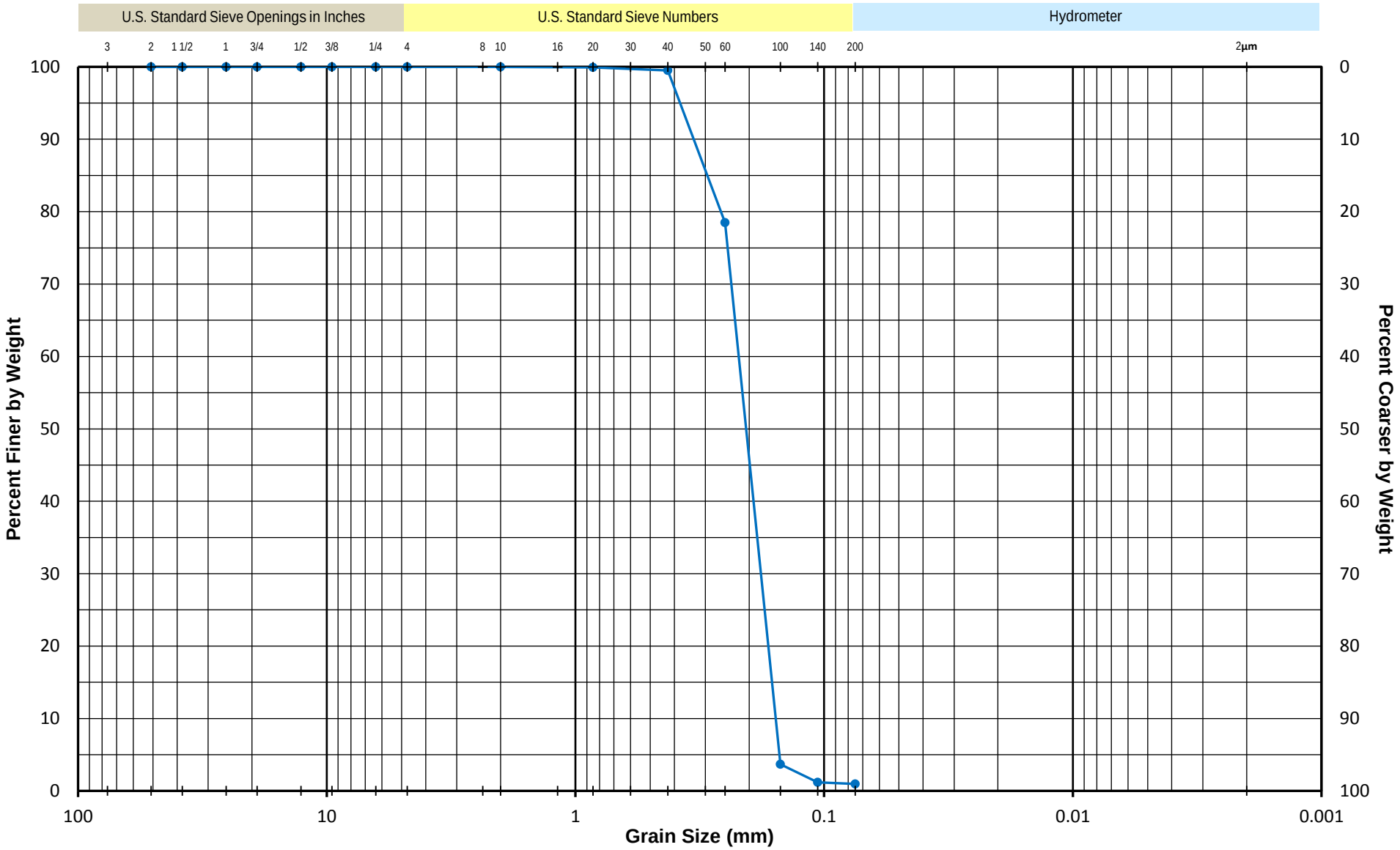


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC49 6-8	Grayish Brown Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

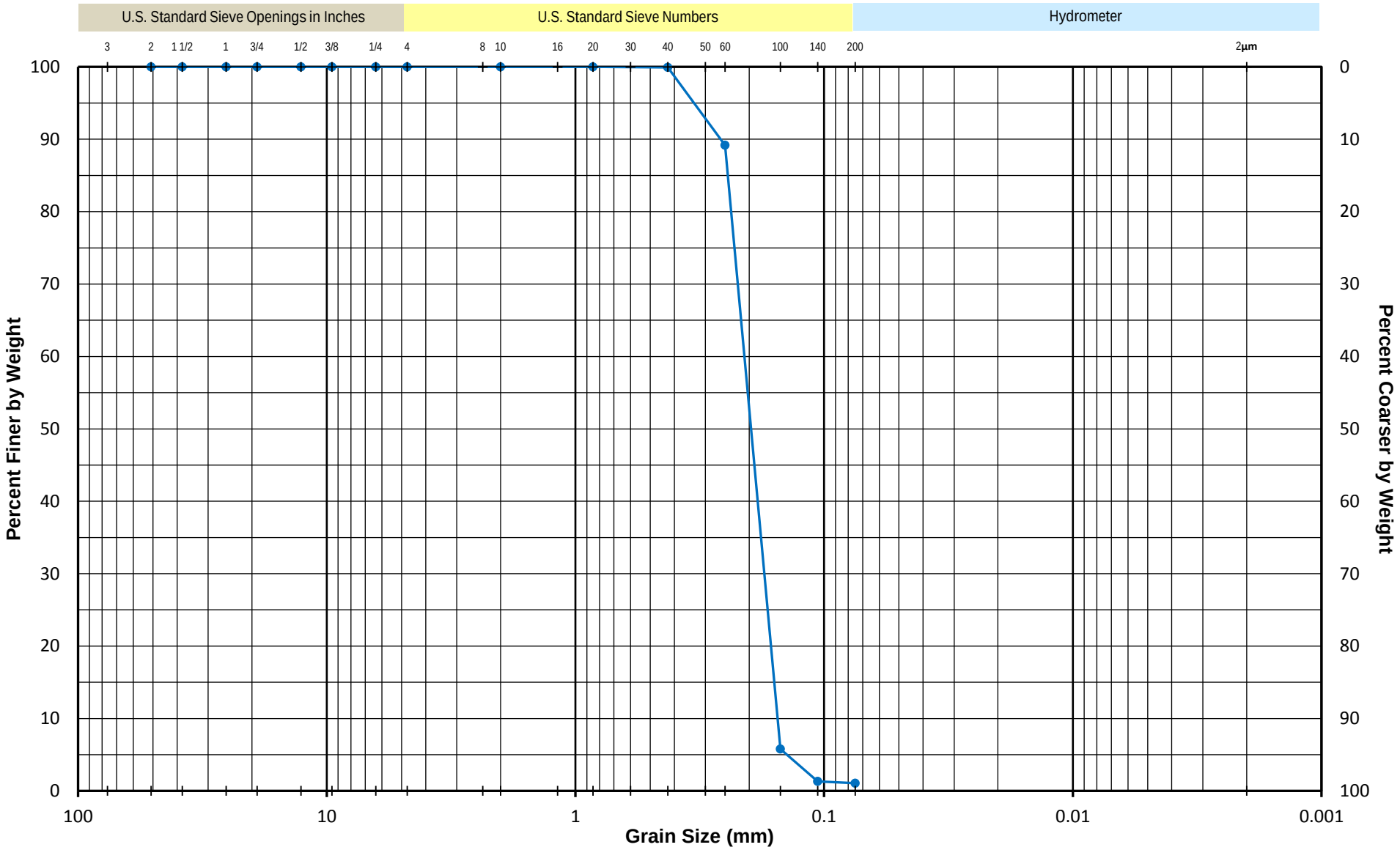


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC49 8-10	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

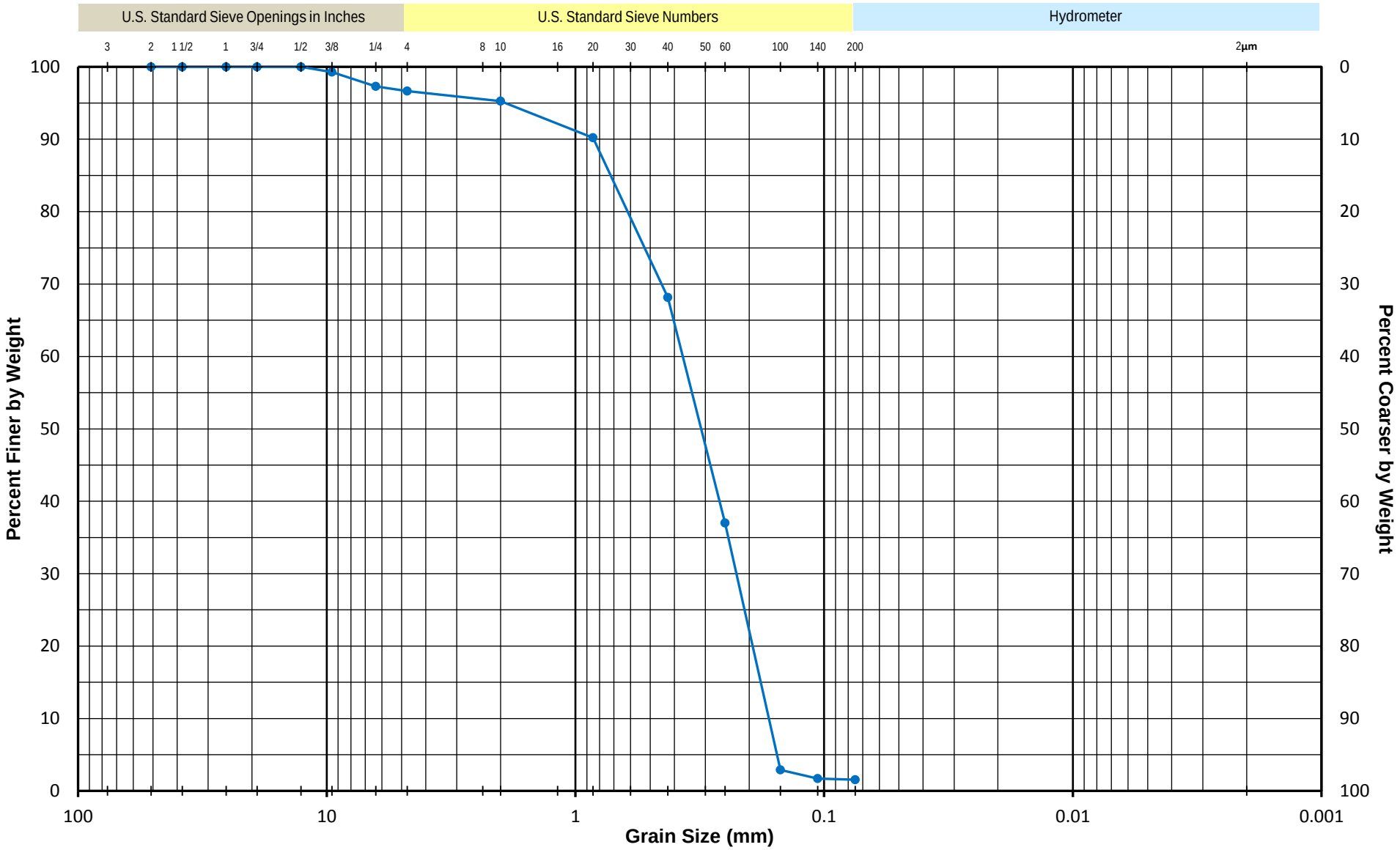


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC49 10-12	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

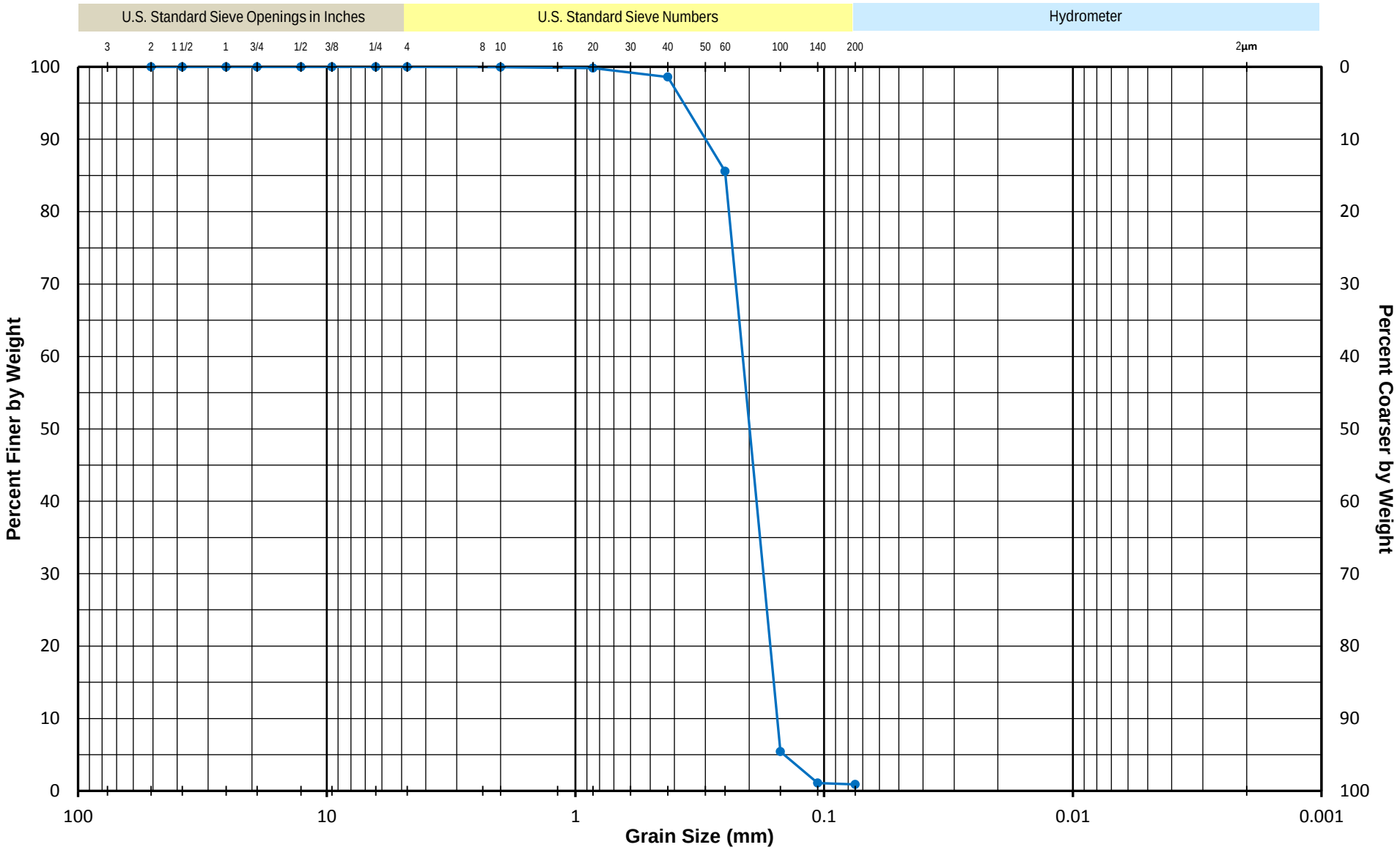


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC50 0-2	Grayish Brown Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

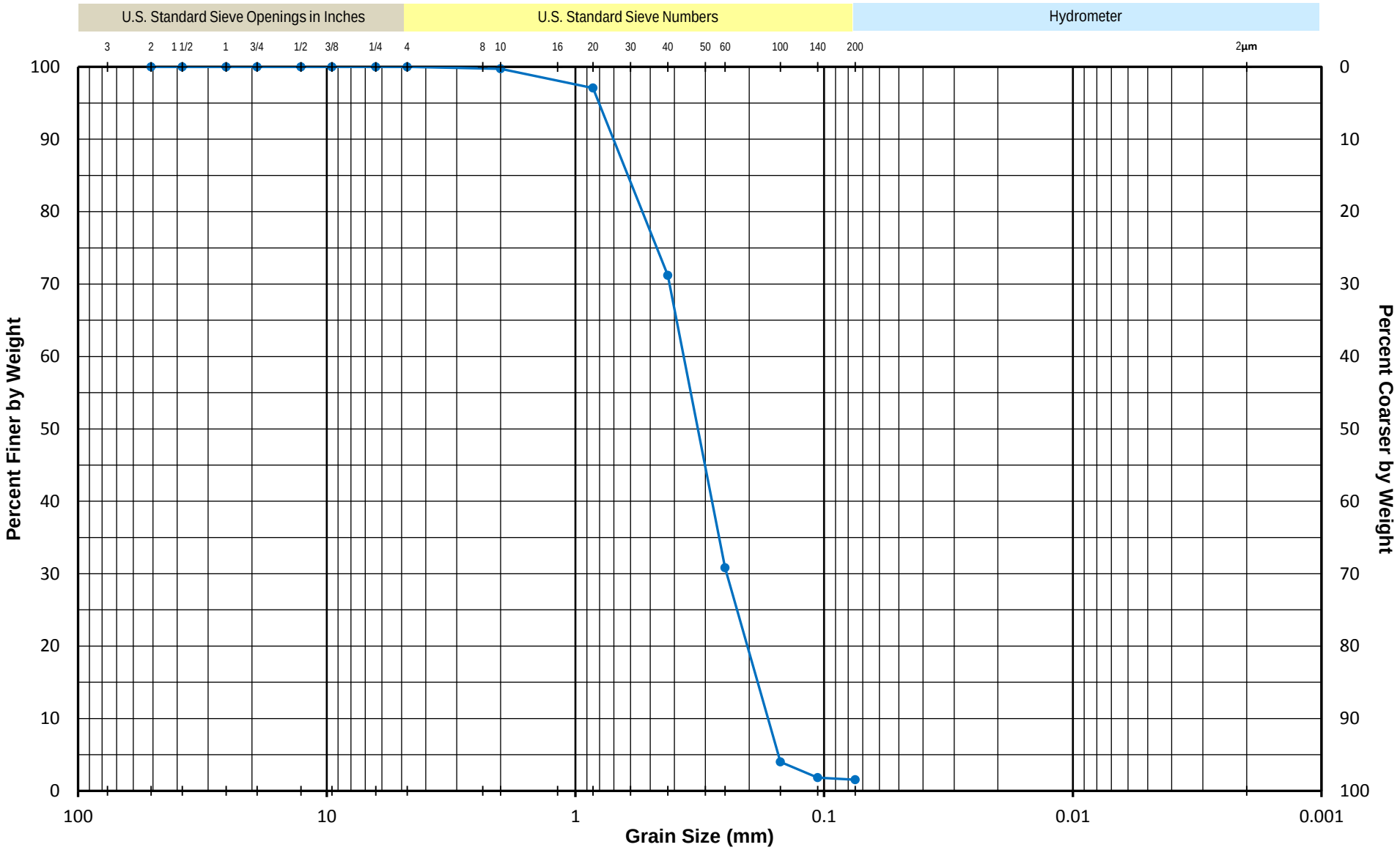


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC50 0-2	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

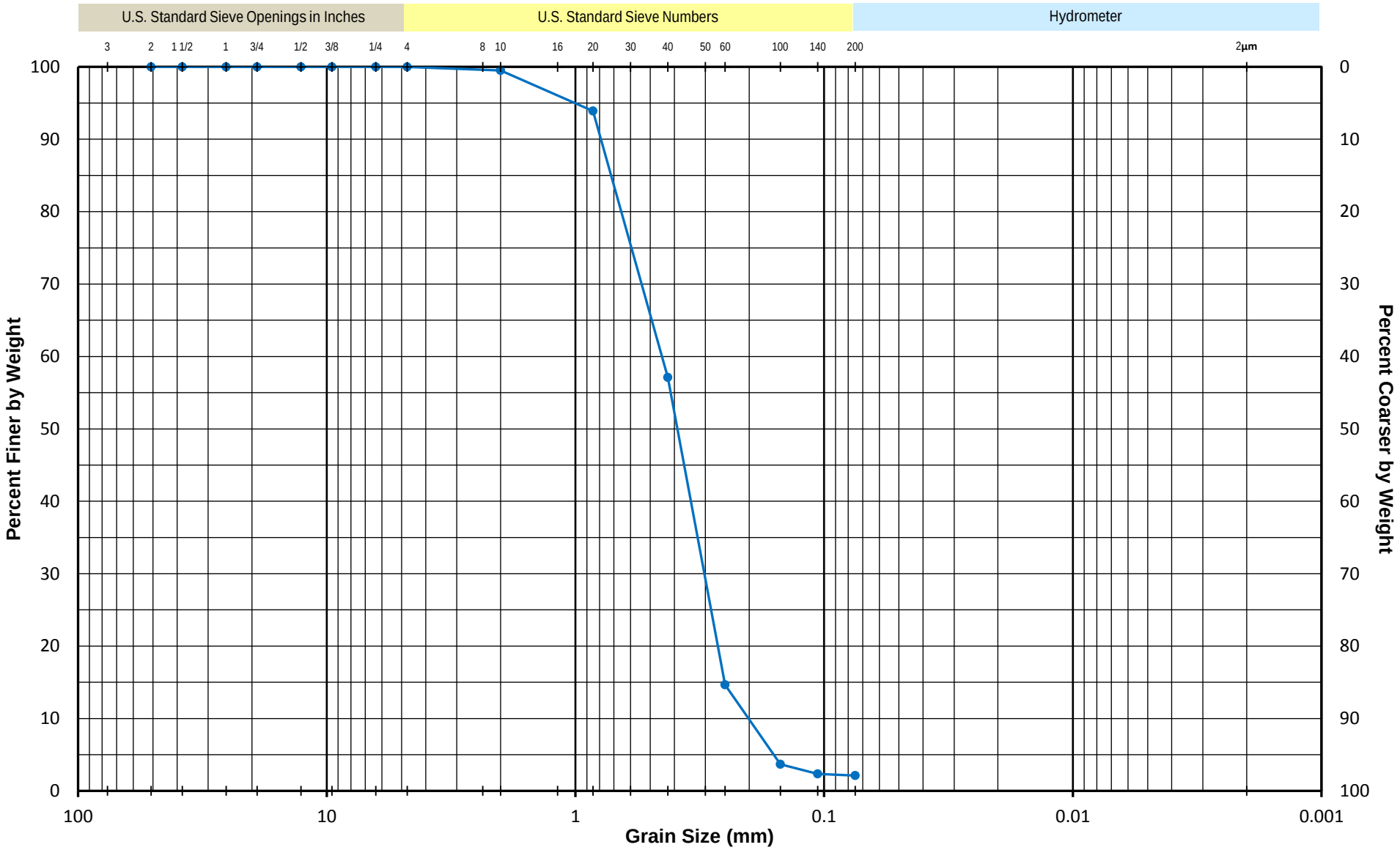


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC50 2-4	Grayish Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

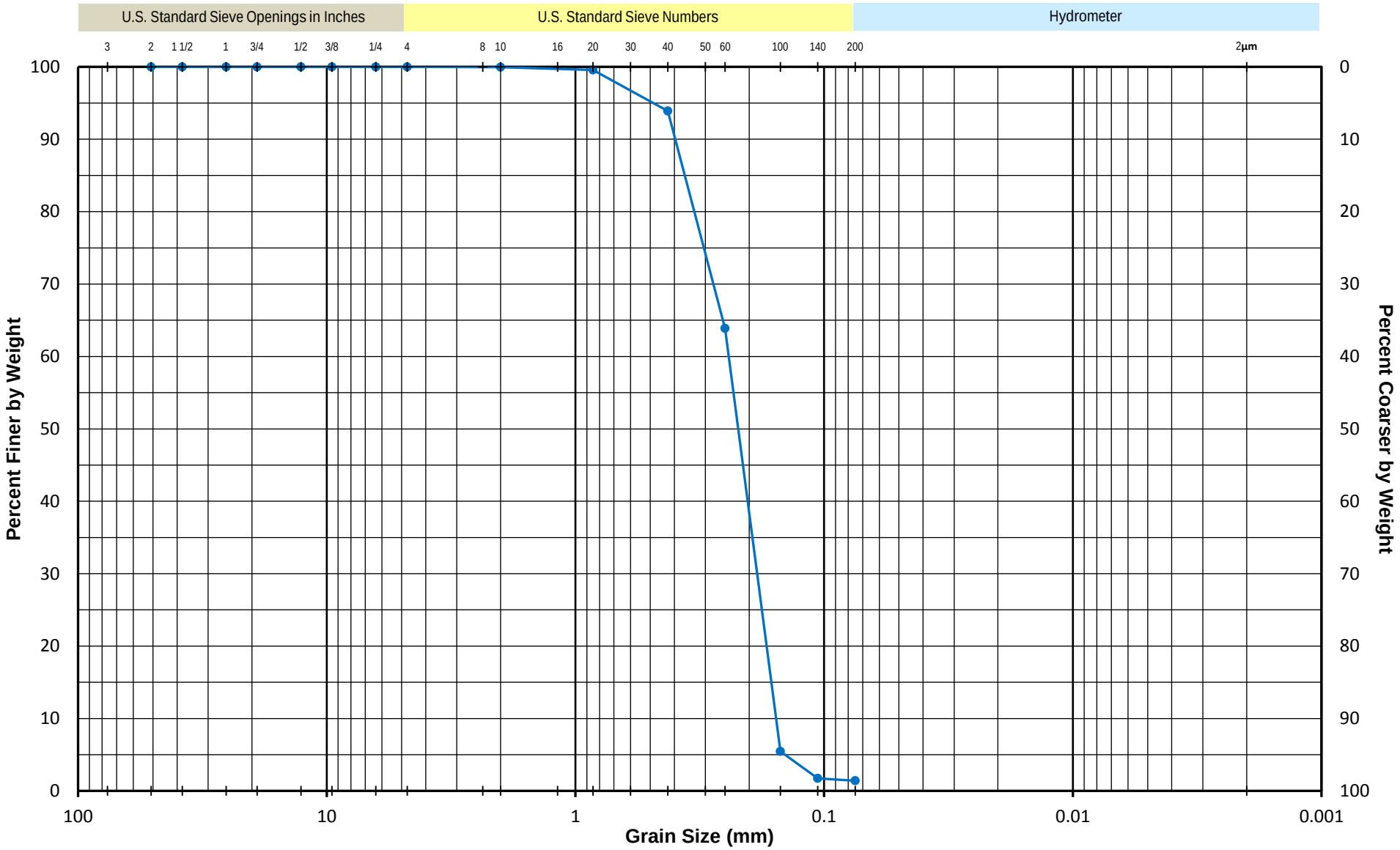


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC50 4-6	Grayish Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

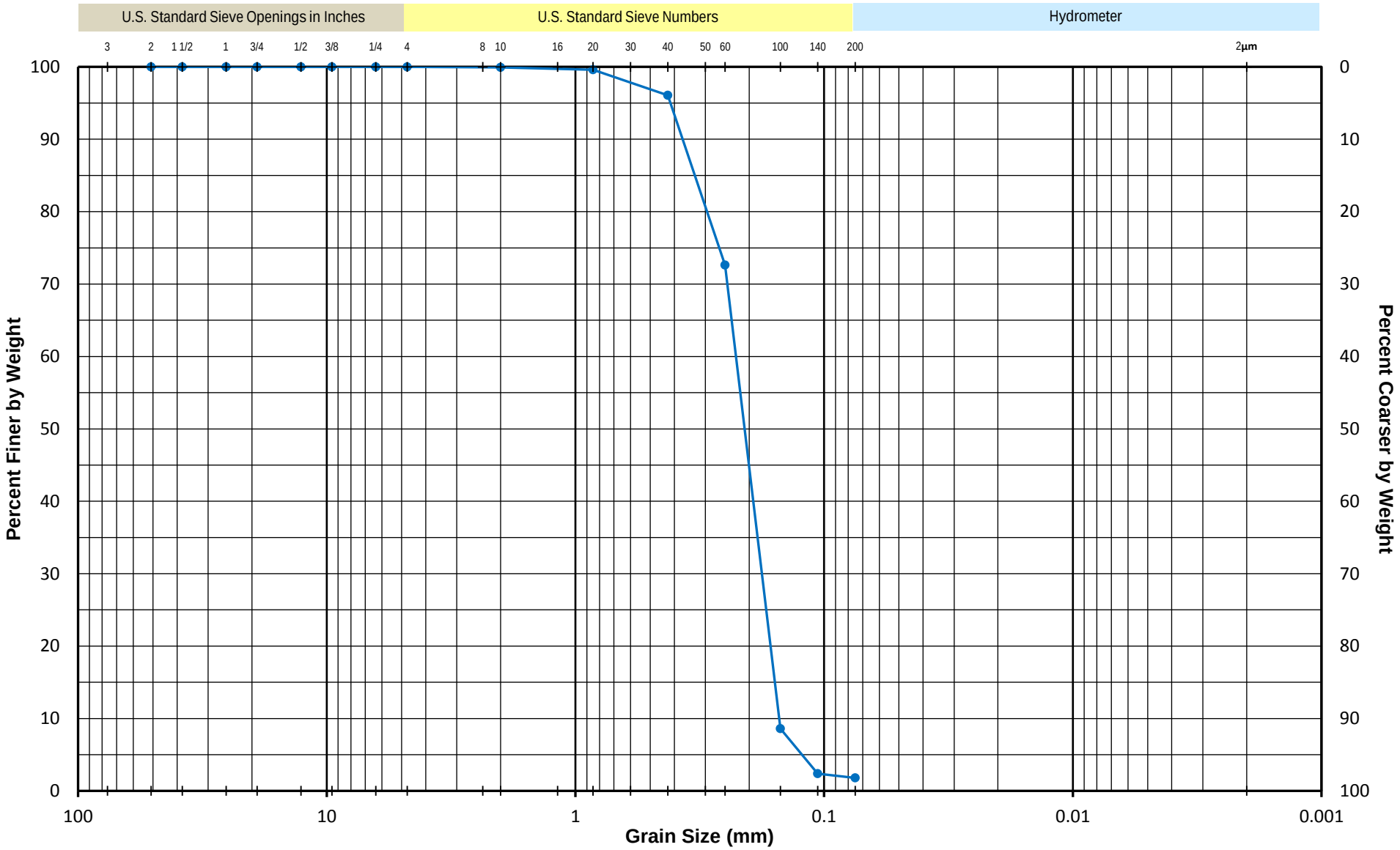


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC50 6-8	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

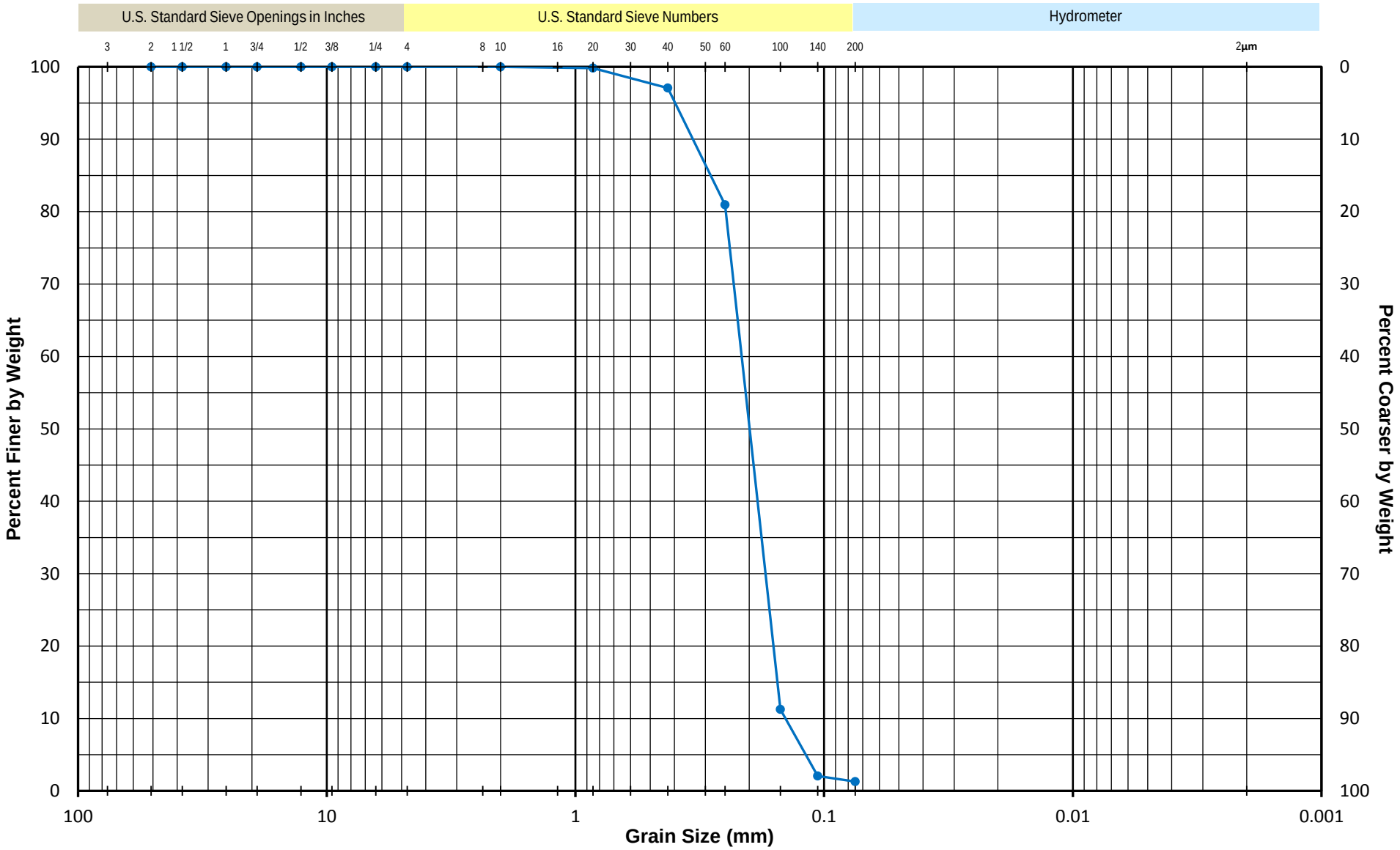


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC50 8-10	Dark Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

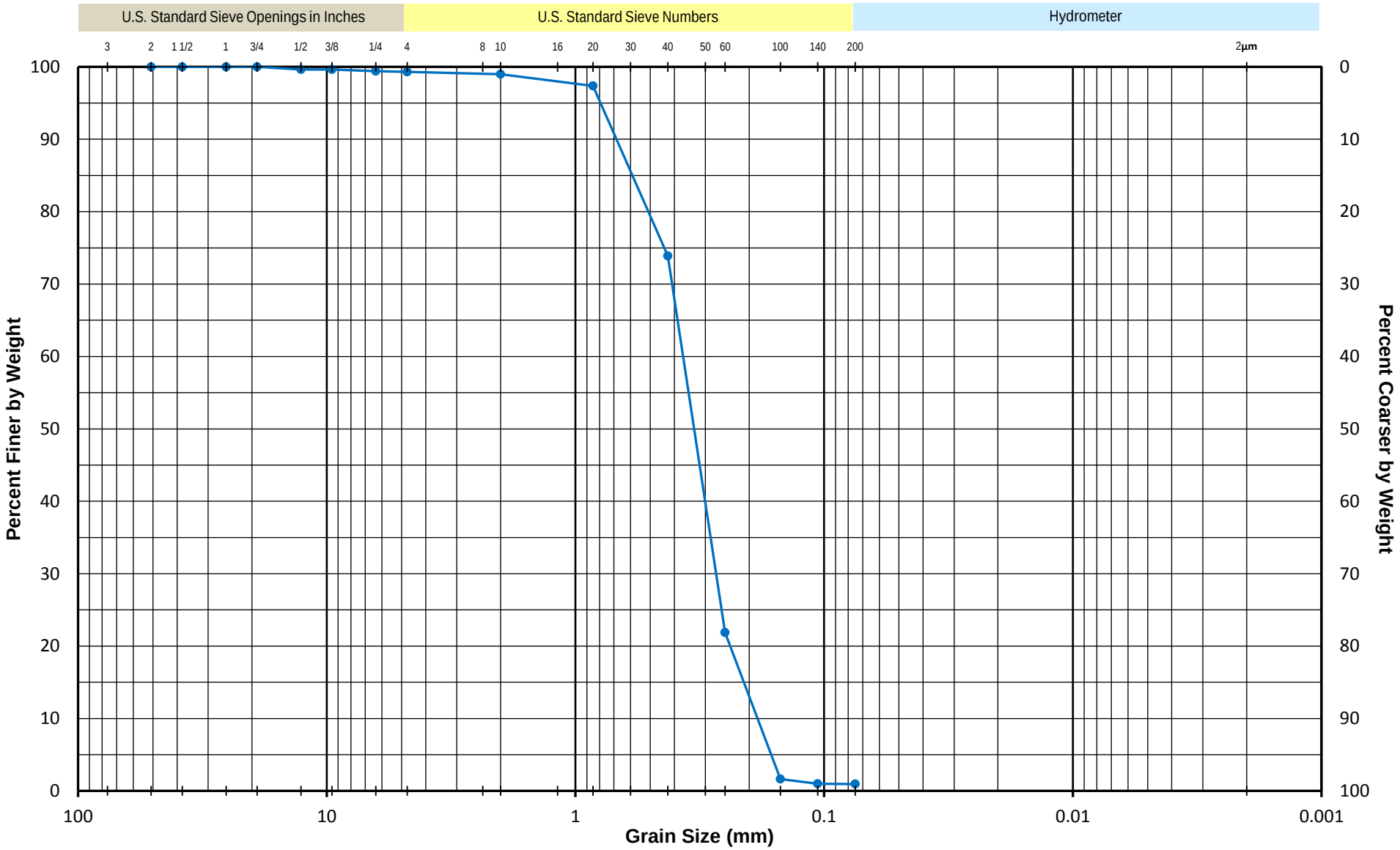


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC50 10-12	Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

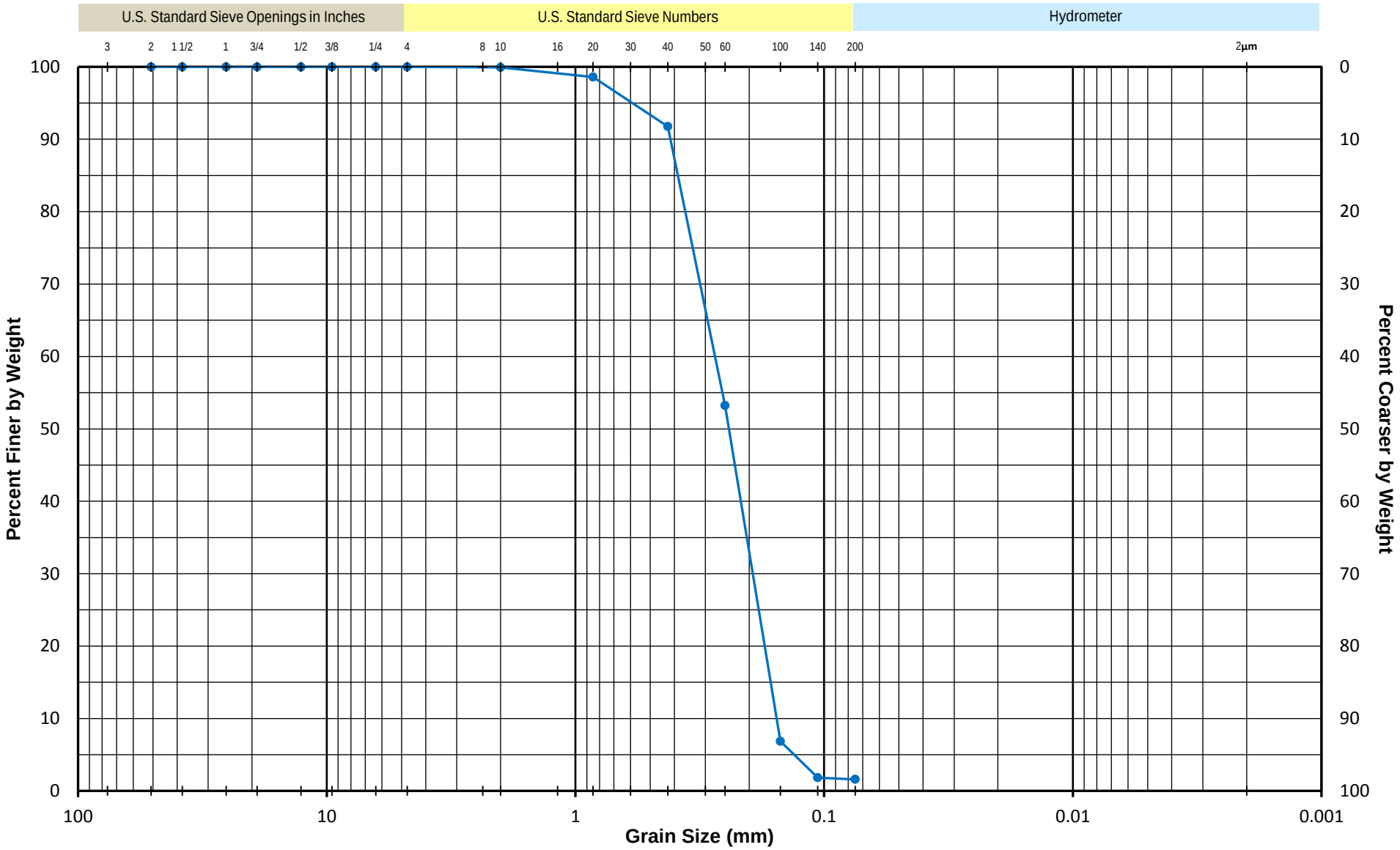


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC51 0-2	Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

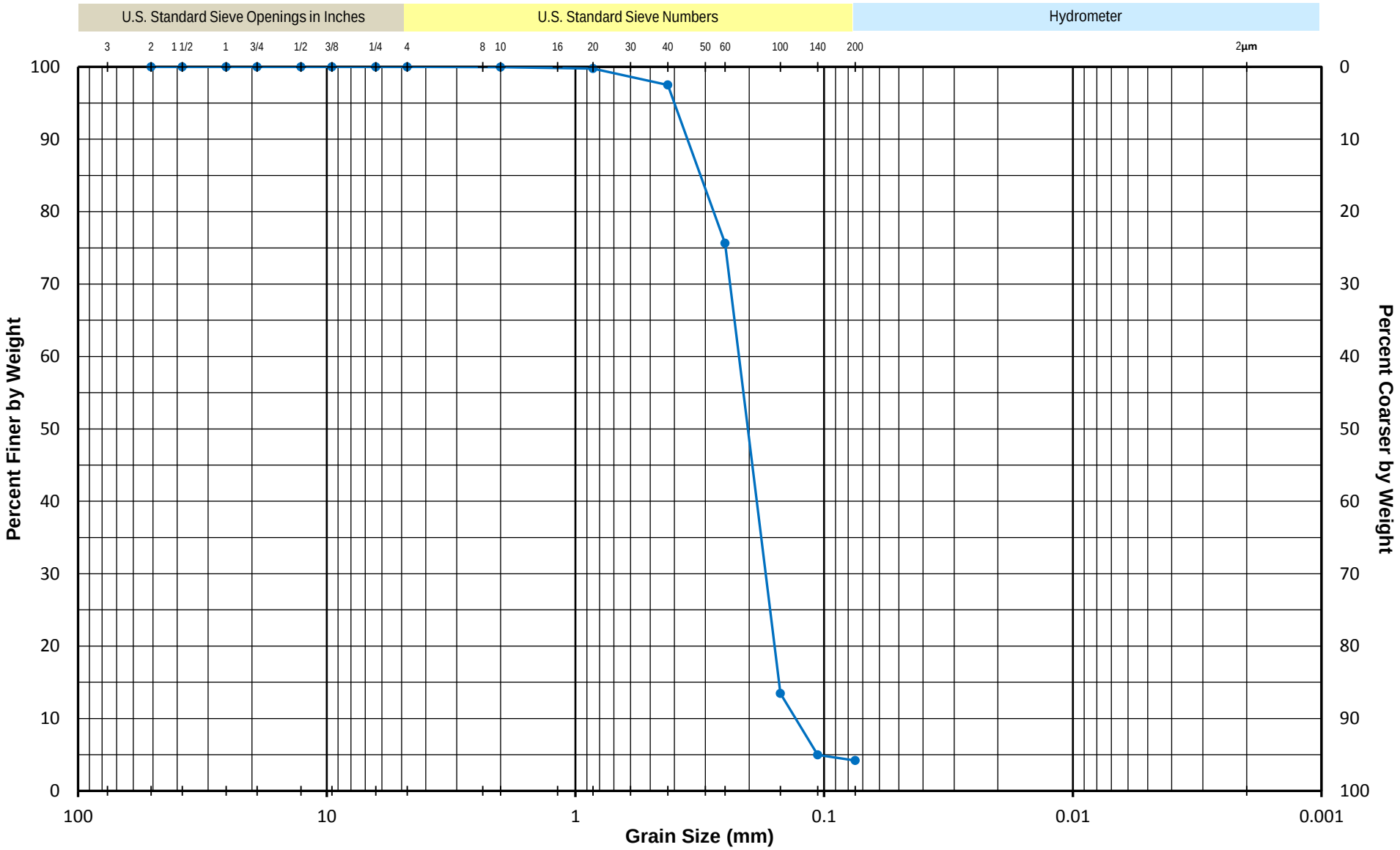


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC51 0-2	Dark Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

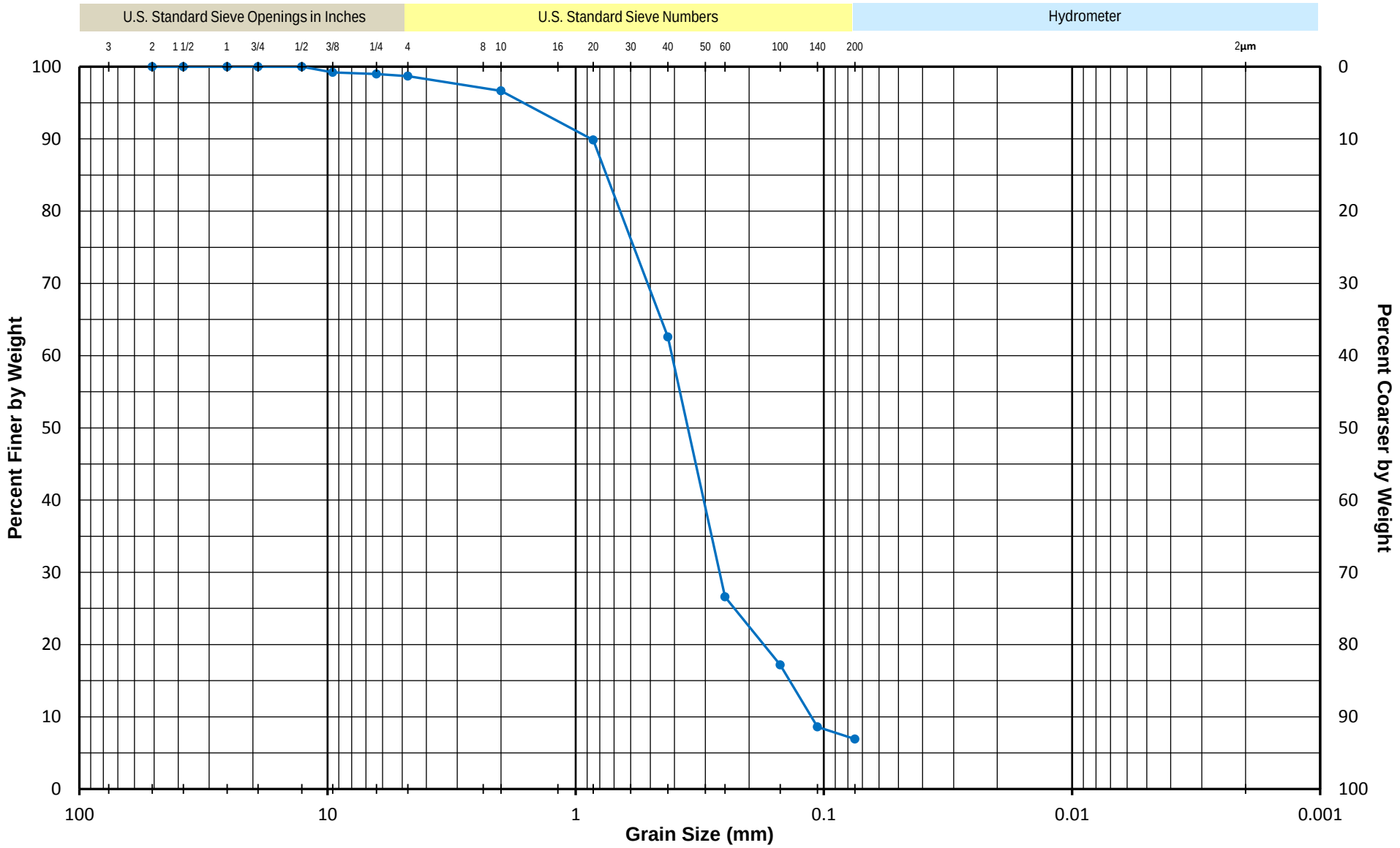


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC51 2-4	Very Dark Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

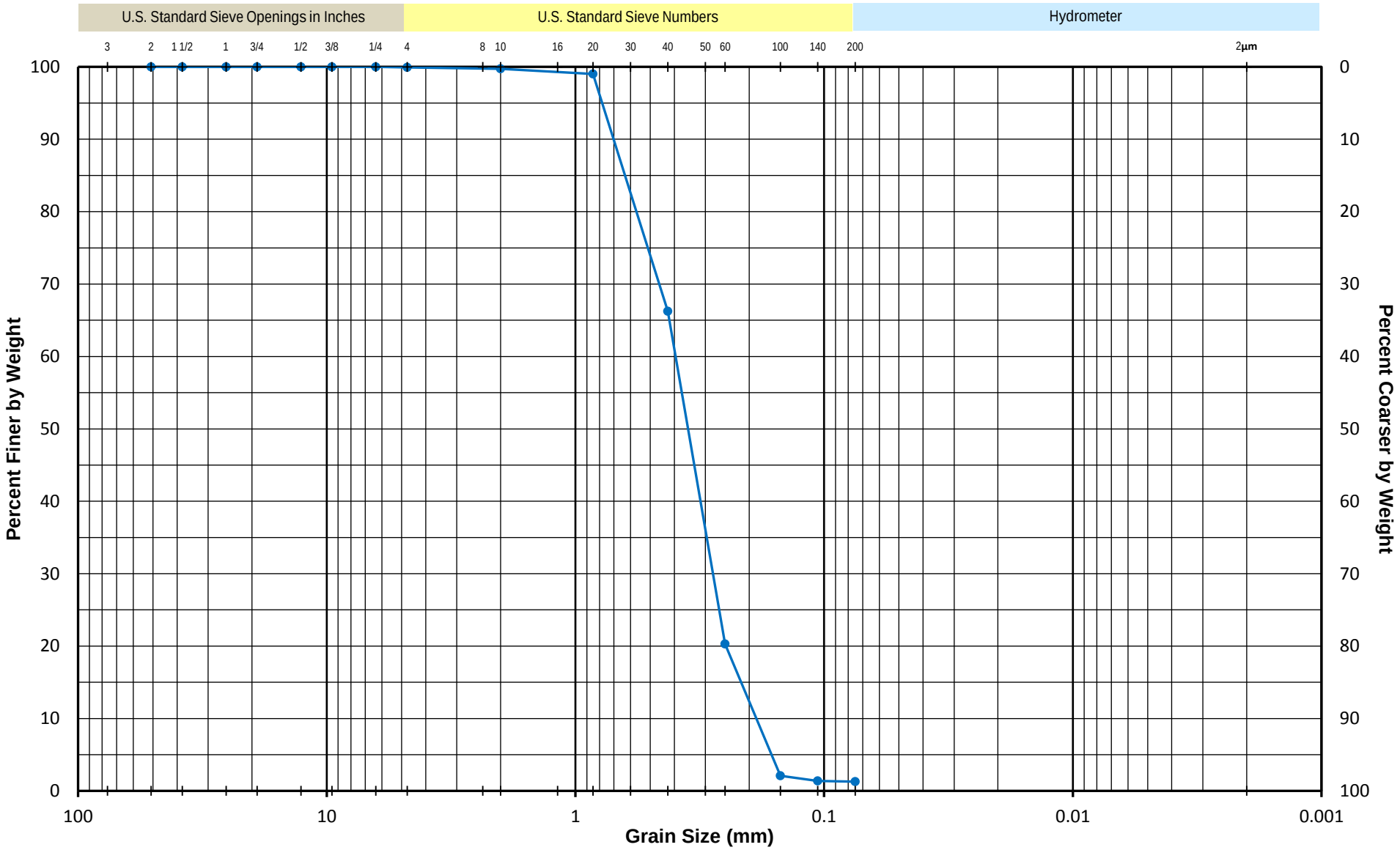


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC51 4-6	Very Dark Gray Fine to Medium Sand with silt and shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

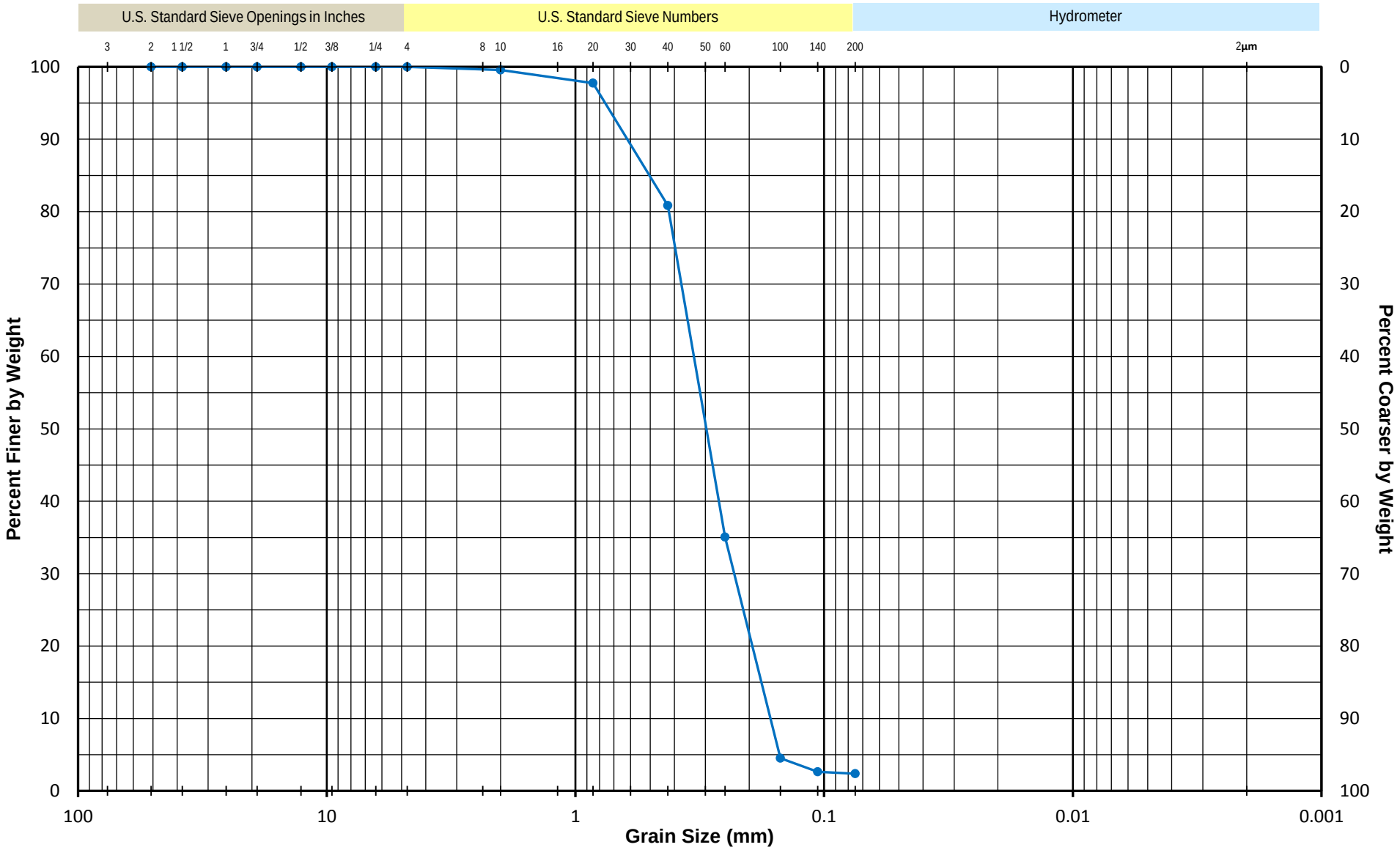


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC51 6-8	Grayish Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

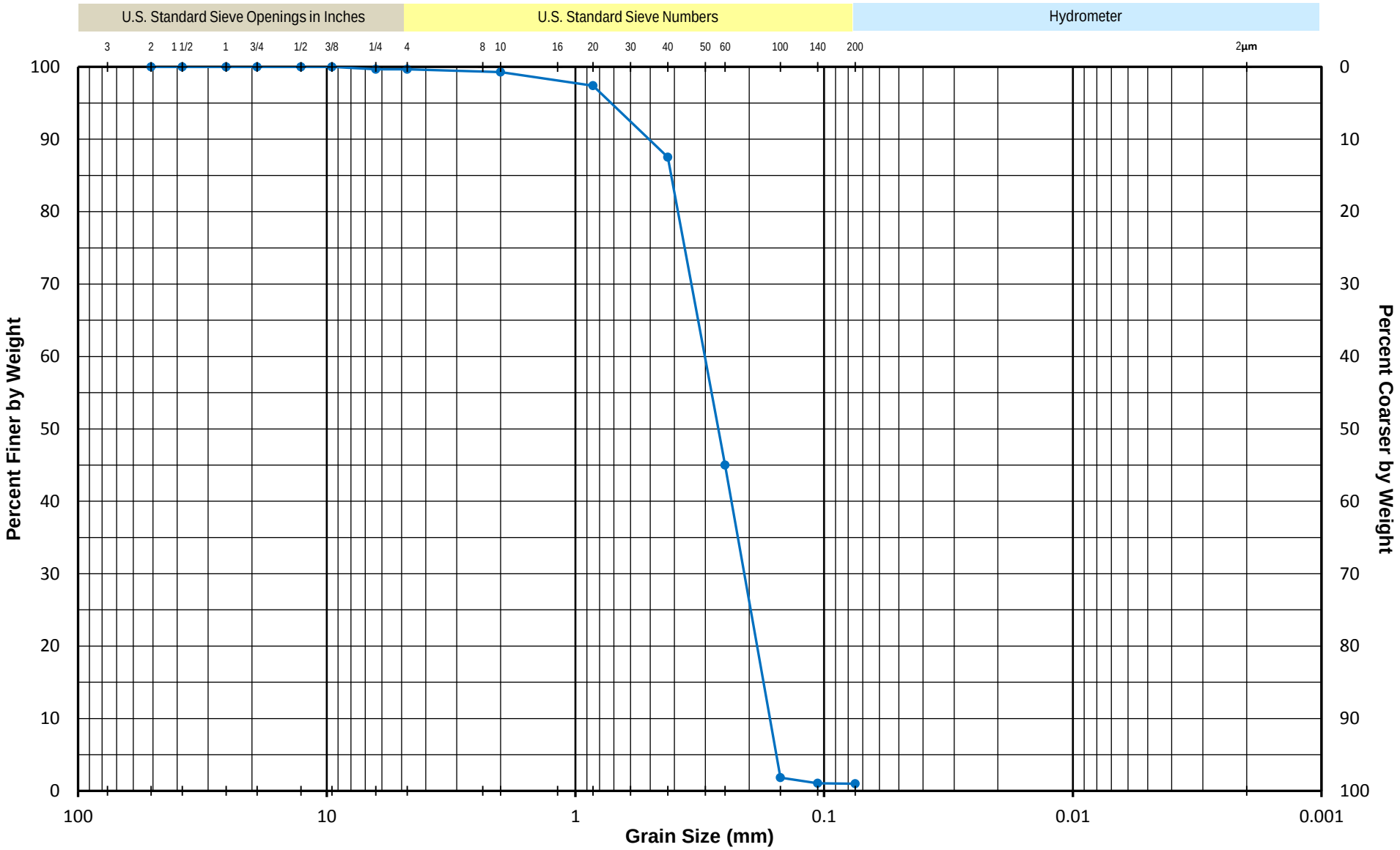


Test Depth	Core ID Section/Sample	Material Description
9.3 ft	VC51 8-10	Grayish Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

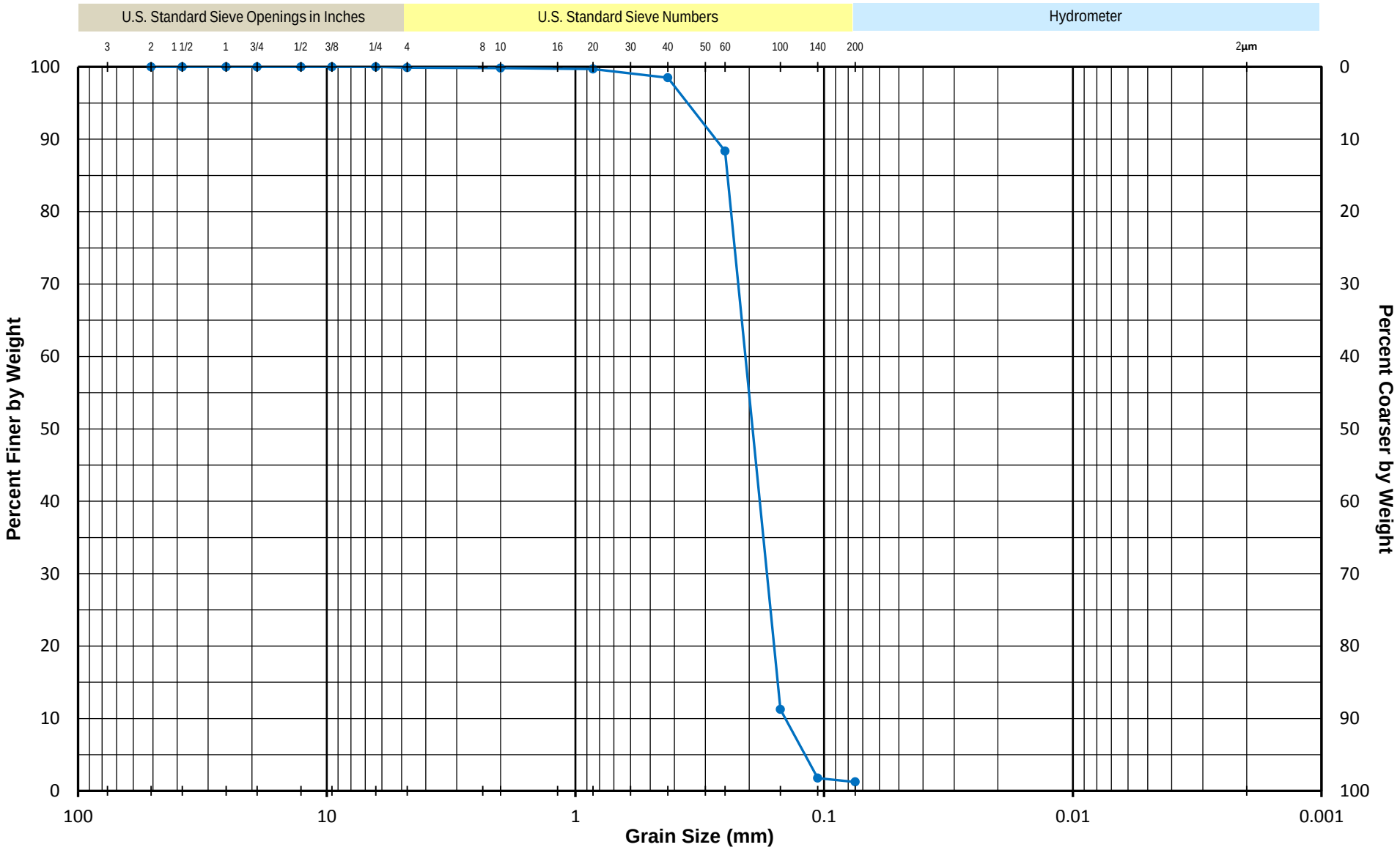


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC52 0-2	Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

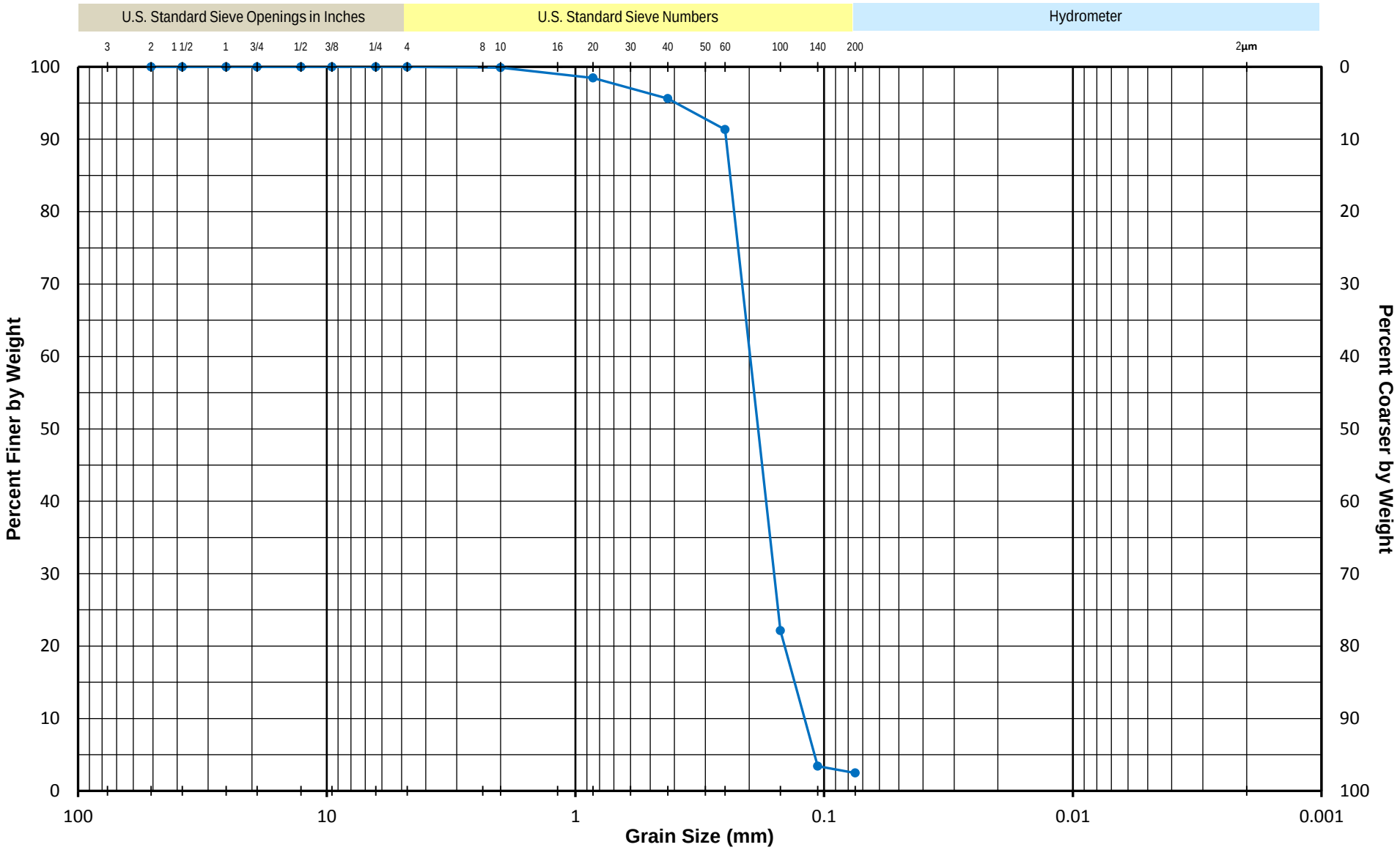


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC52 0-2	Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

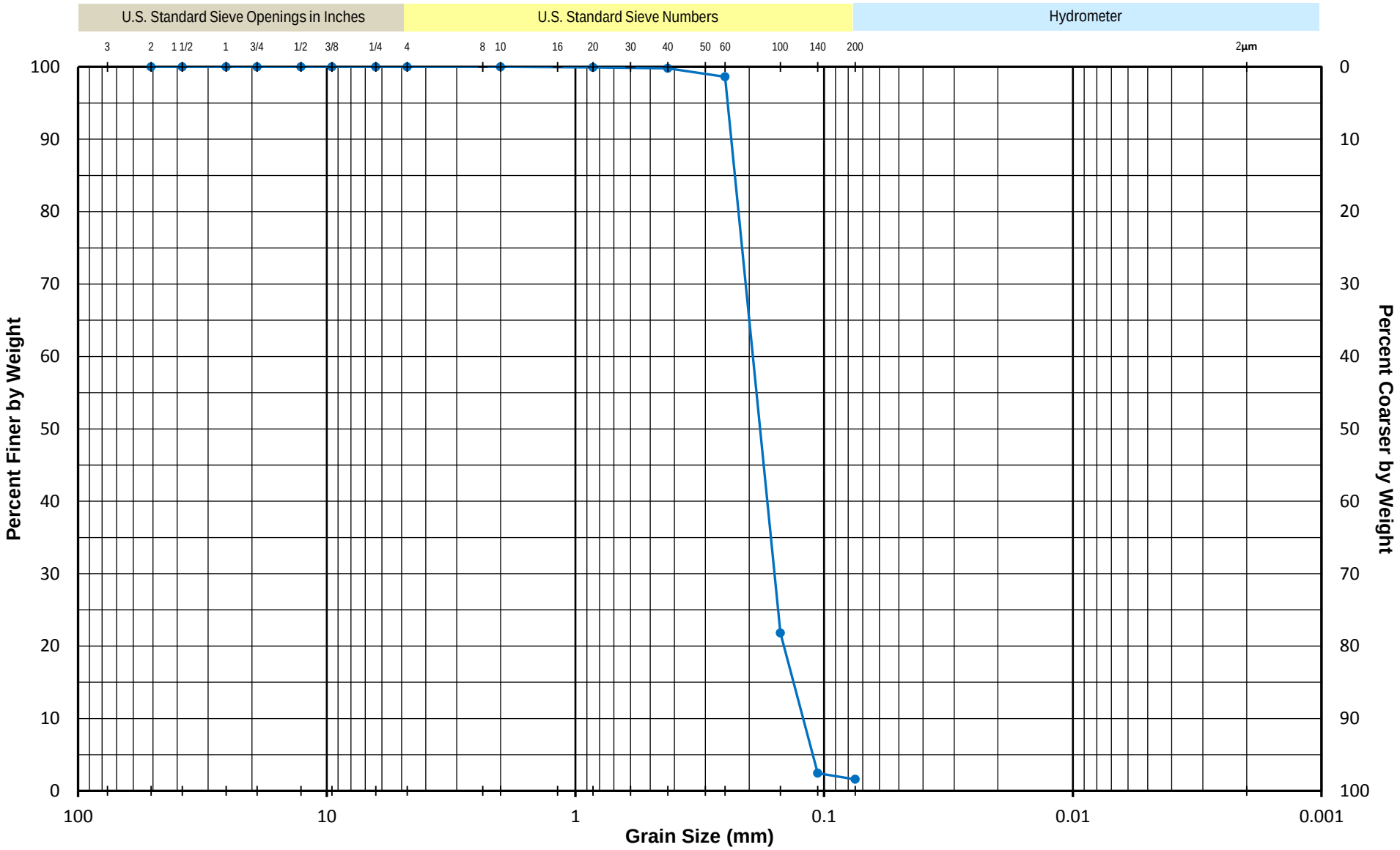


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC52 2-4	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

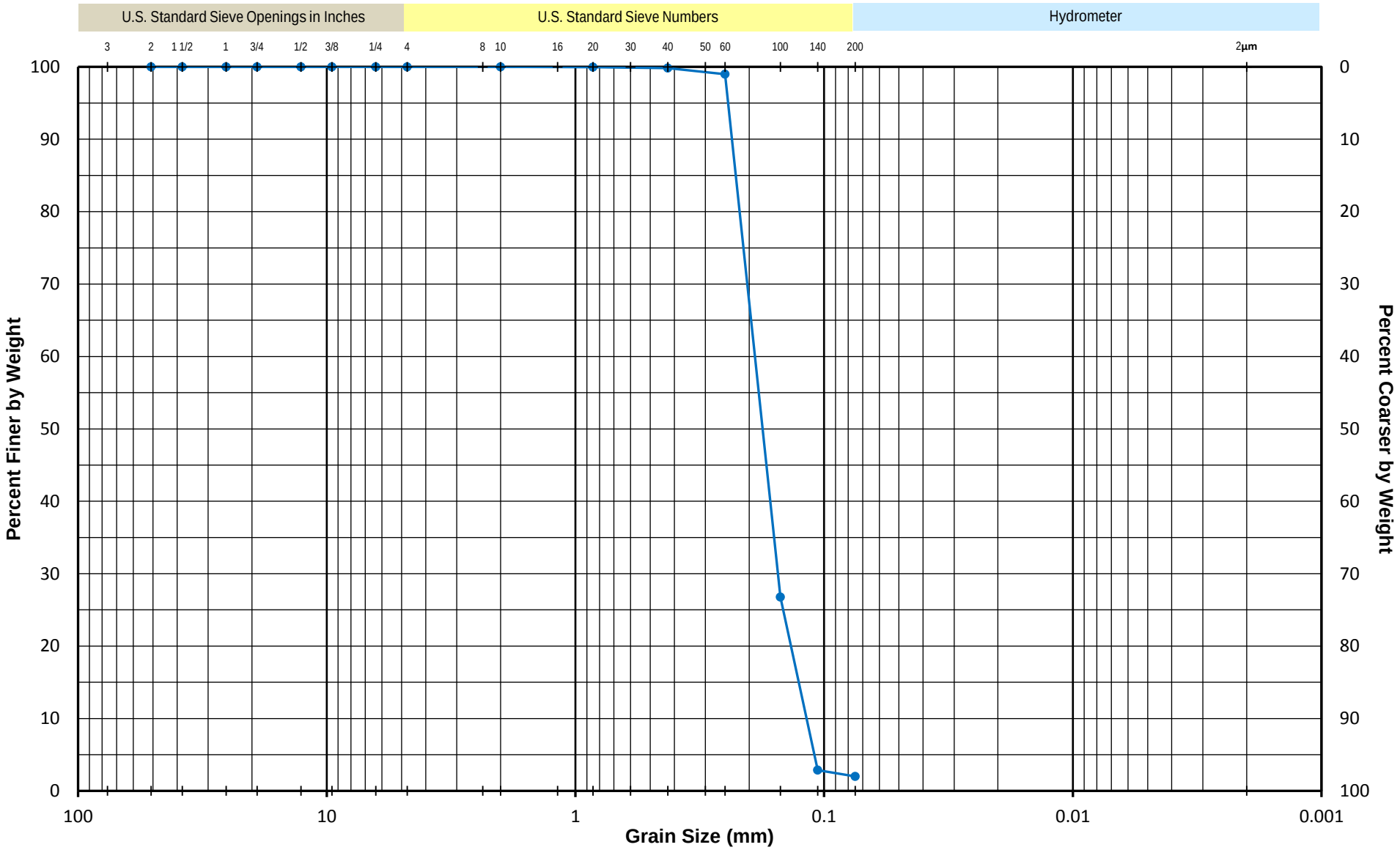


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC52 4-6	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

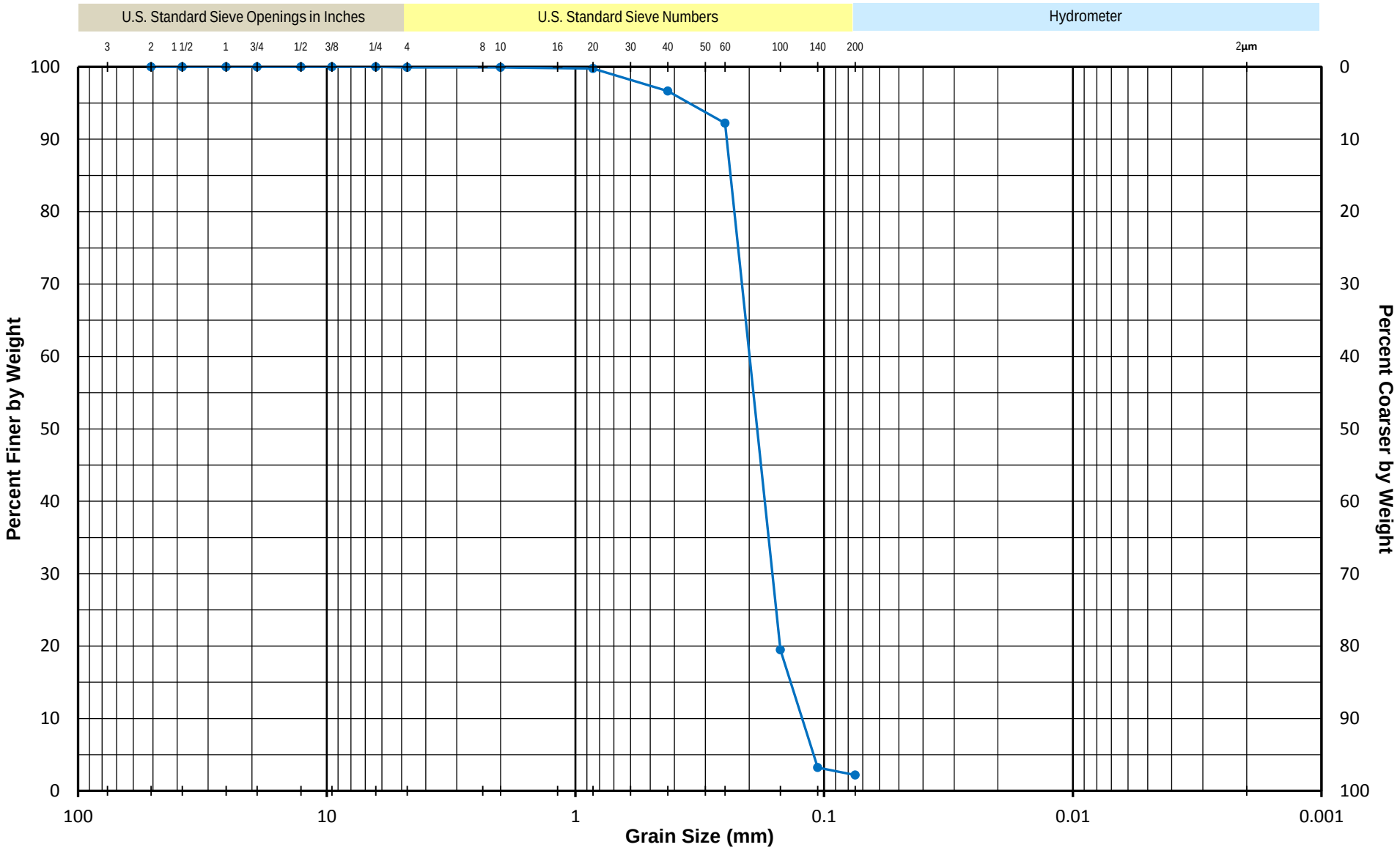


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC52 6-8	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

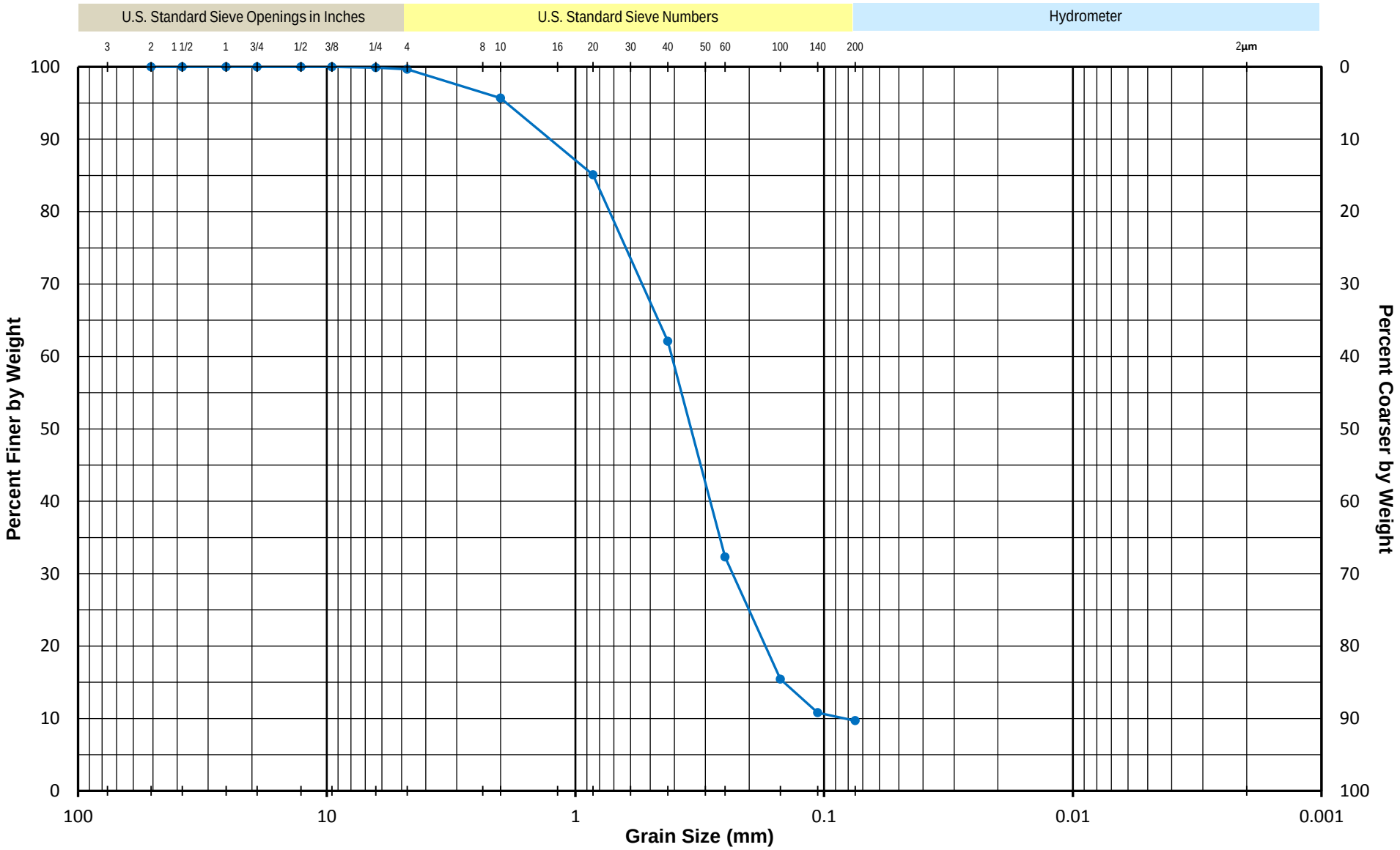


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC52 8-10	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

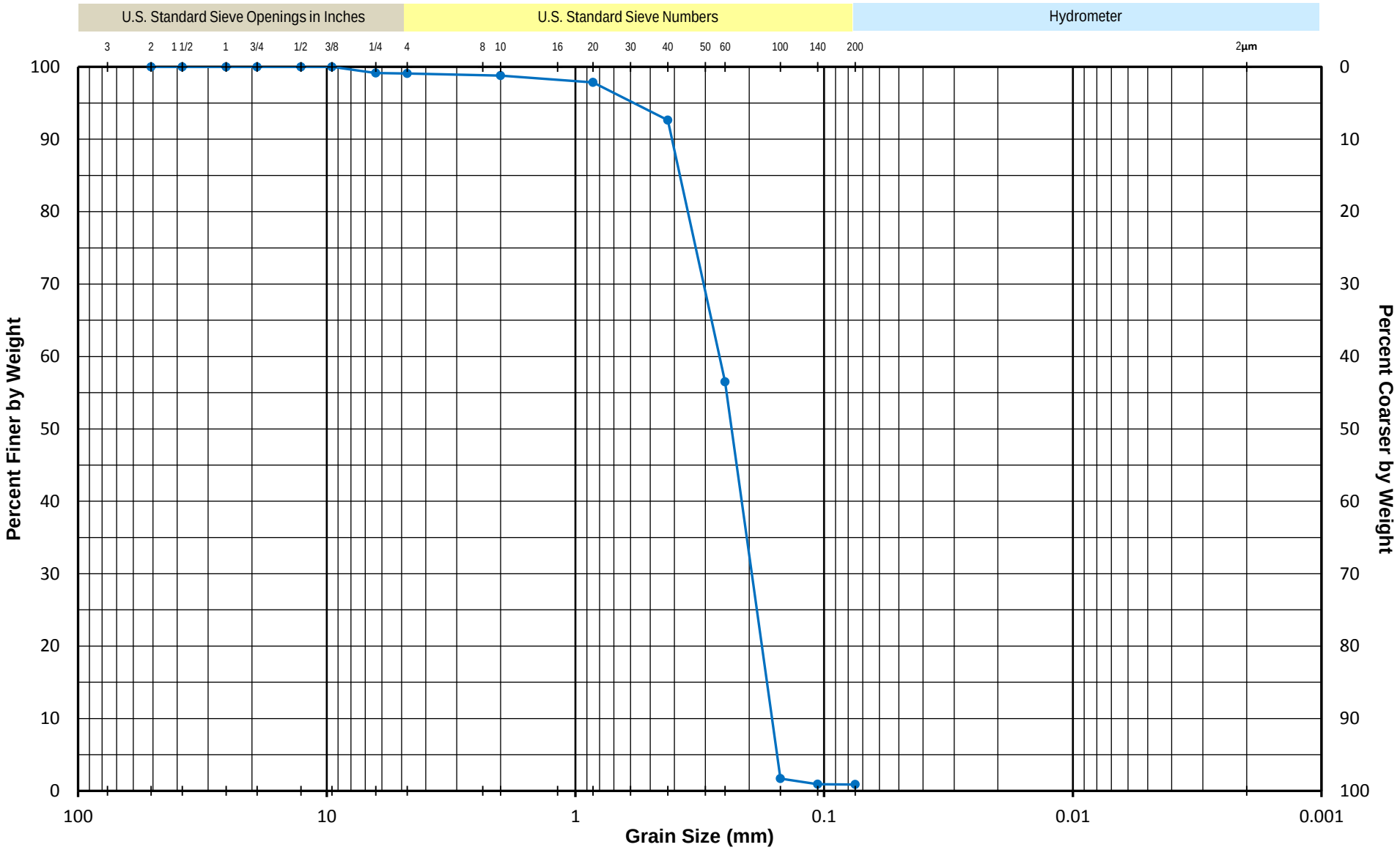


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC52 10-12	Gray Fine to Medium Sand with black silty fine sand seam

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

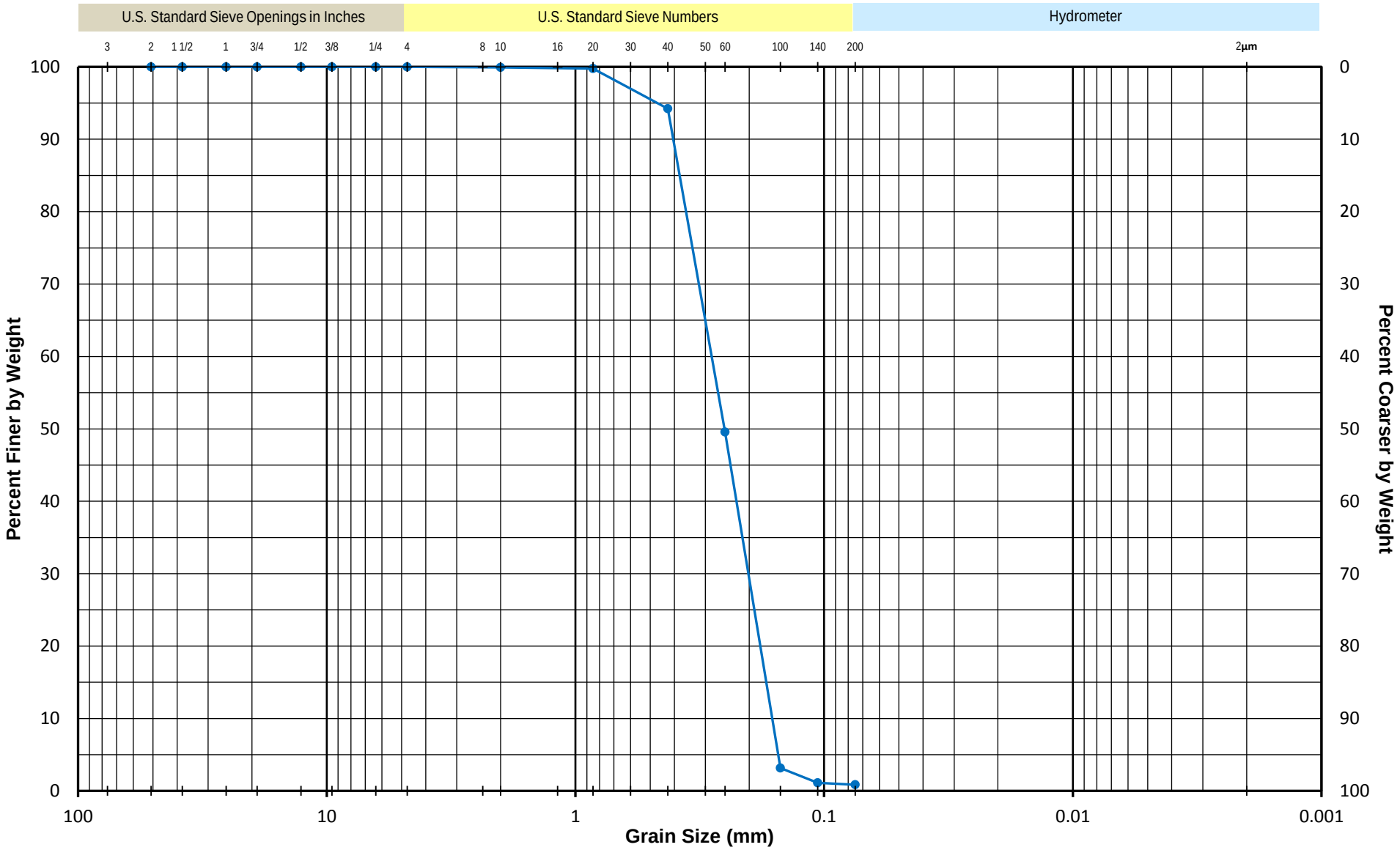


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC53 0-2	Gray and Brown Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

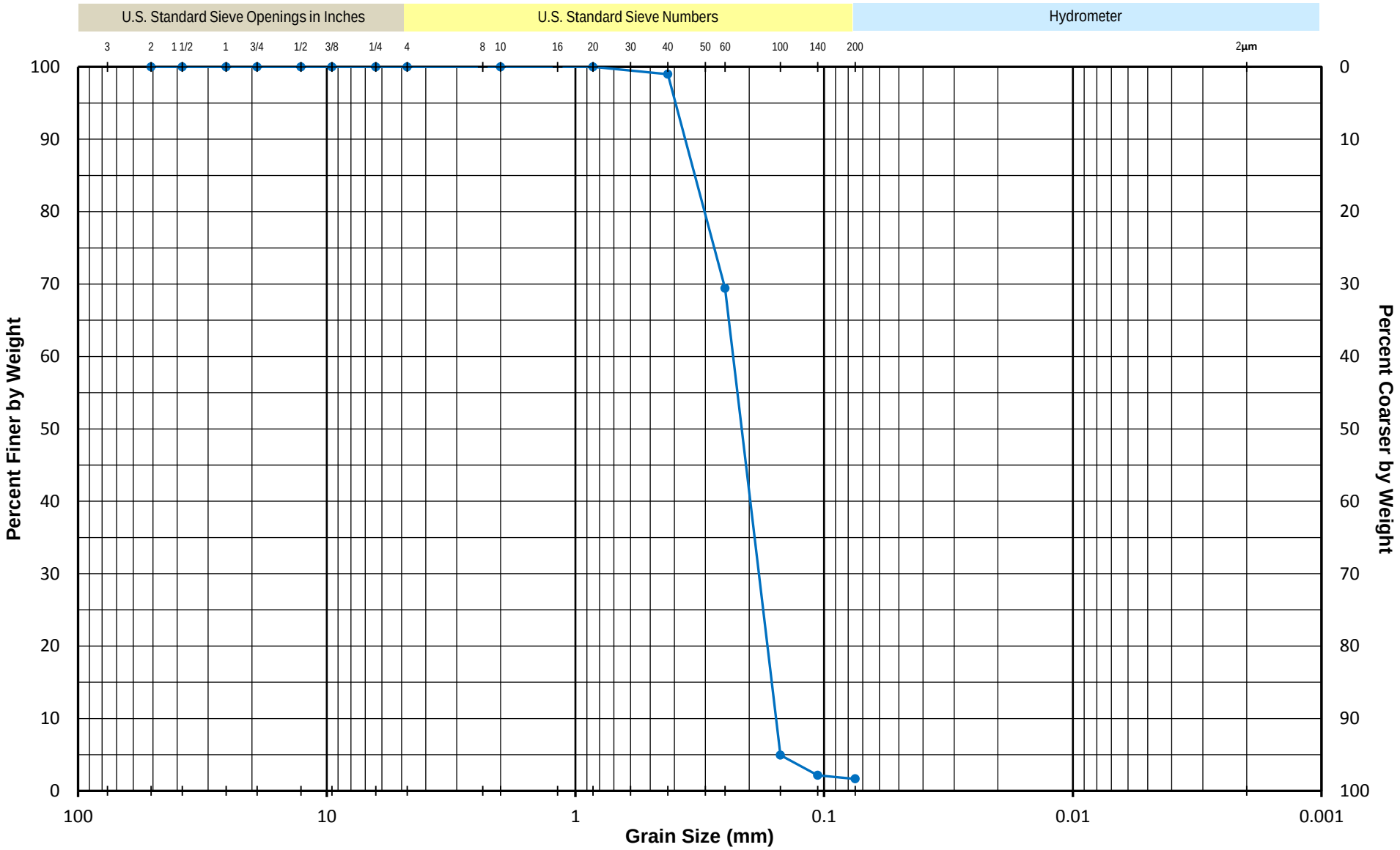


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC53 0-2	Very Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

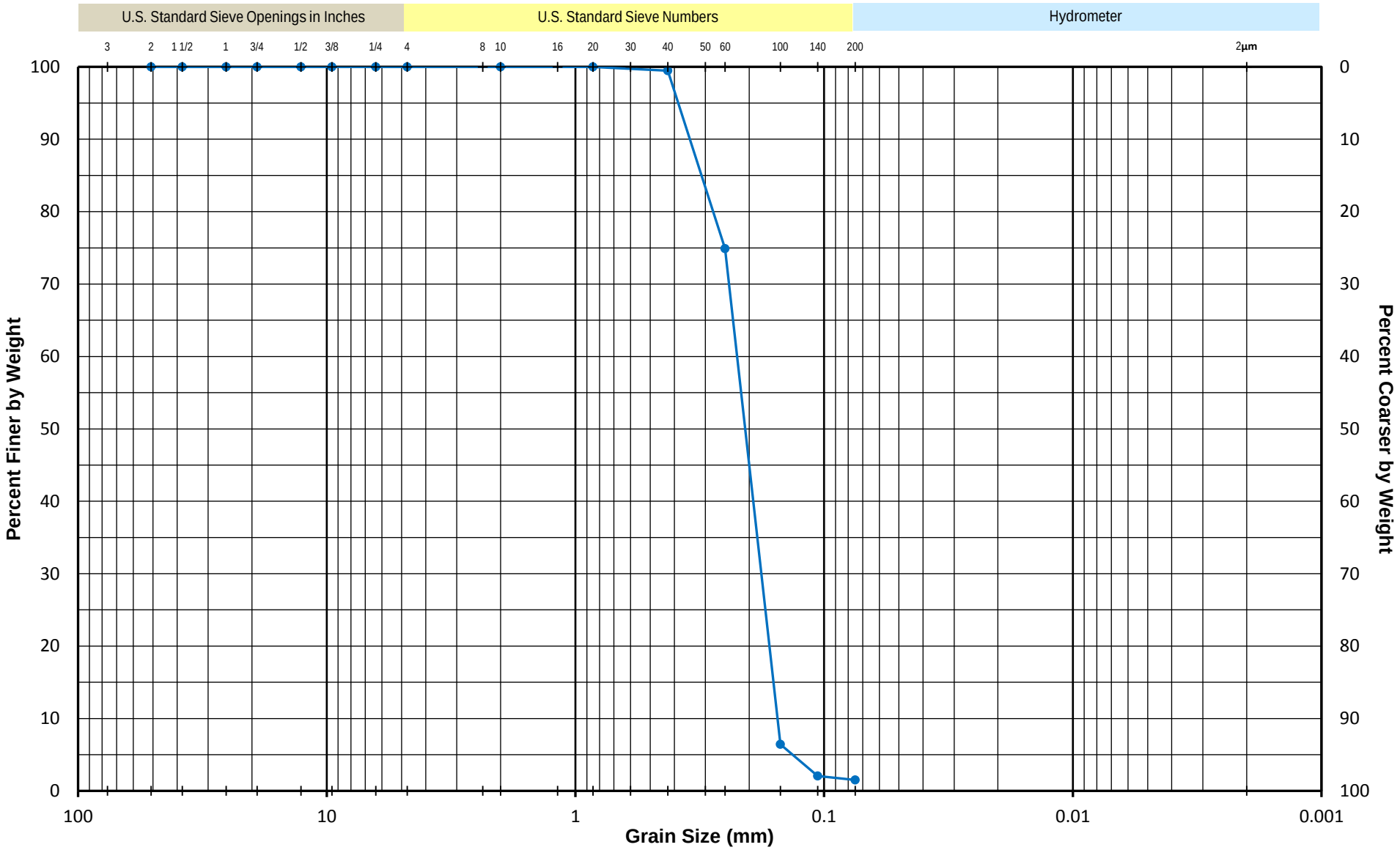


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC53 2-4	Very Dark Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

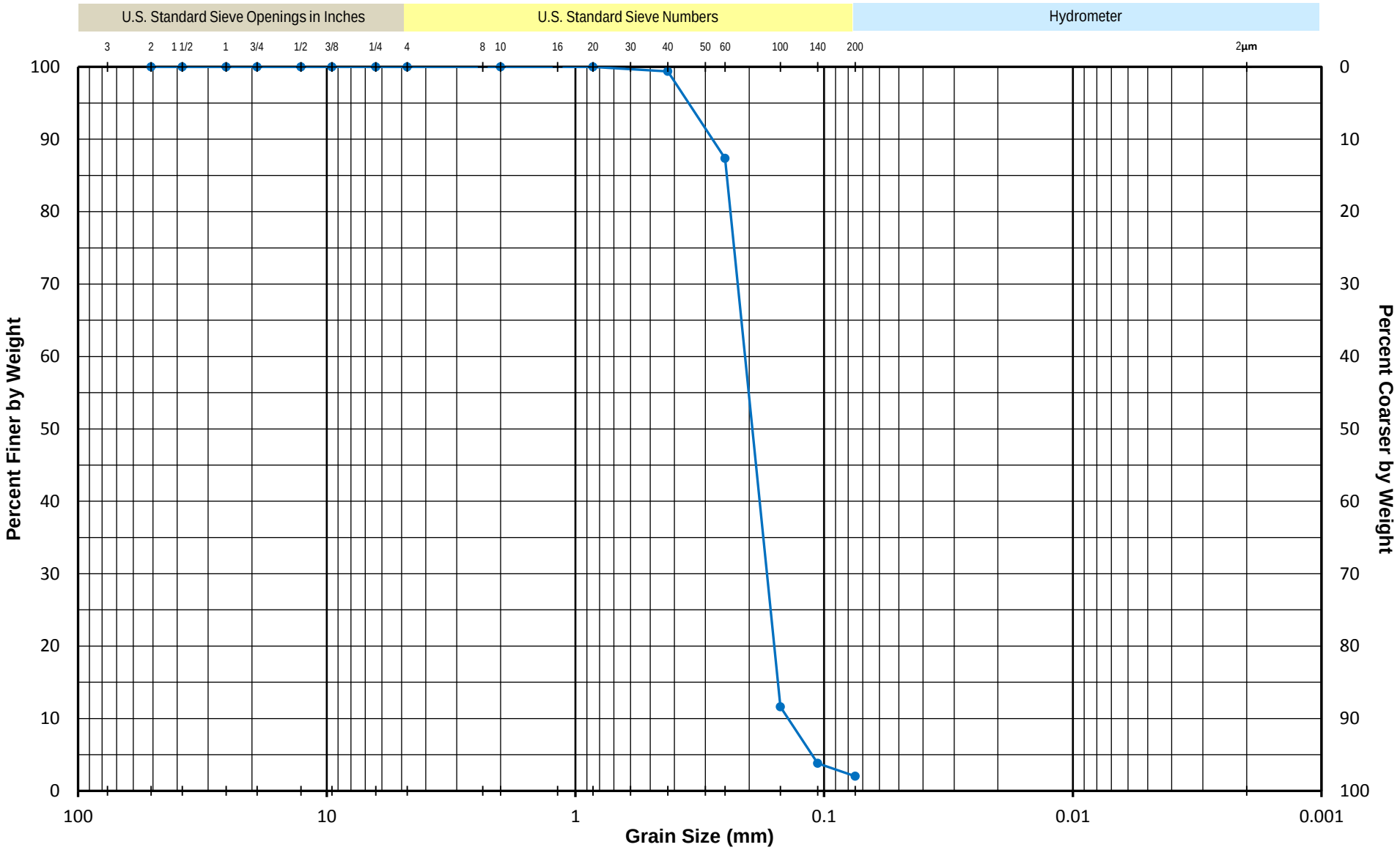


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC53 4-6	Very Dark Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

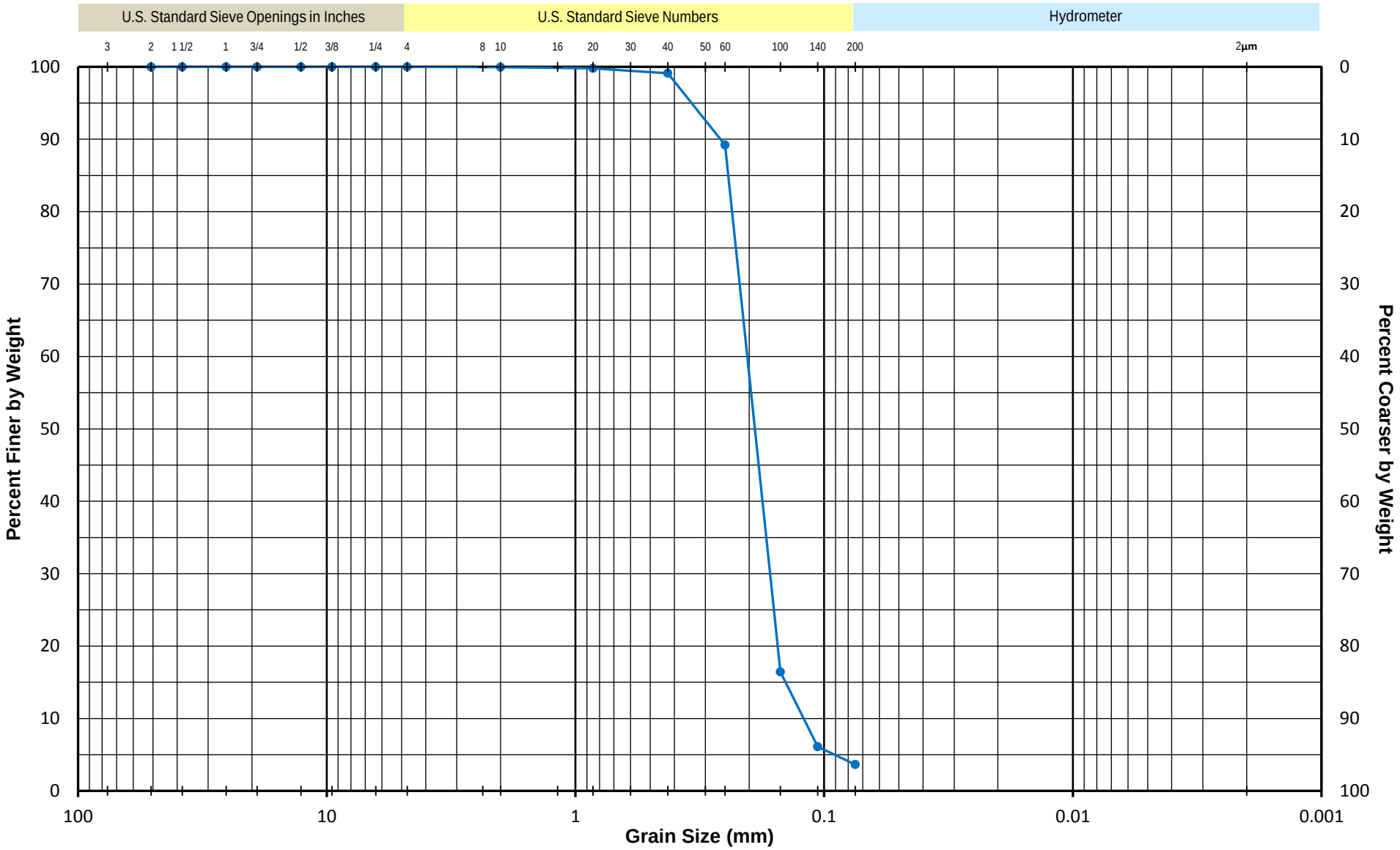


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC53 6-8	Very Dark Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

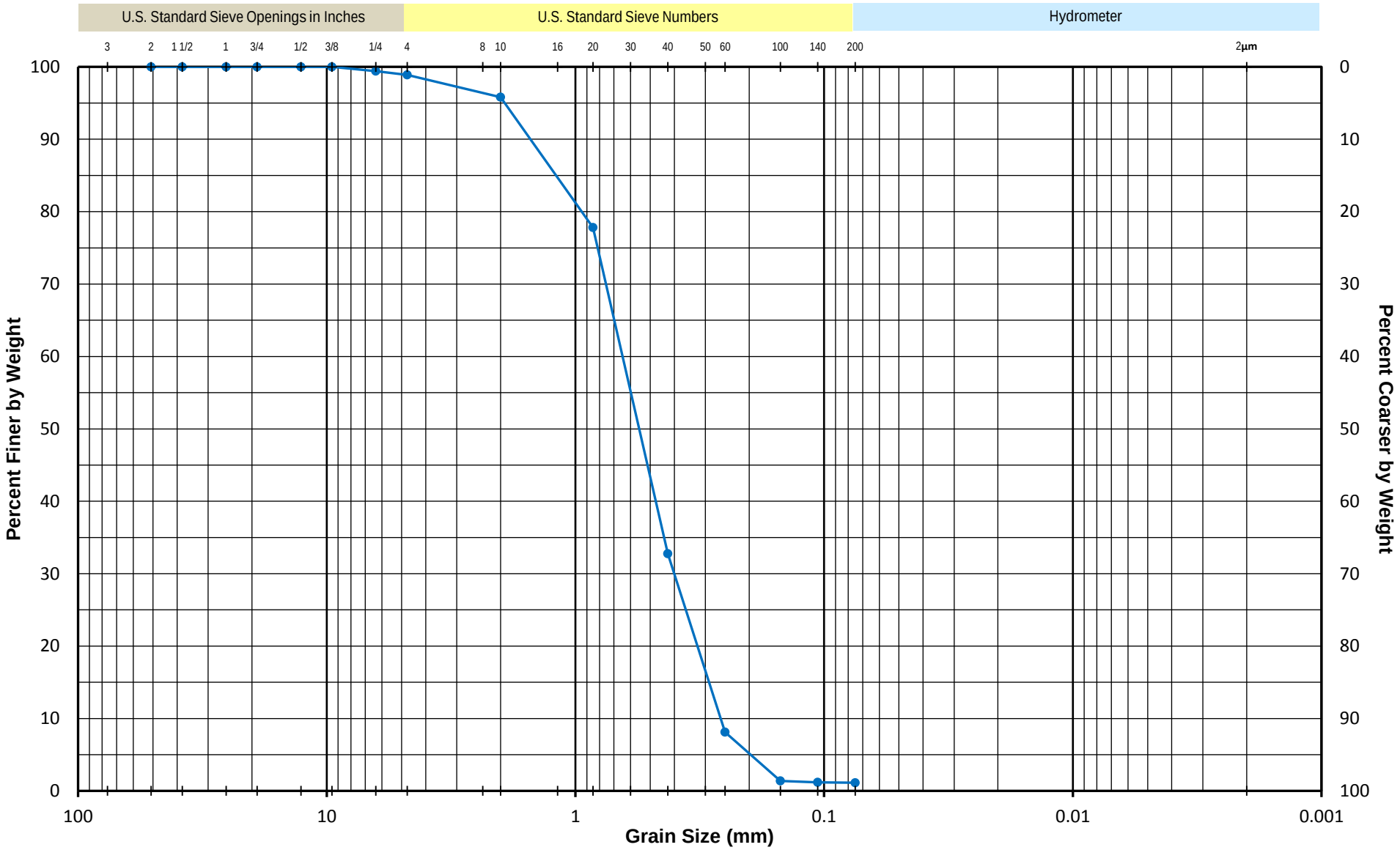


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC53 8-10	Very Dark Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

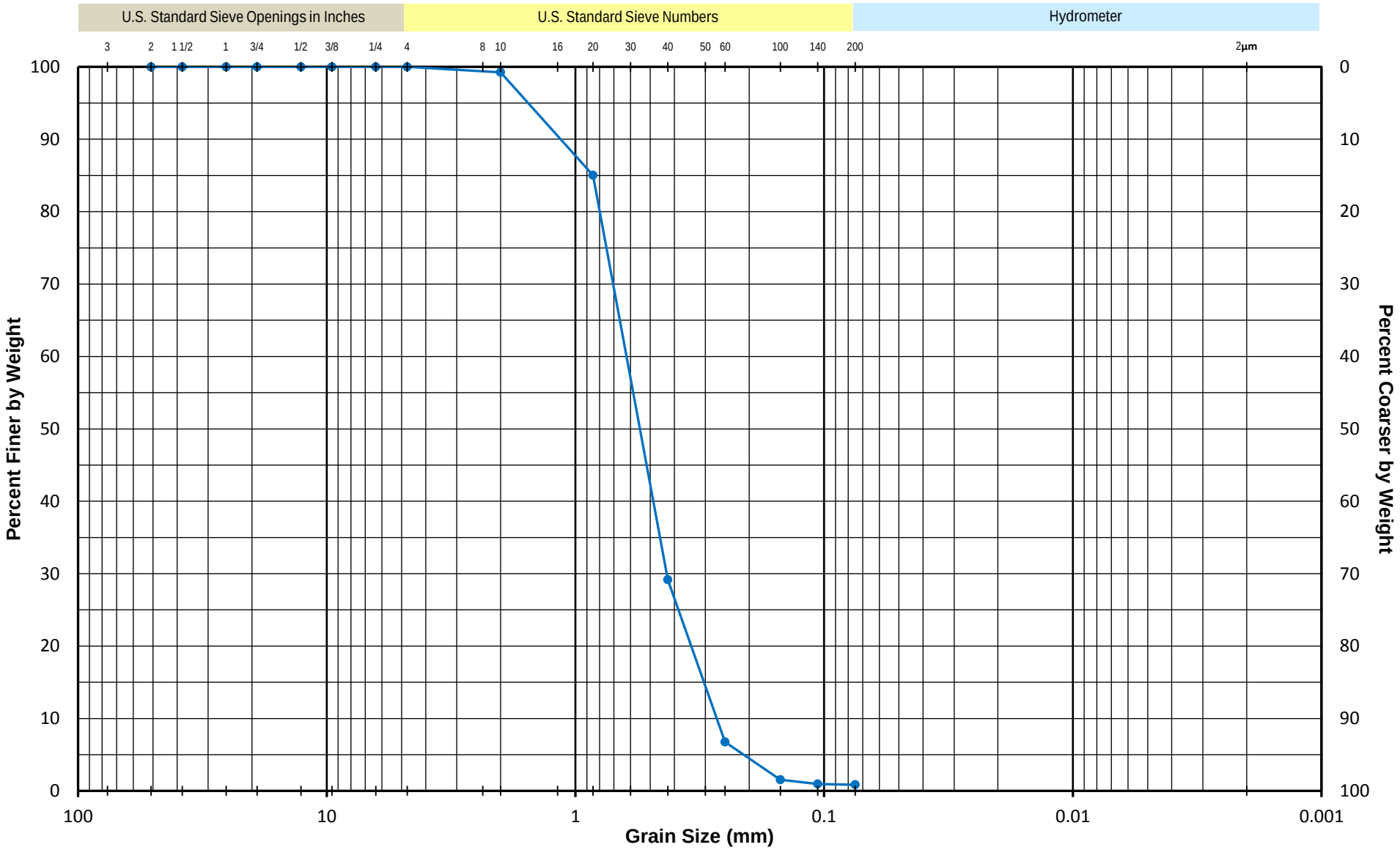


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC55 0-2	Grayish Brown Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

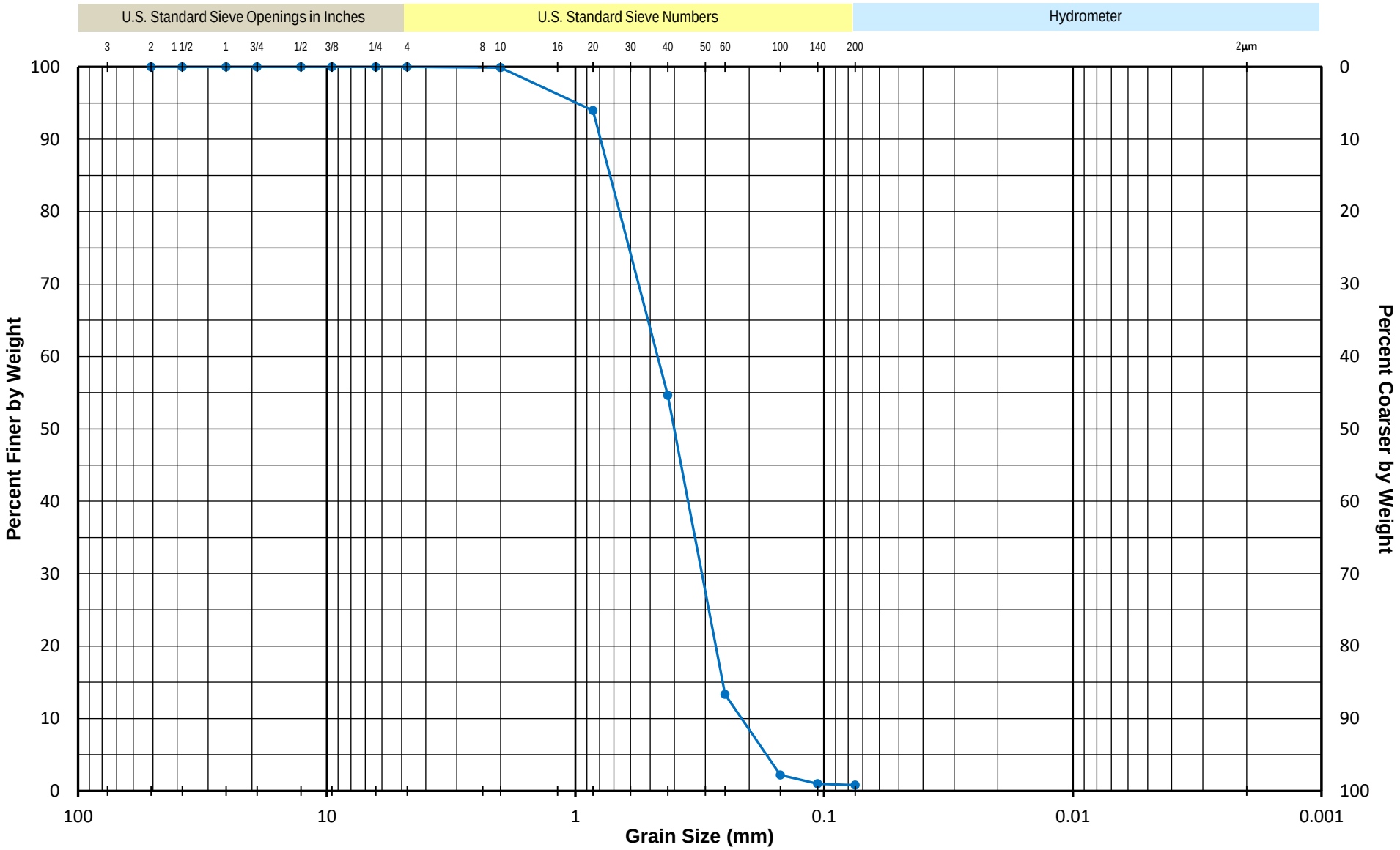


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC55 0-2	Gray Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

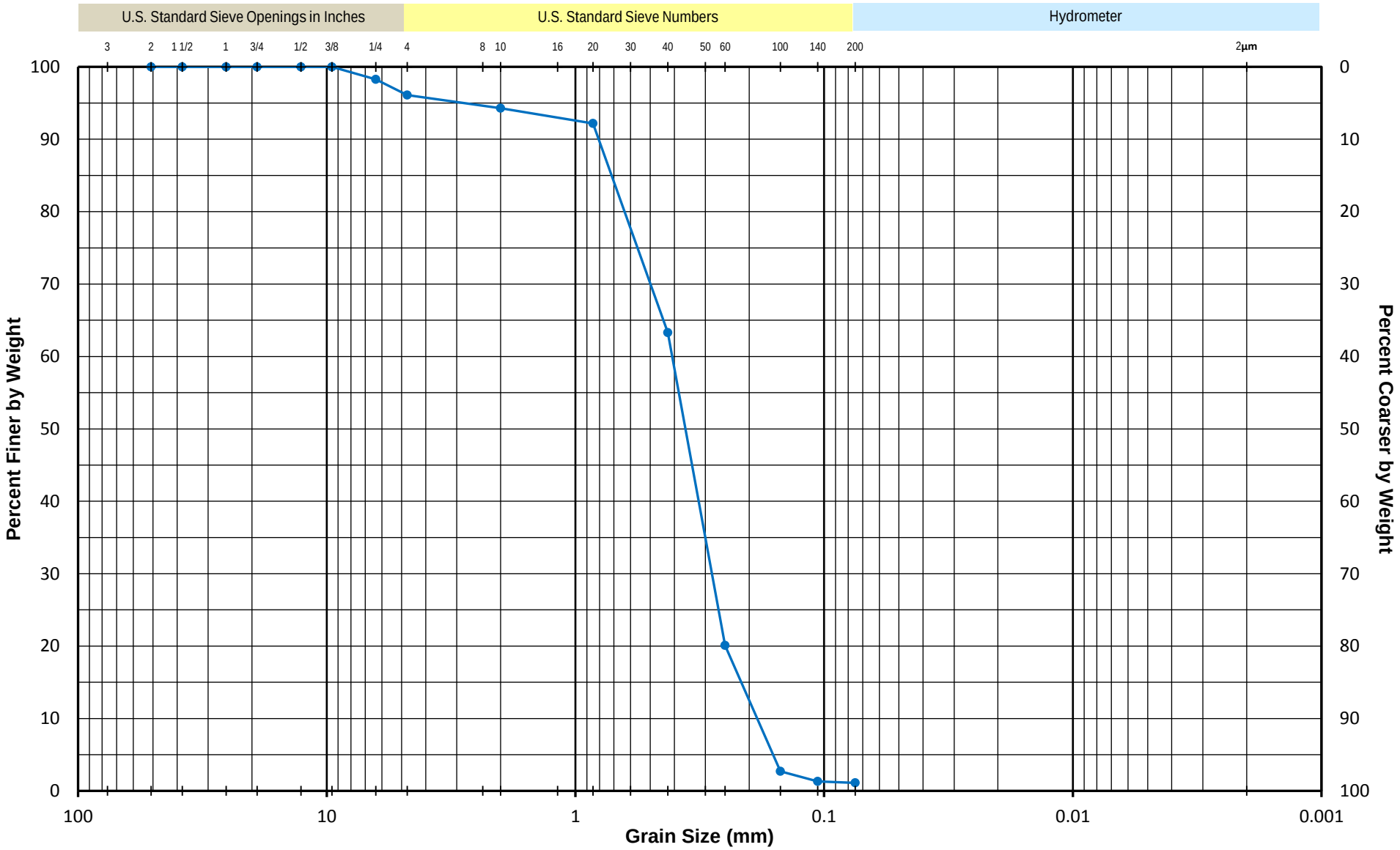


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC55 2-4	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

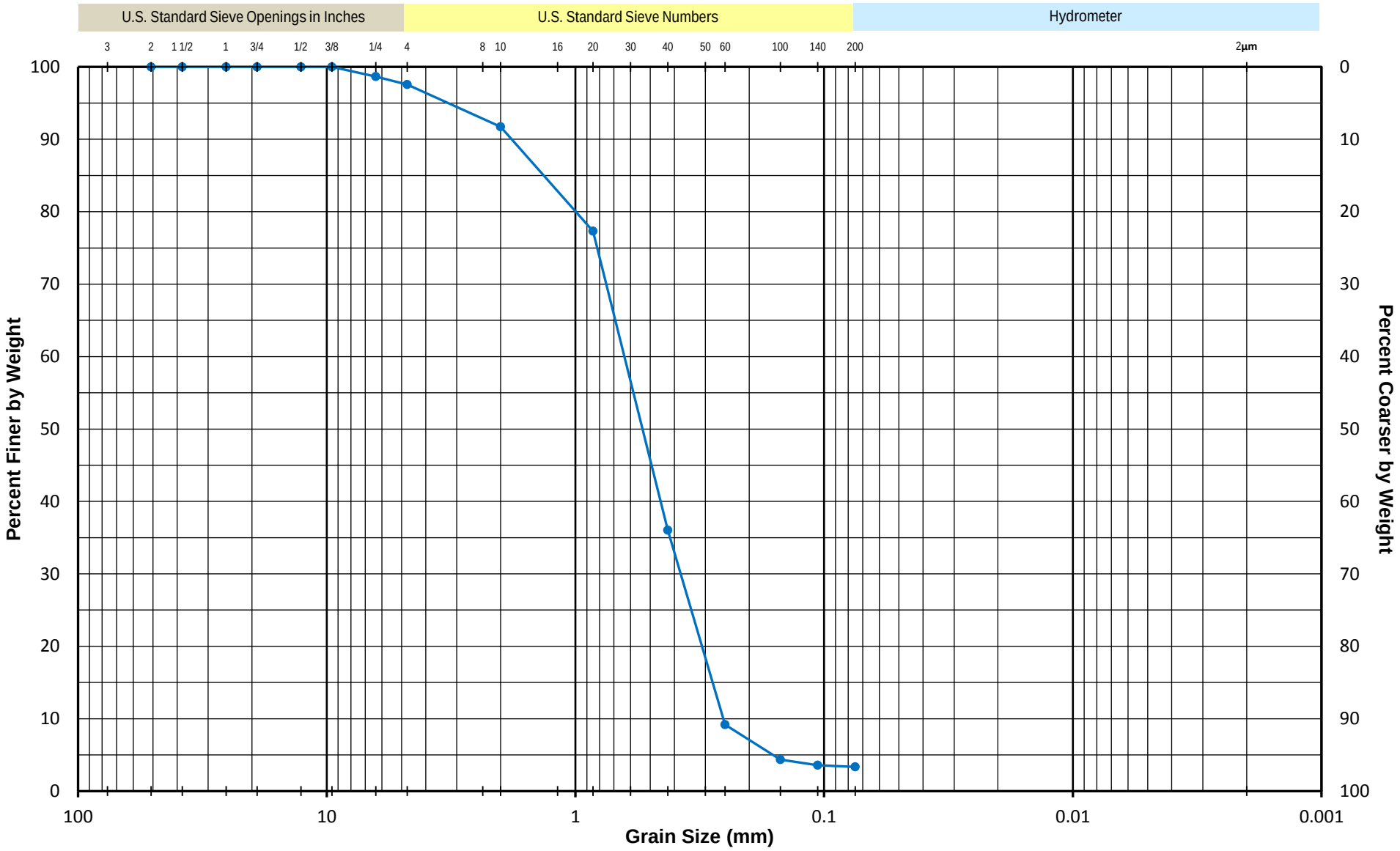


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC55 4-6	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

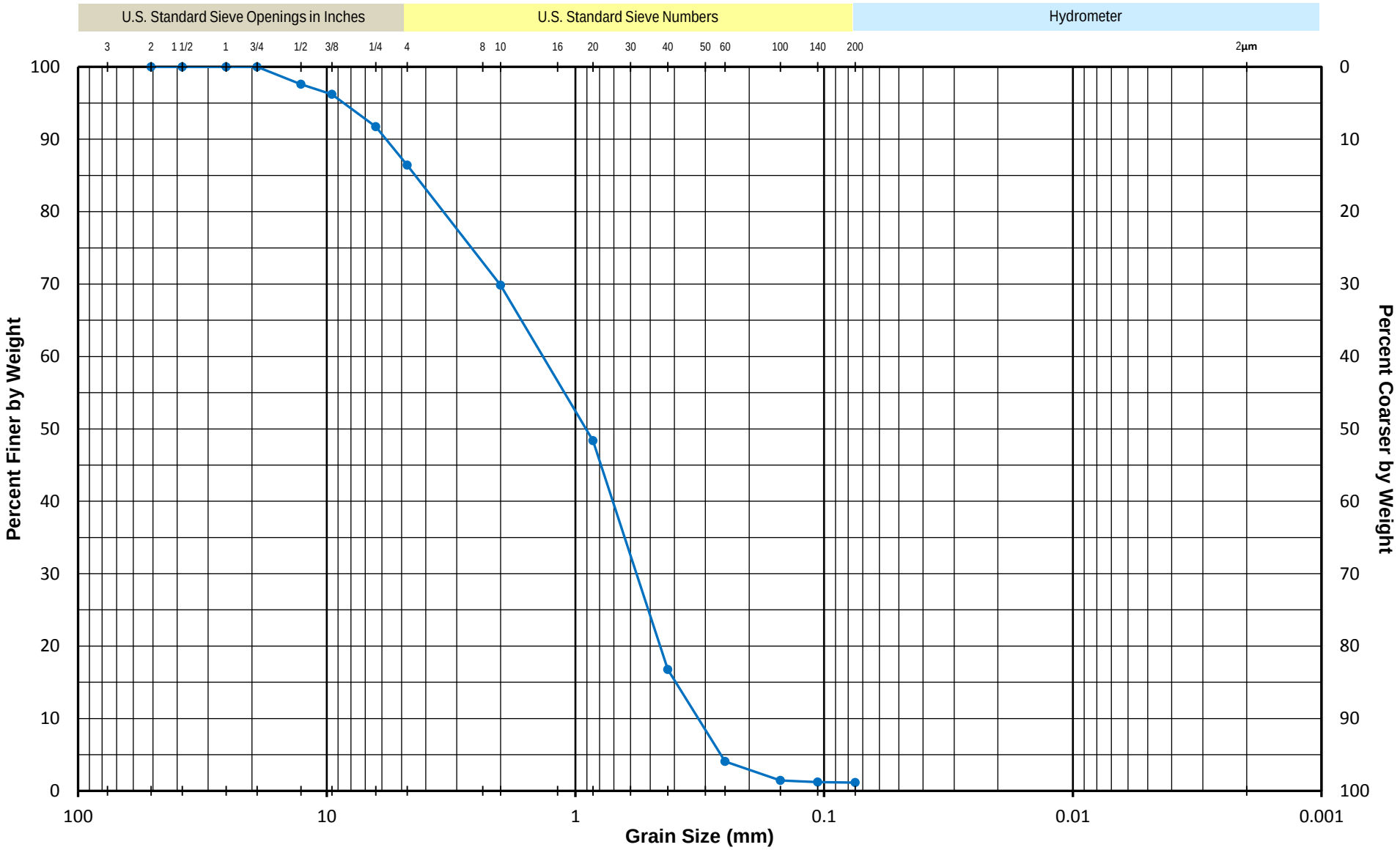


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC55 6-8	Pale Olive Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

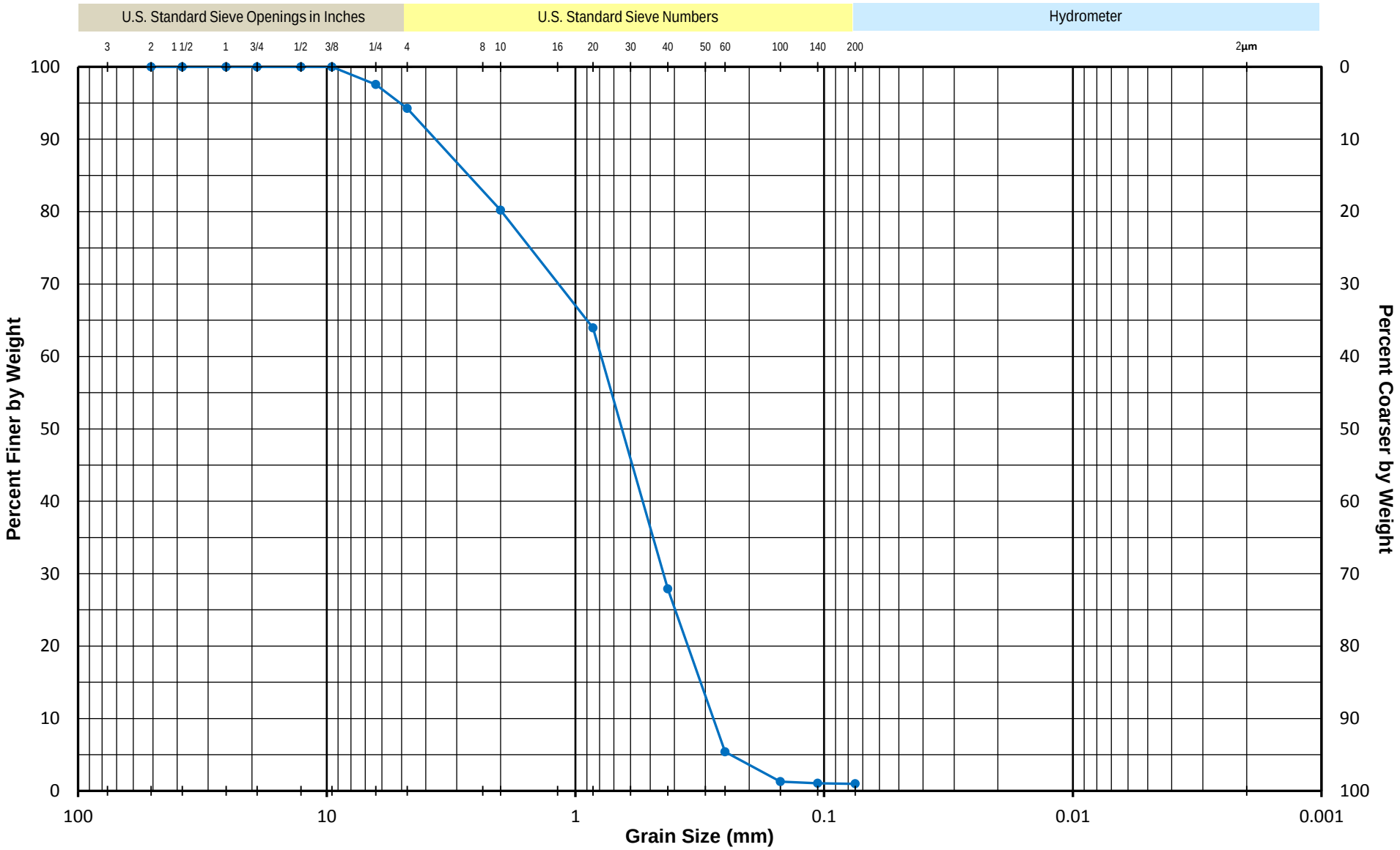


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC55 8-10	Pale Olive Medium to Coarse Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

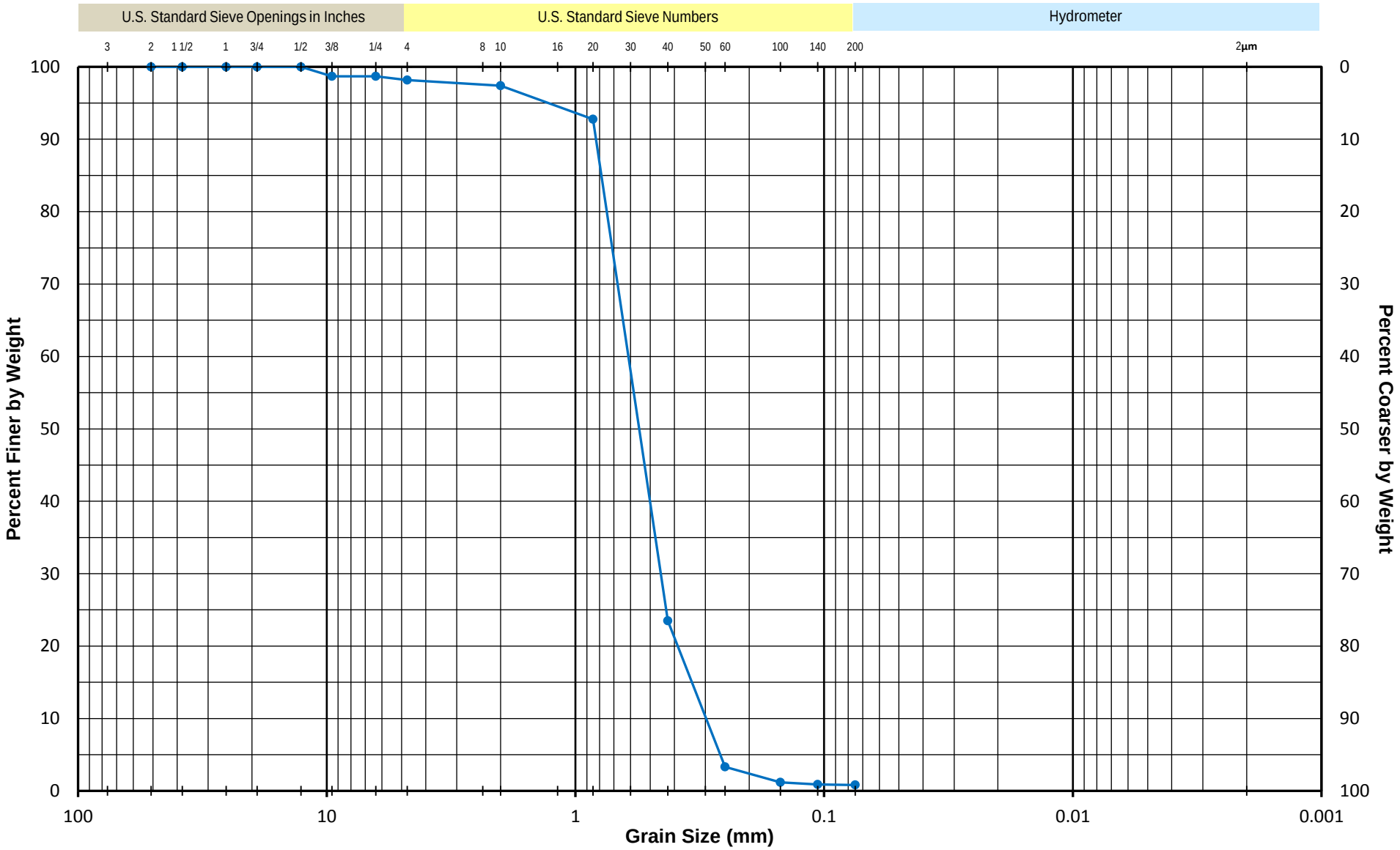


Test Depth	Core ID Section/Sample	Material Description
11.9 ft	VC55 10-12	Pale Olive Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

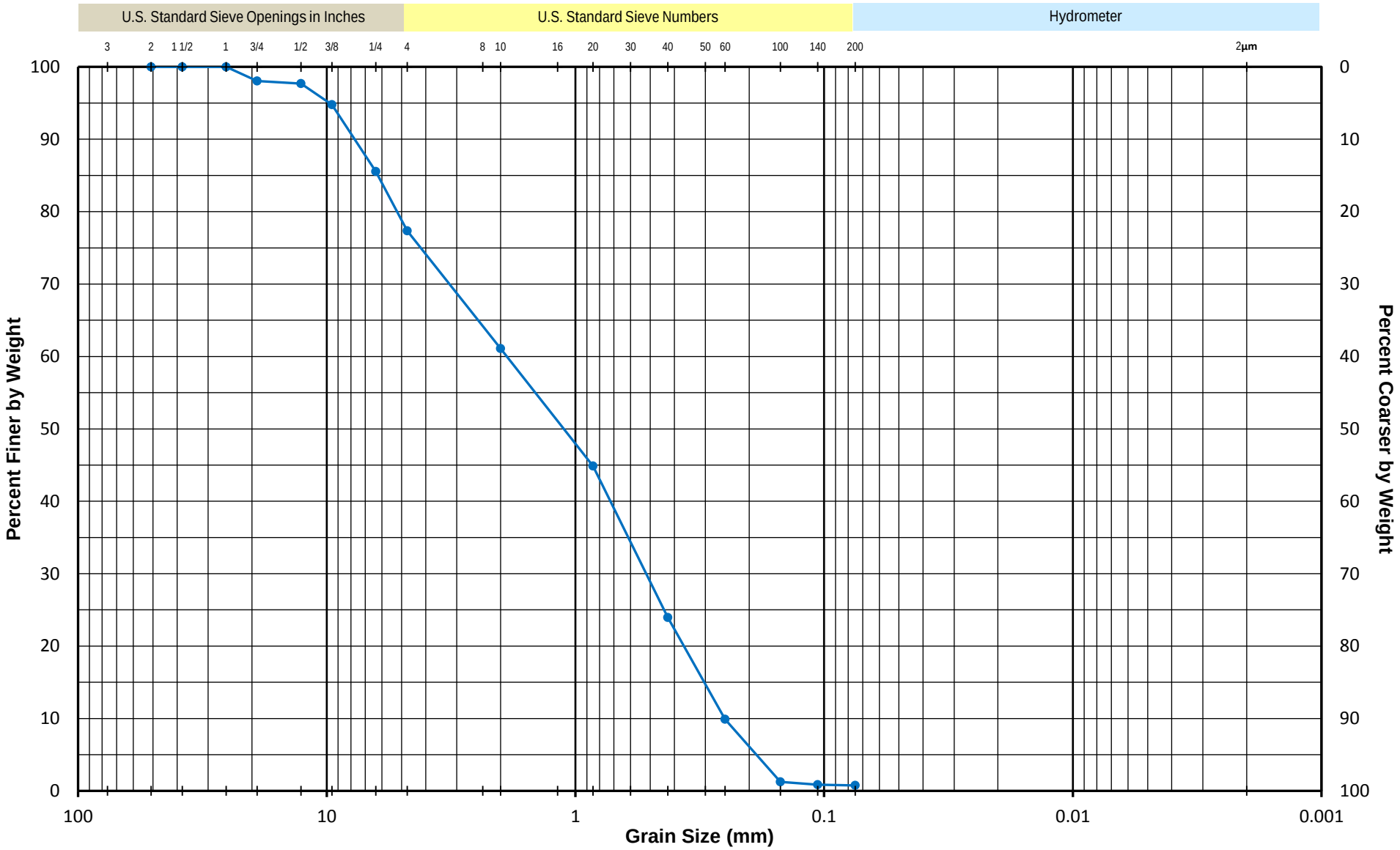


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC56 0-2	Brown and Gray Medium to Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

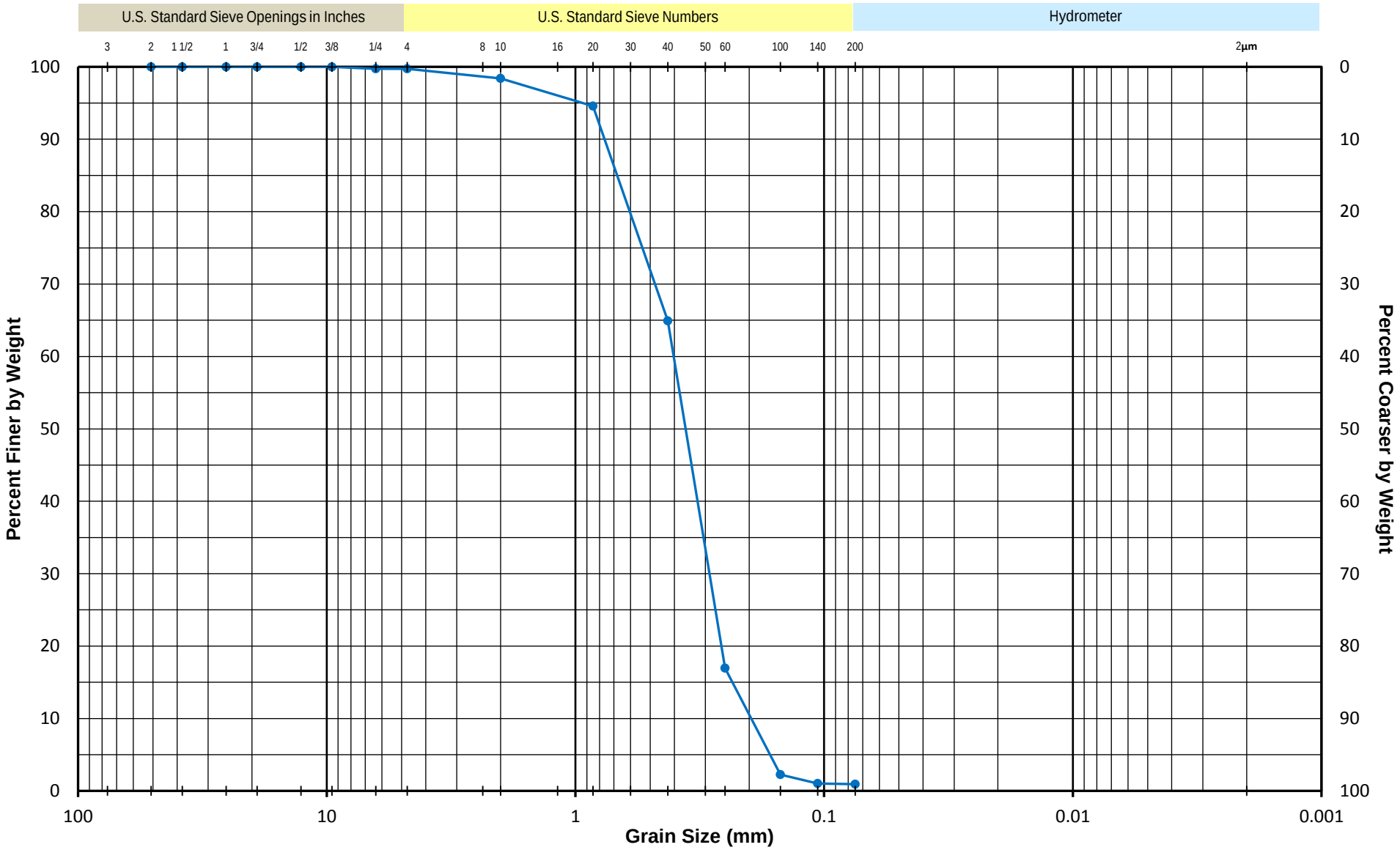


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC56 0-2	Light Yellowish Brown Medium to Fine Sand with gravel

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

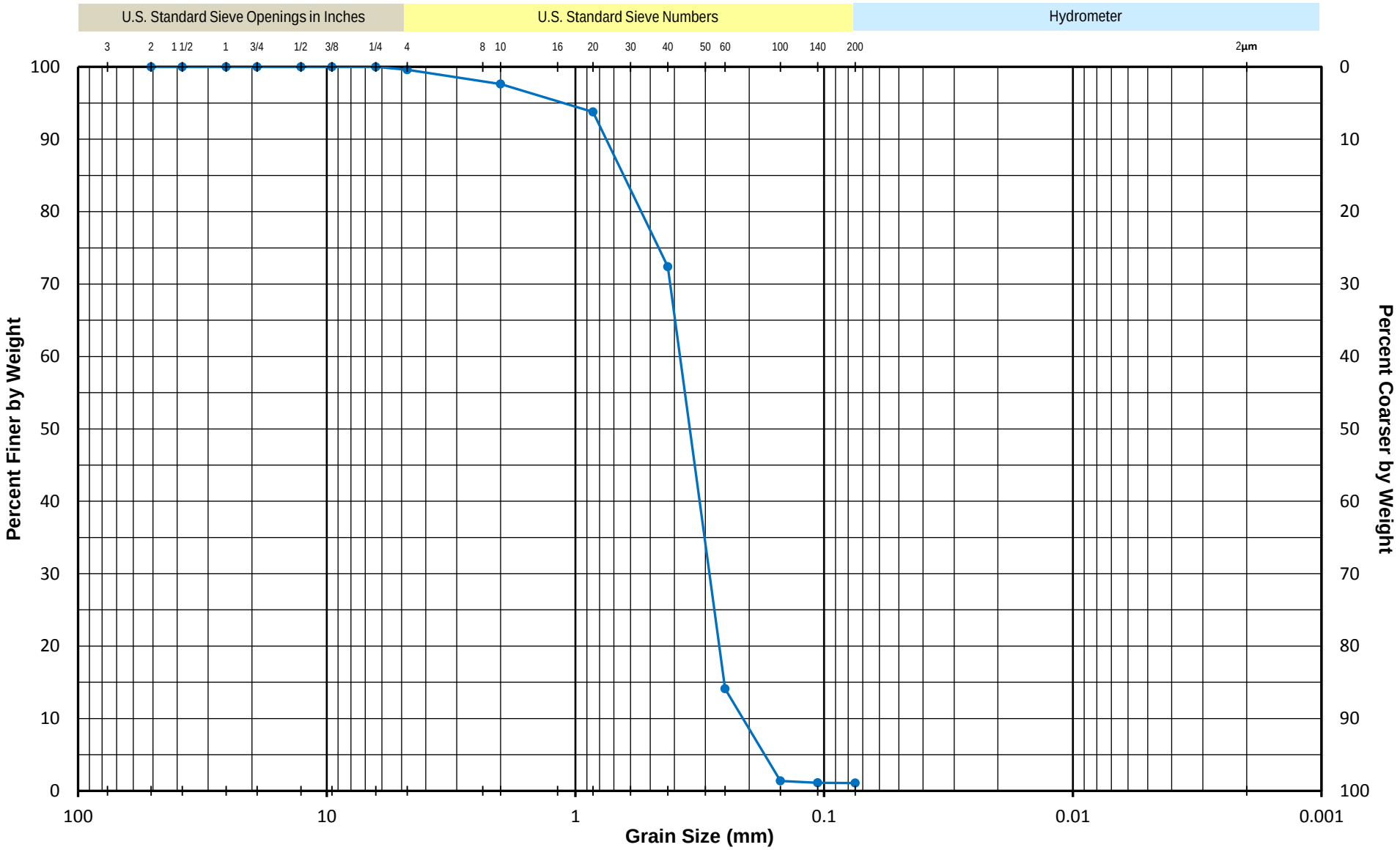


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC56 2-4	Light Brownish Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

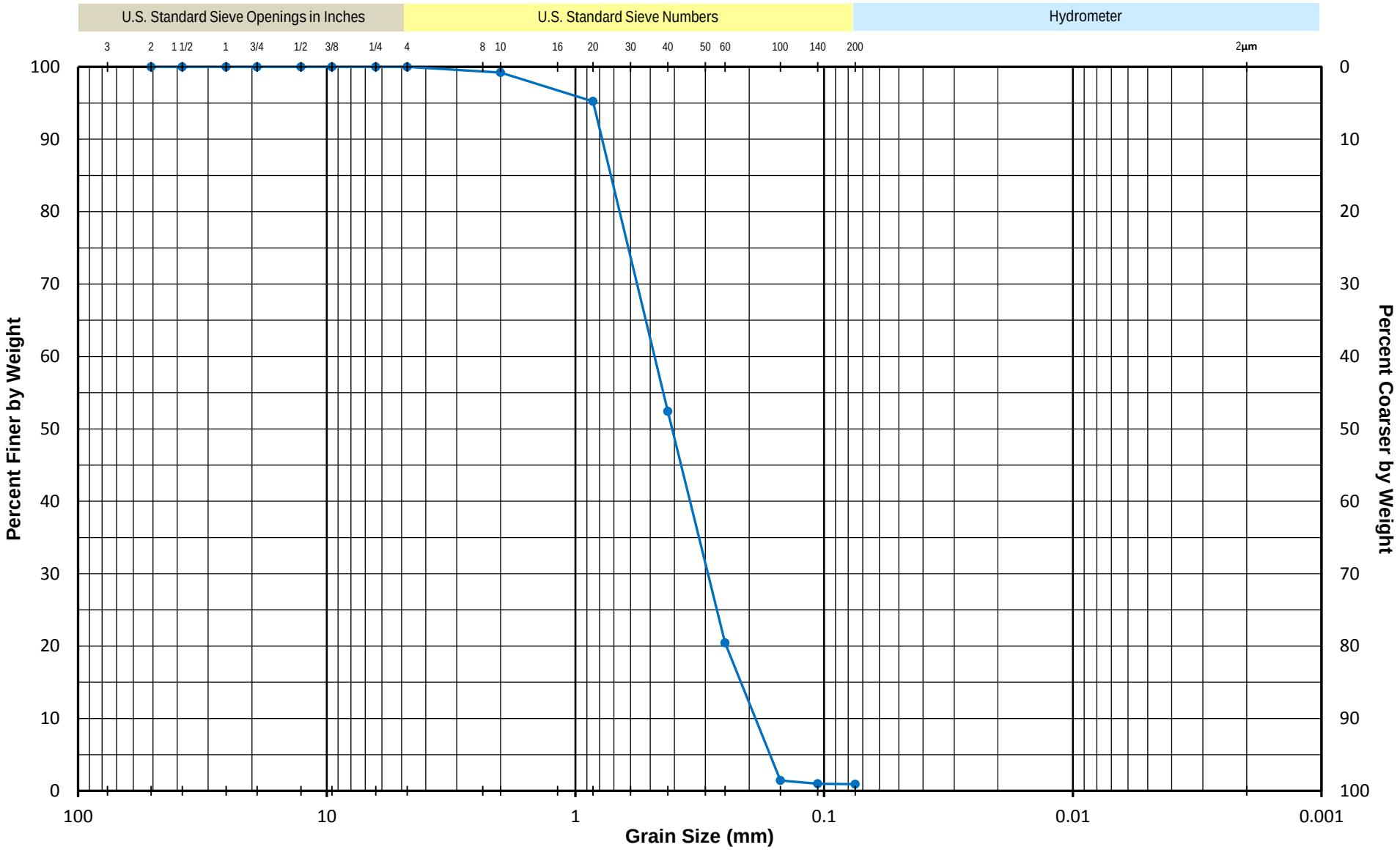


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC56 4-6	Light Brownish Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

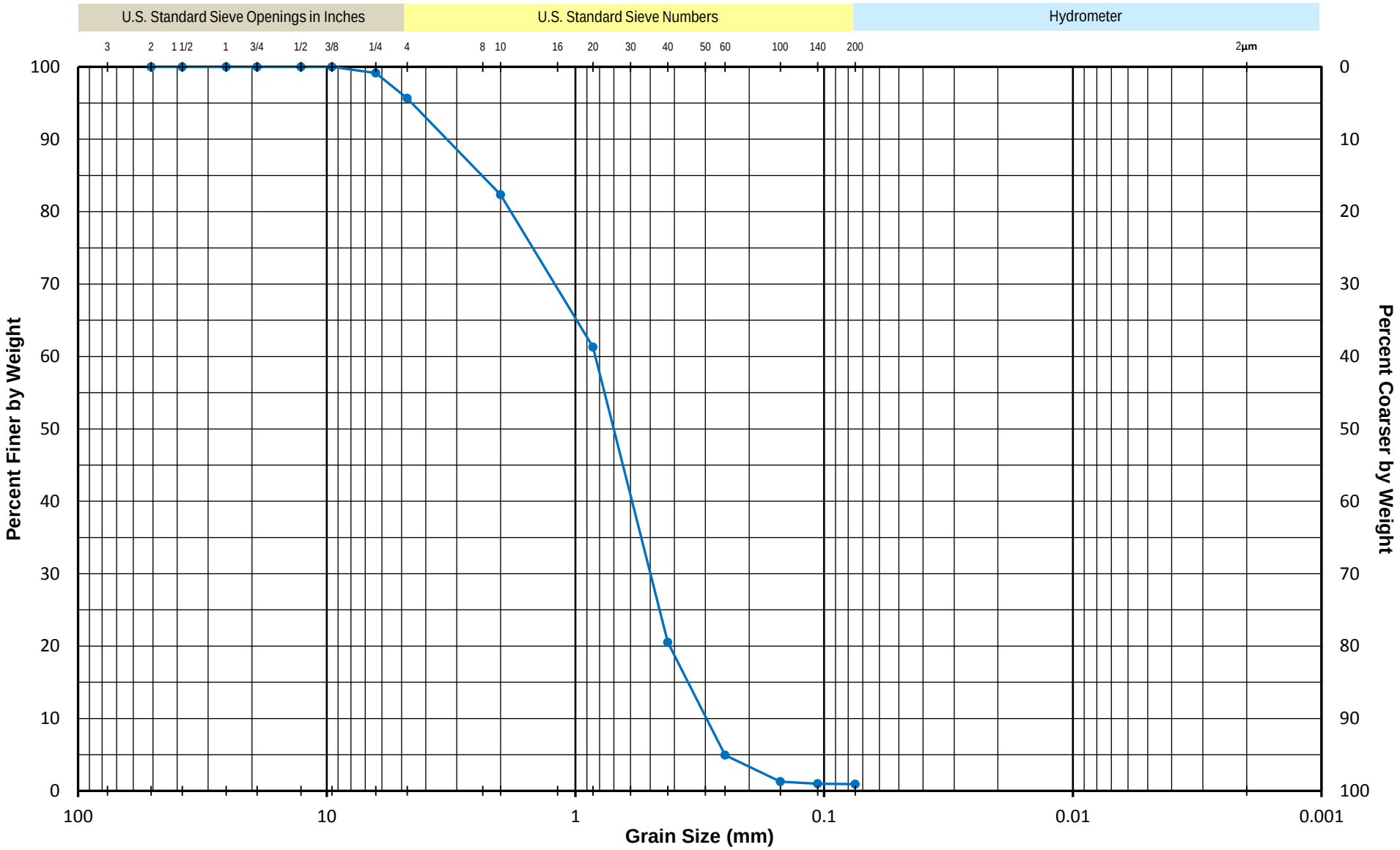


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC56 6-8	Gray and Brownish Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

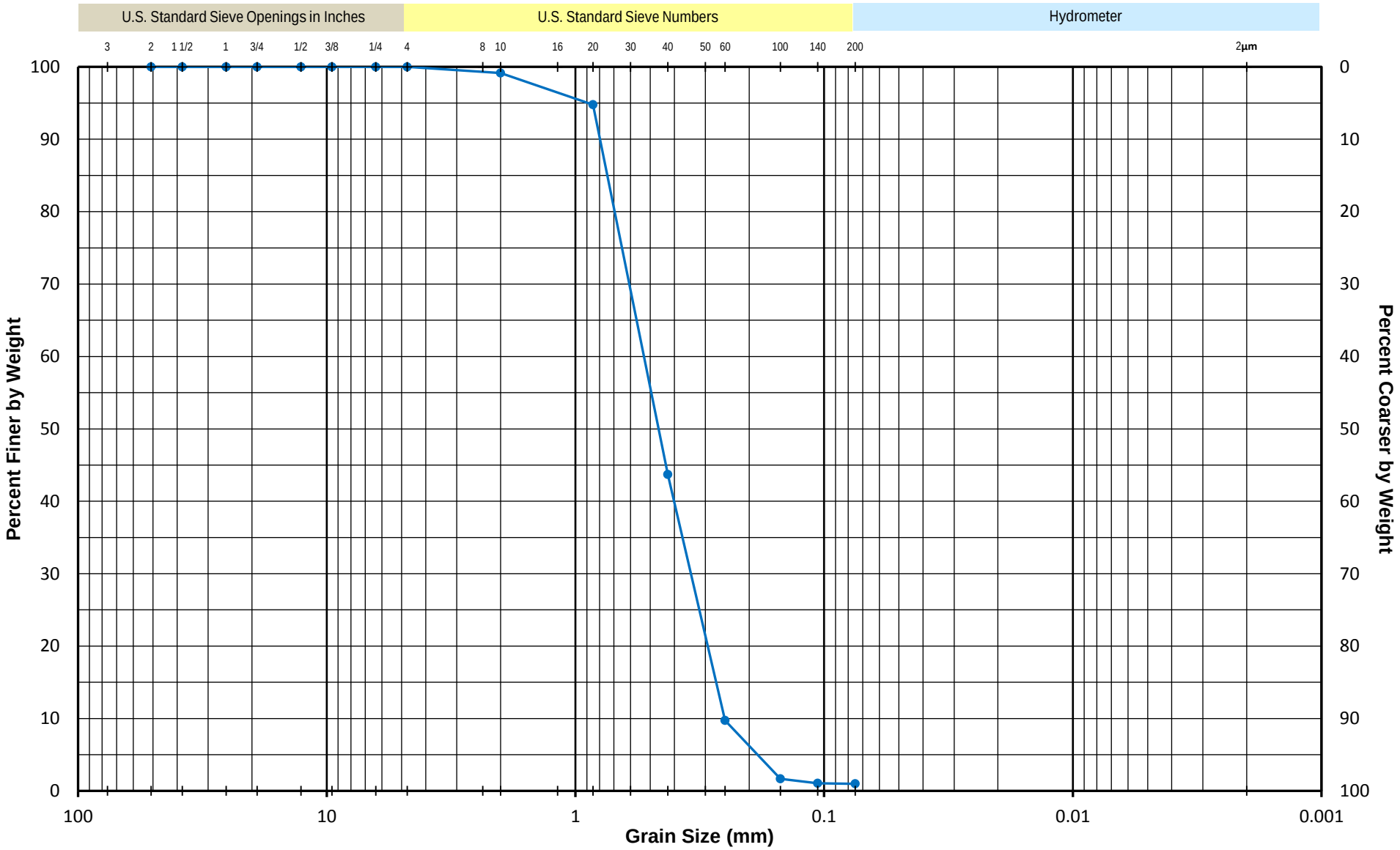


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC56 8-10	Brownish Gray Fine to Coarse Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

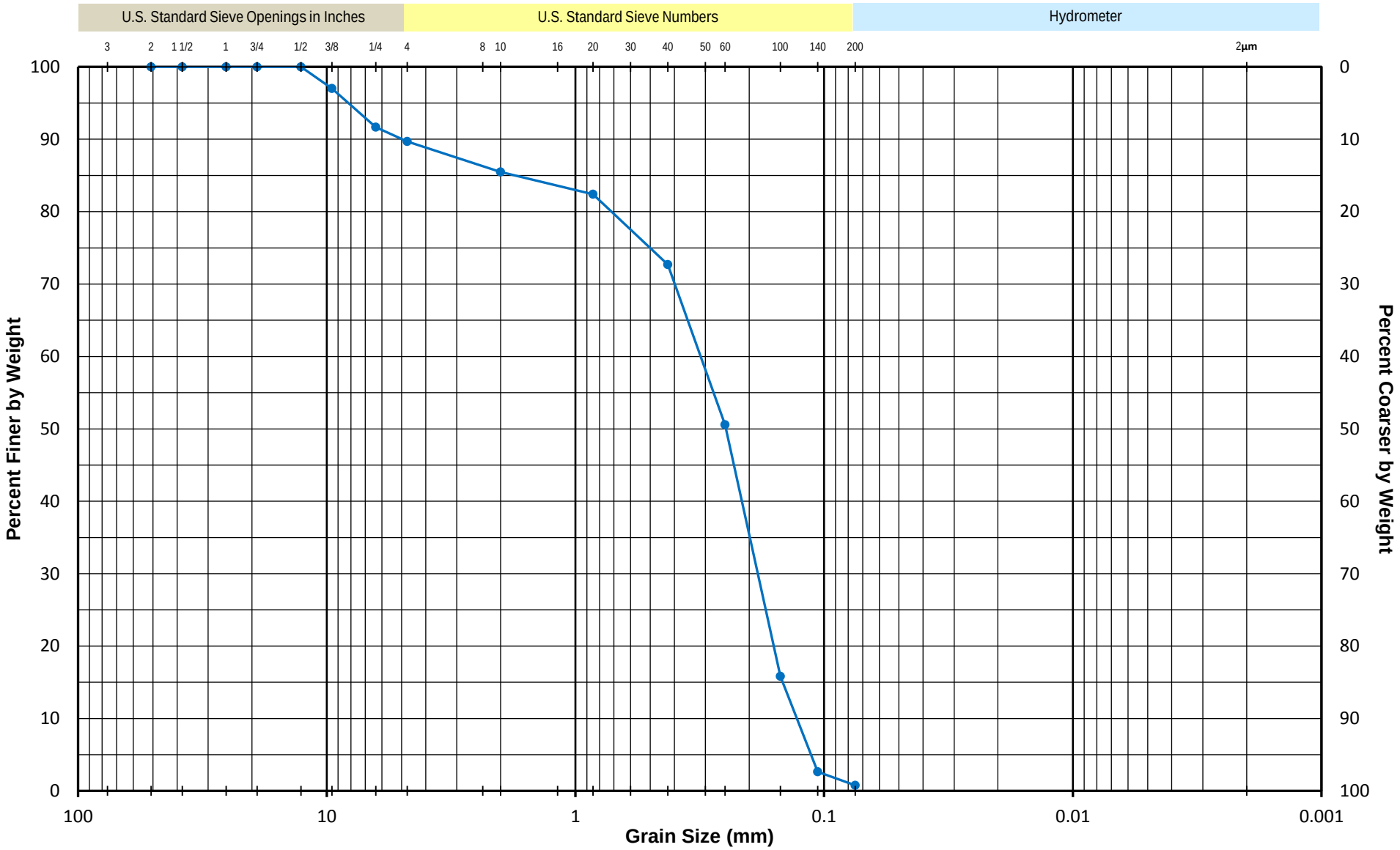


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC56 10-12	Brownish Gray Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

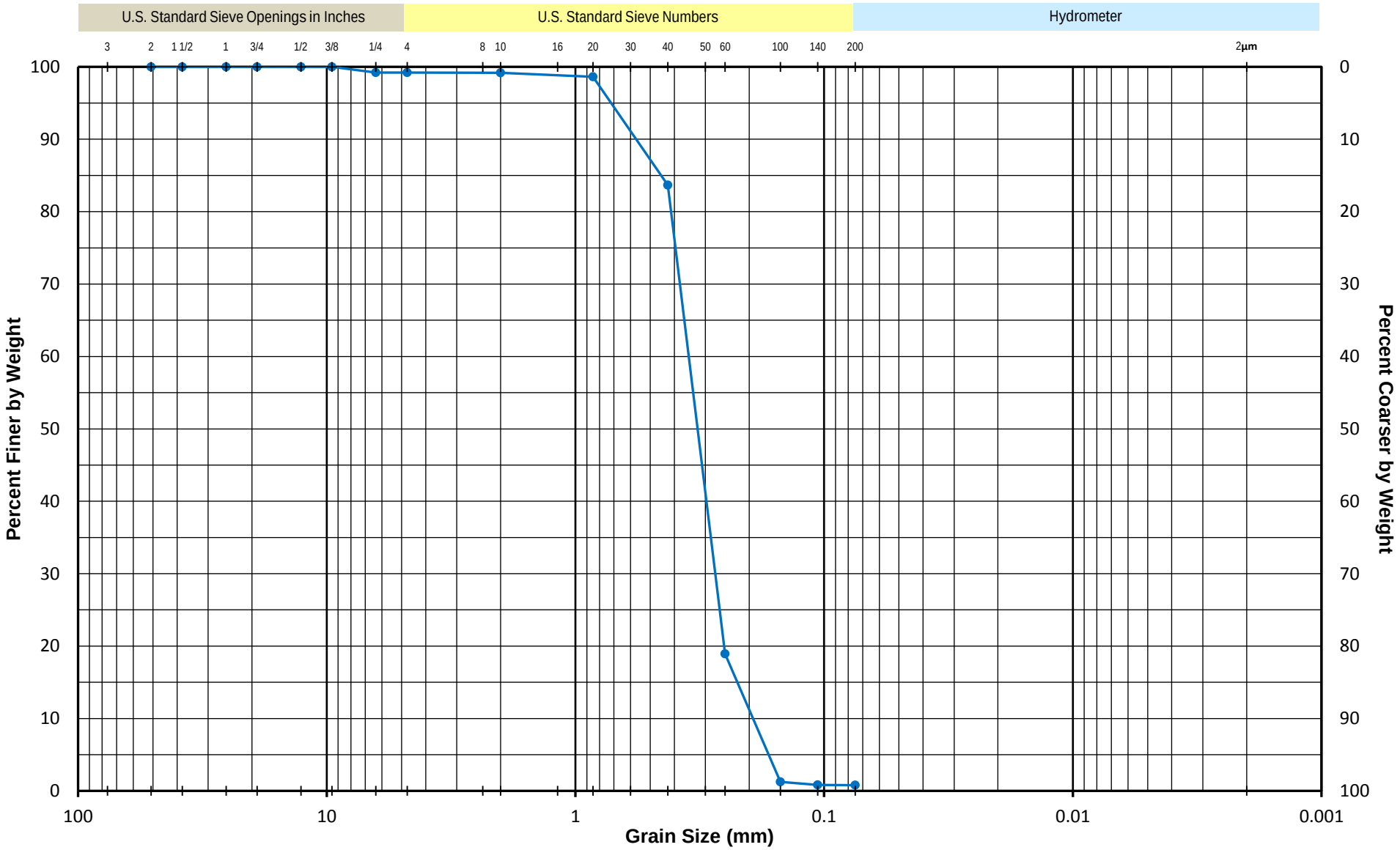


Test Depth	Core ID Section/Sample	Material Description
0.2 ft	VC58 0-2	Black and Olive Brown Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

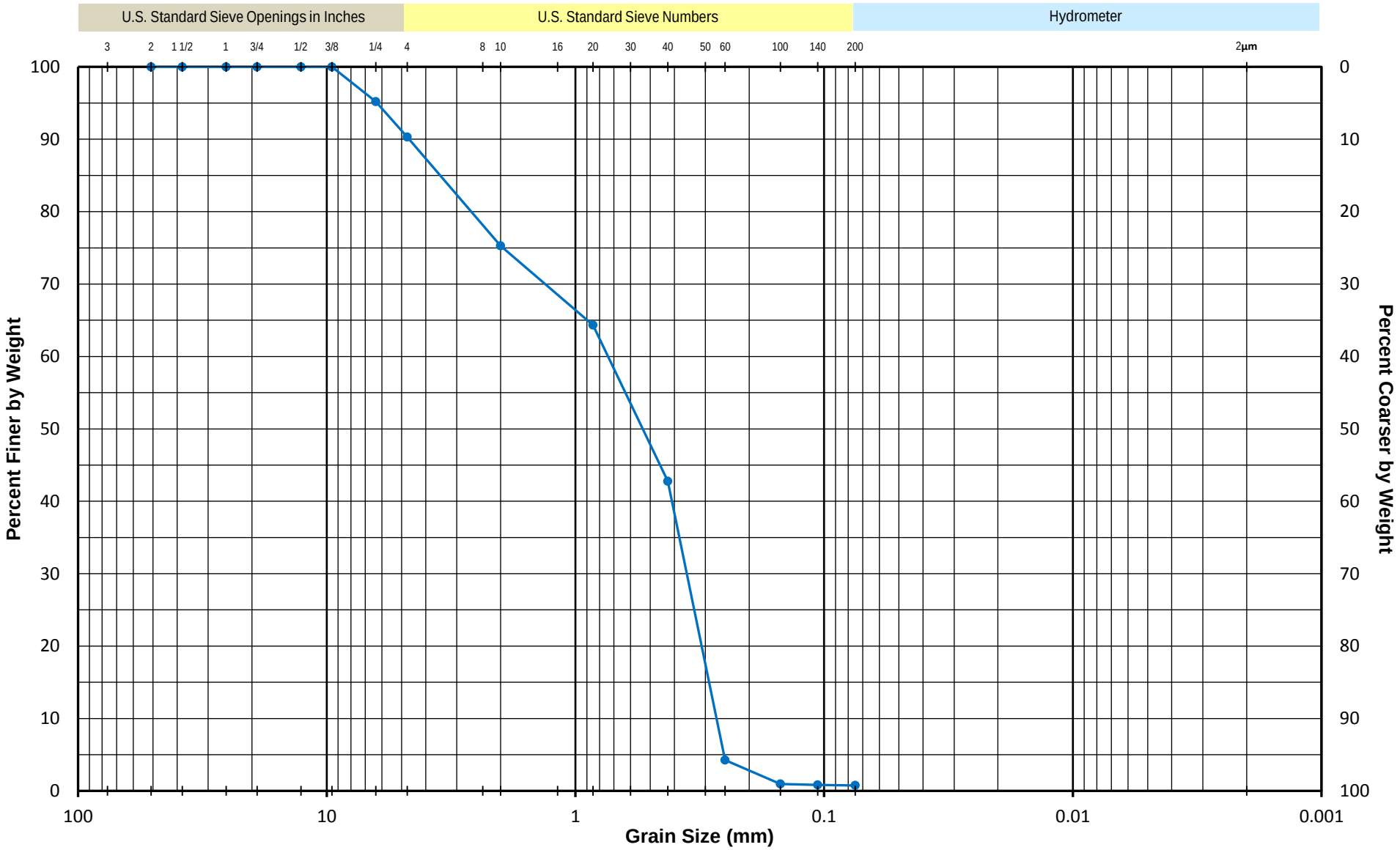


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC58 0-2	Dark Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

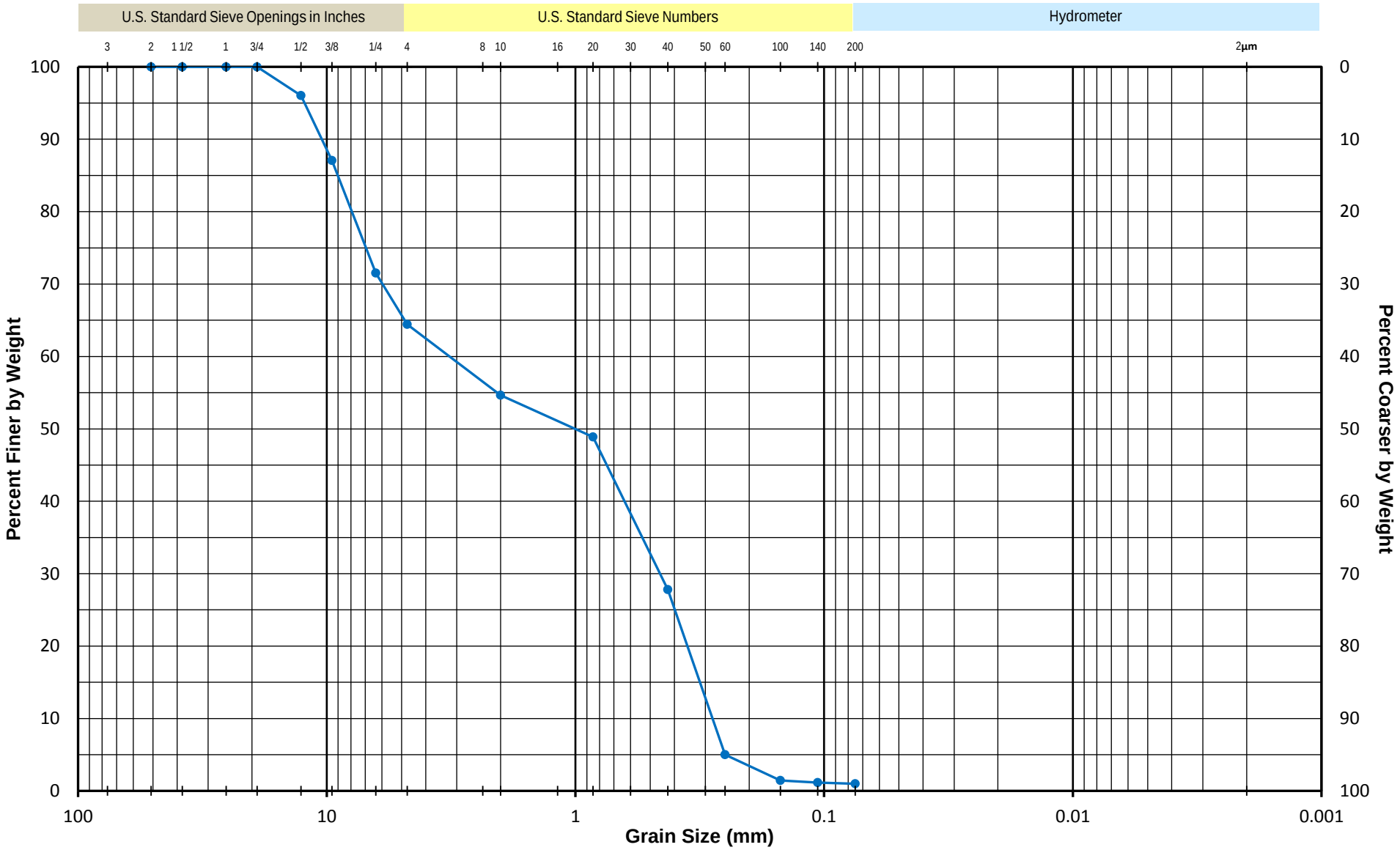


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC58 2-4	Dark Gray Fine to Coarse Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

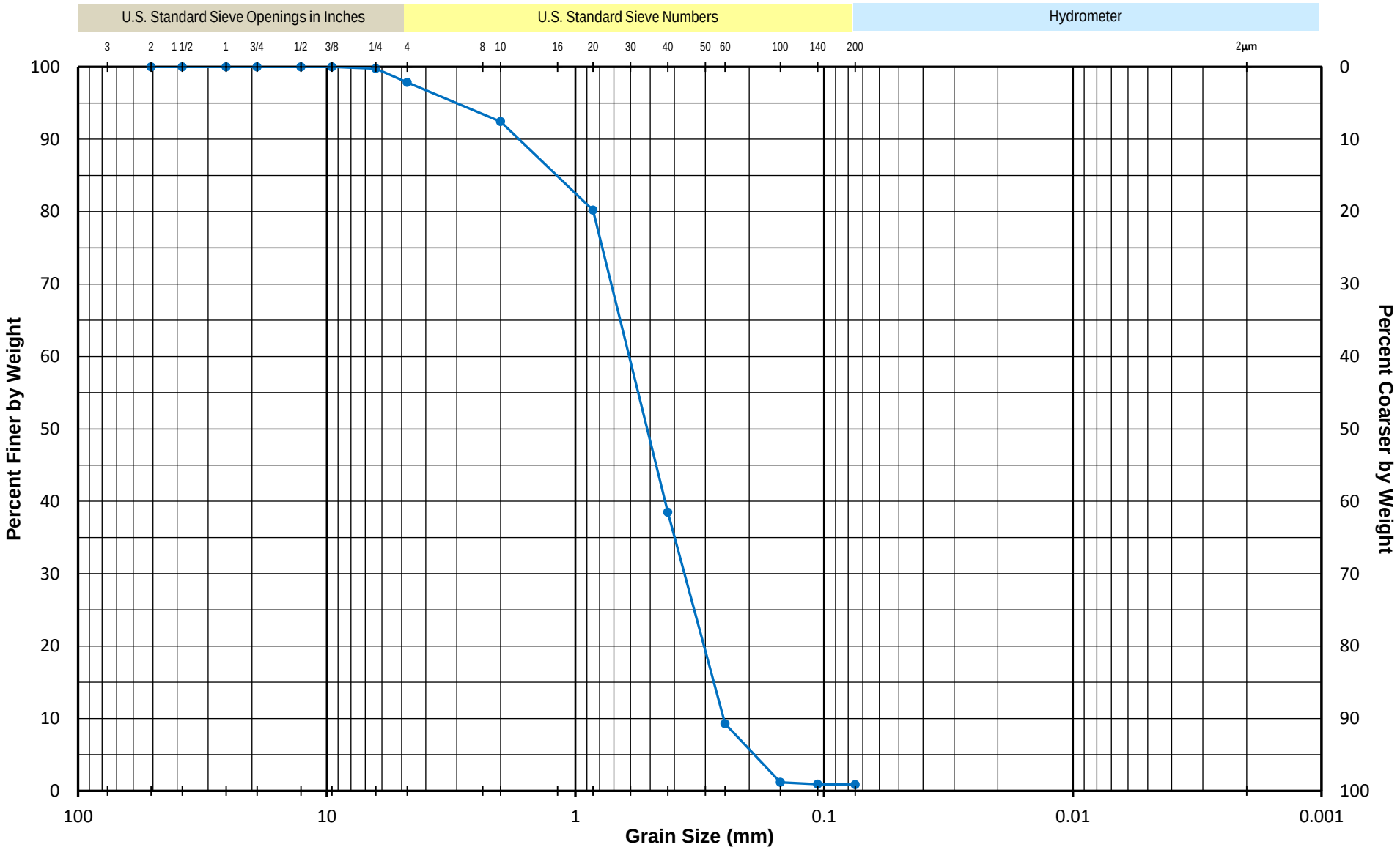


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC58 4-6	Dark Gray Fine to Coarse Sand with gravel

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

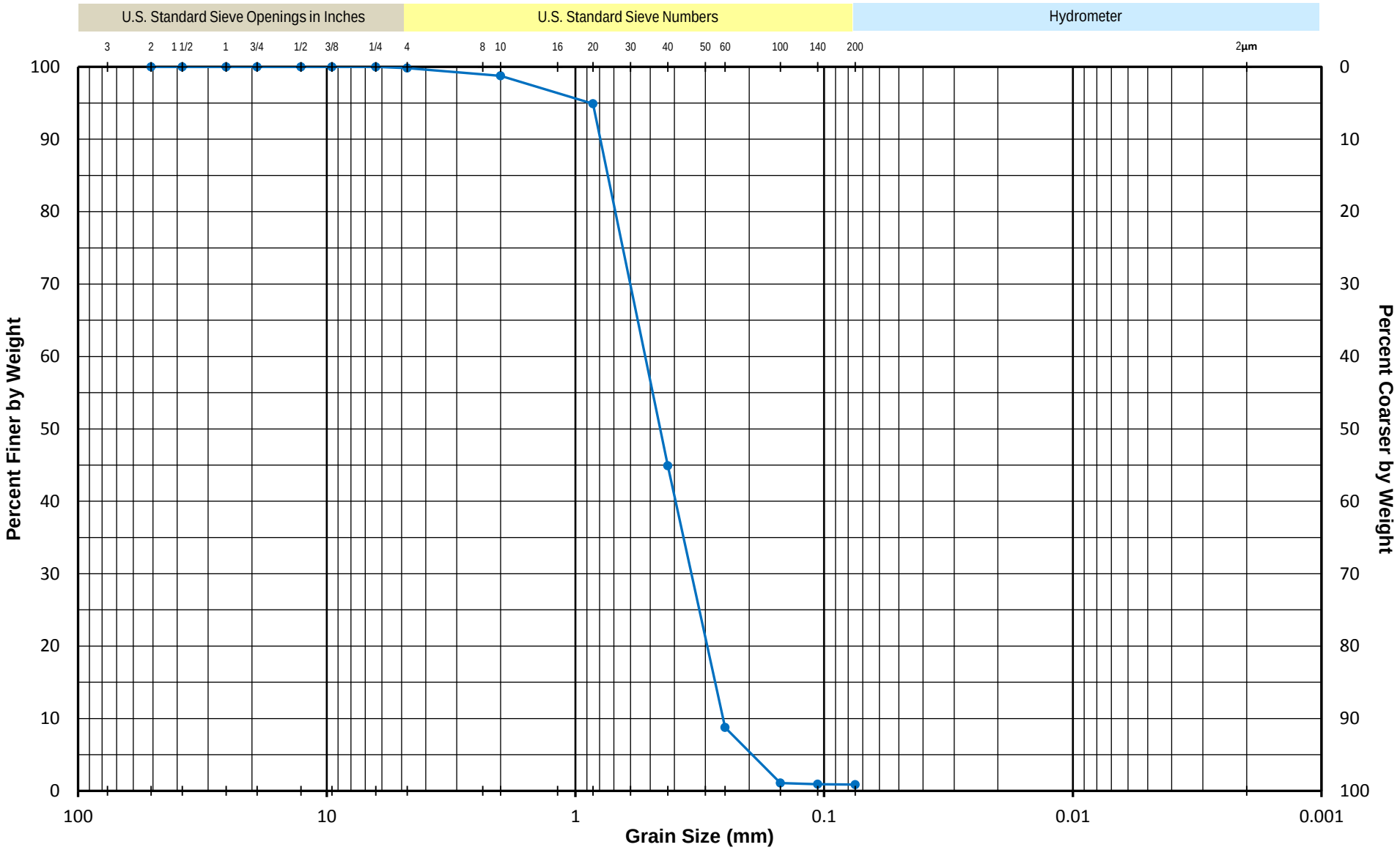


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC58 6-8	Dark Gray Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

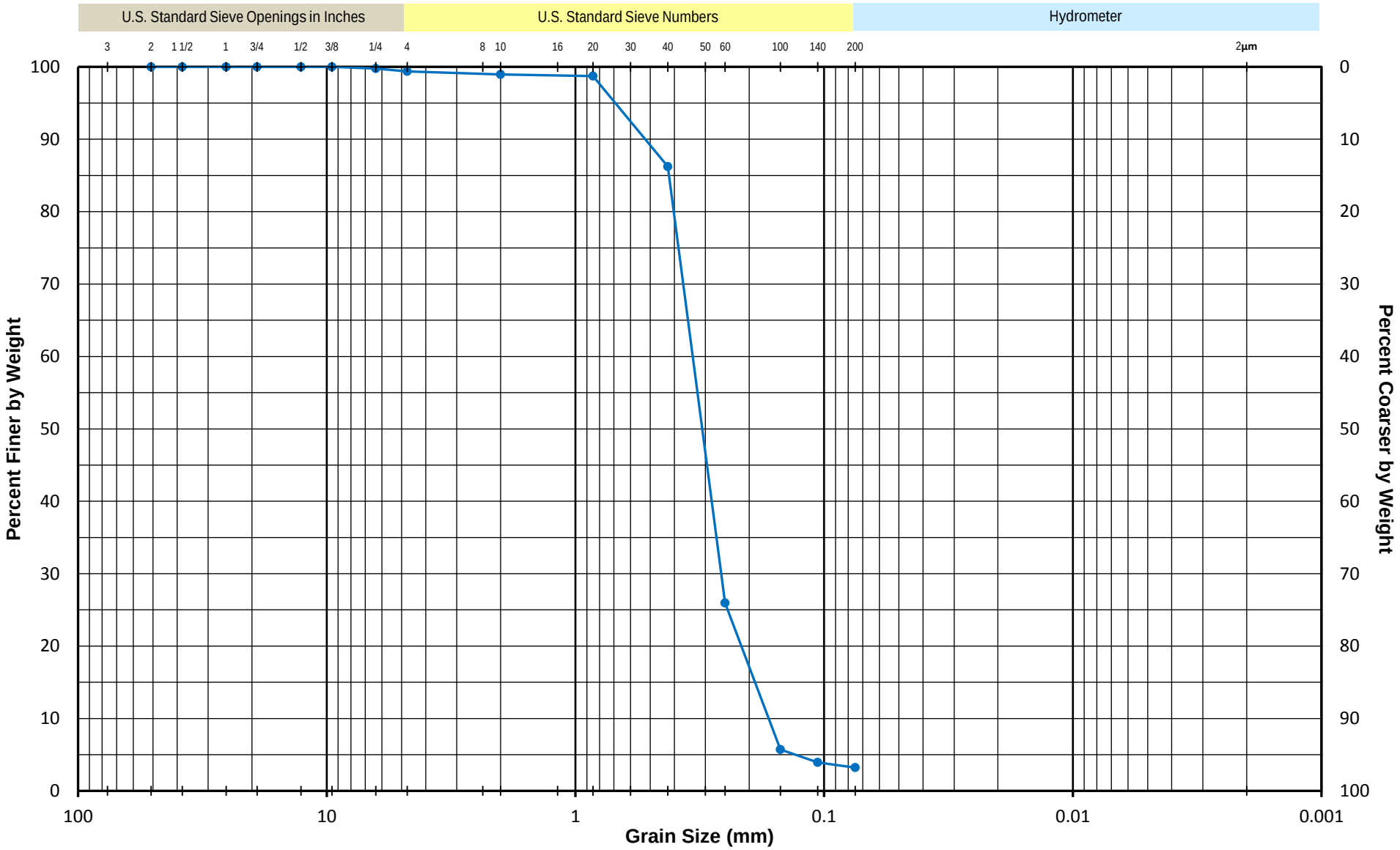


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC58 8-10	Dark Gray Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

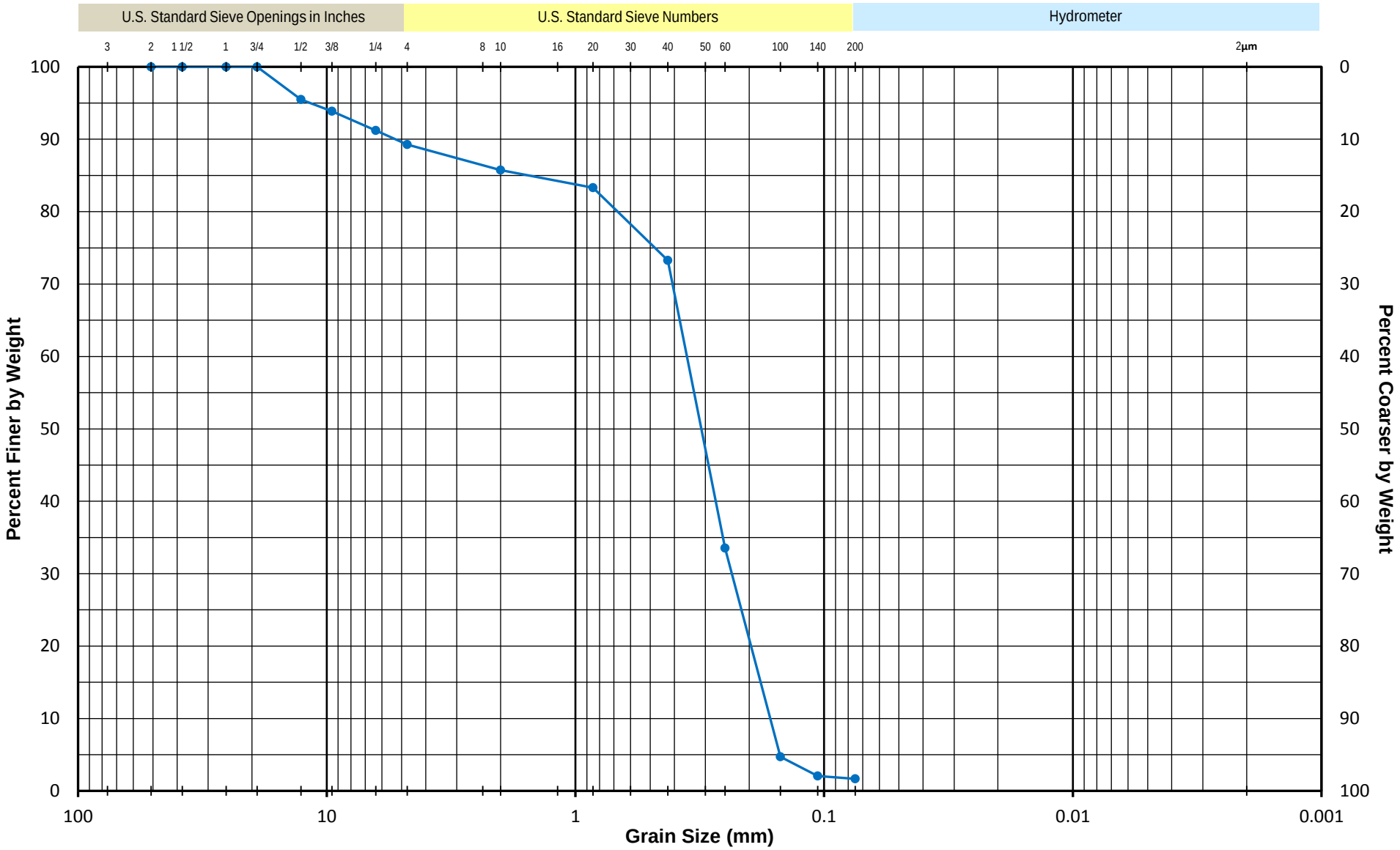


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC58 10-12	Dark Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

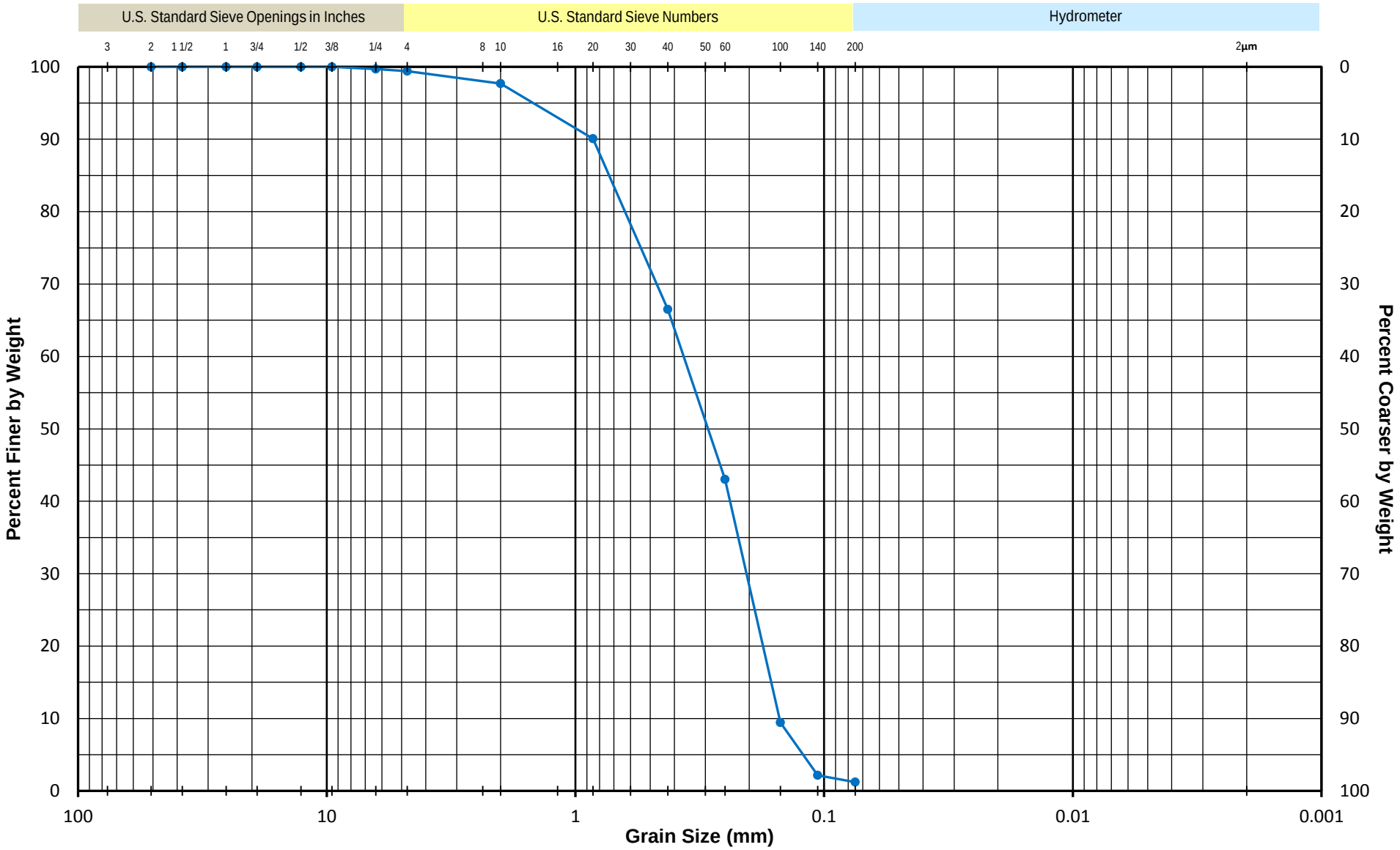


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC59 0-2	Dark Brownish Gray Fine to Coarse Sand with shell, shell fragments, and organics

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

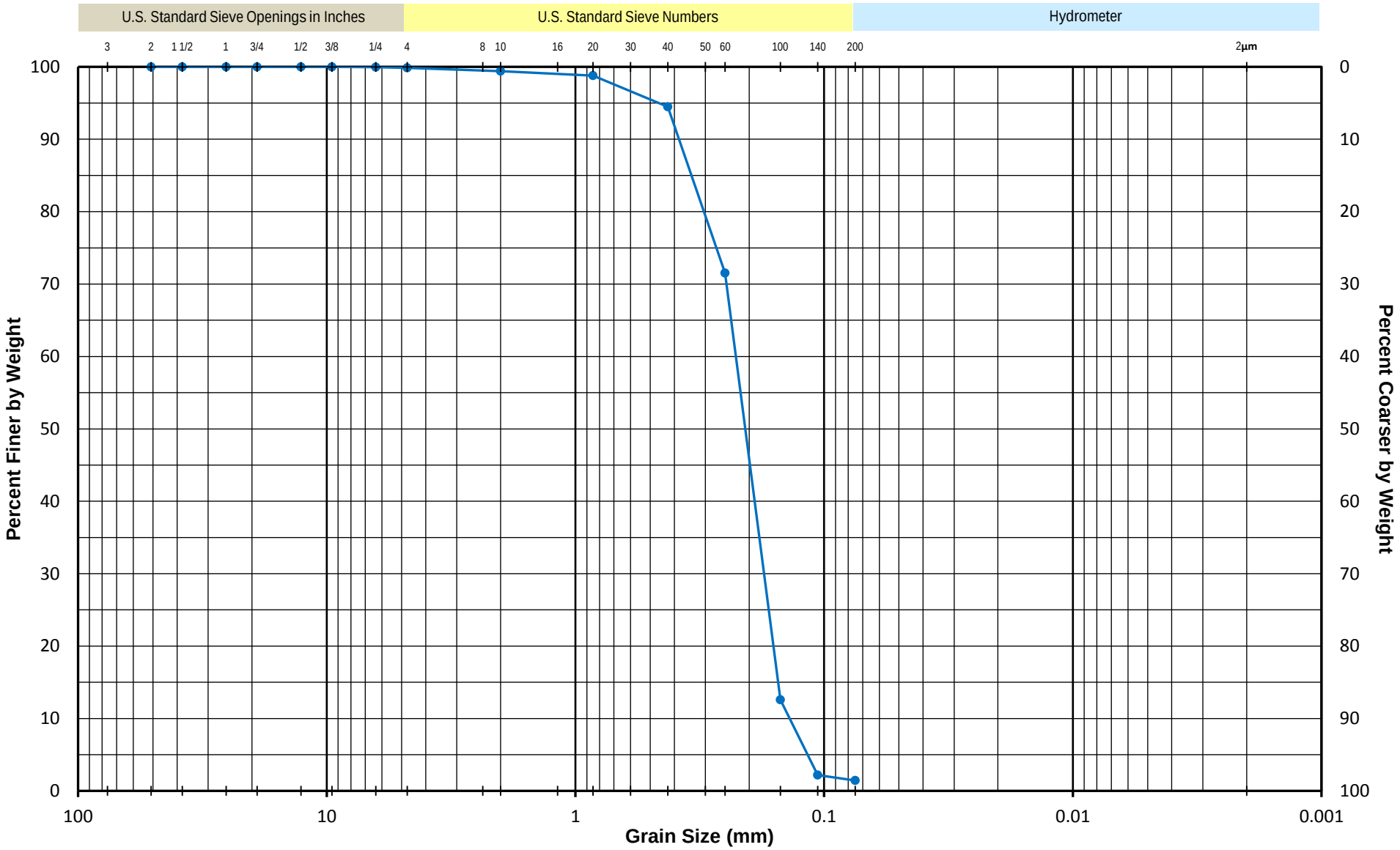


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC59 0-2	Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

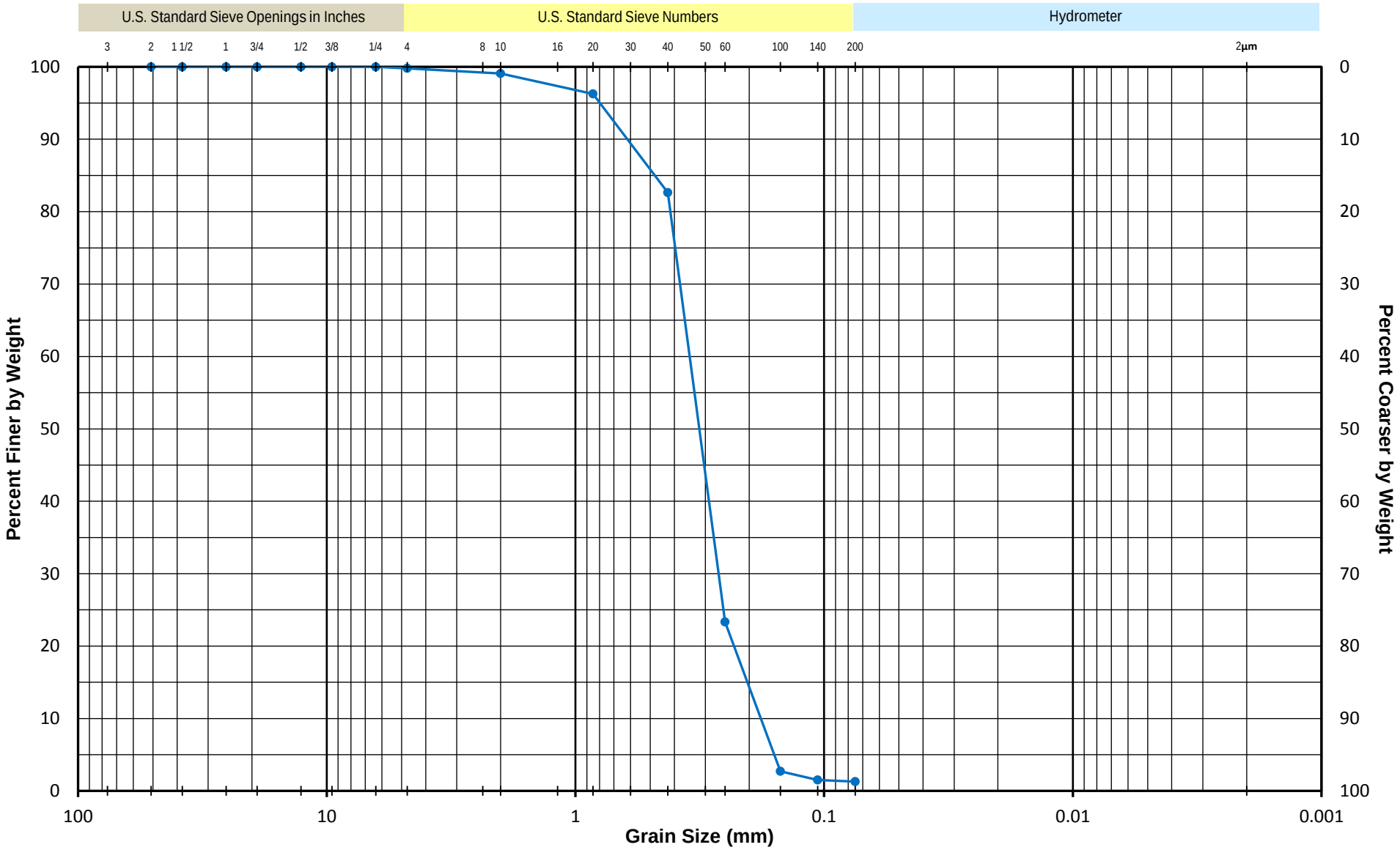


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC59 2-4	Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

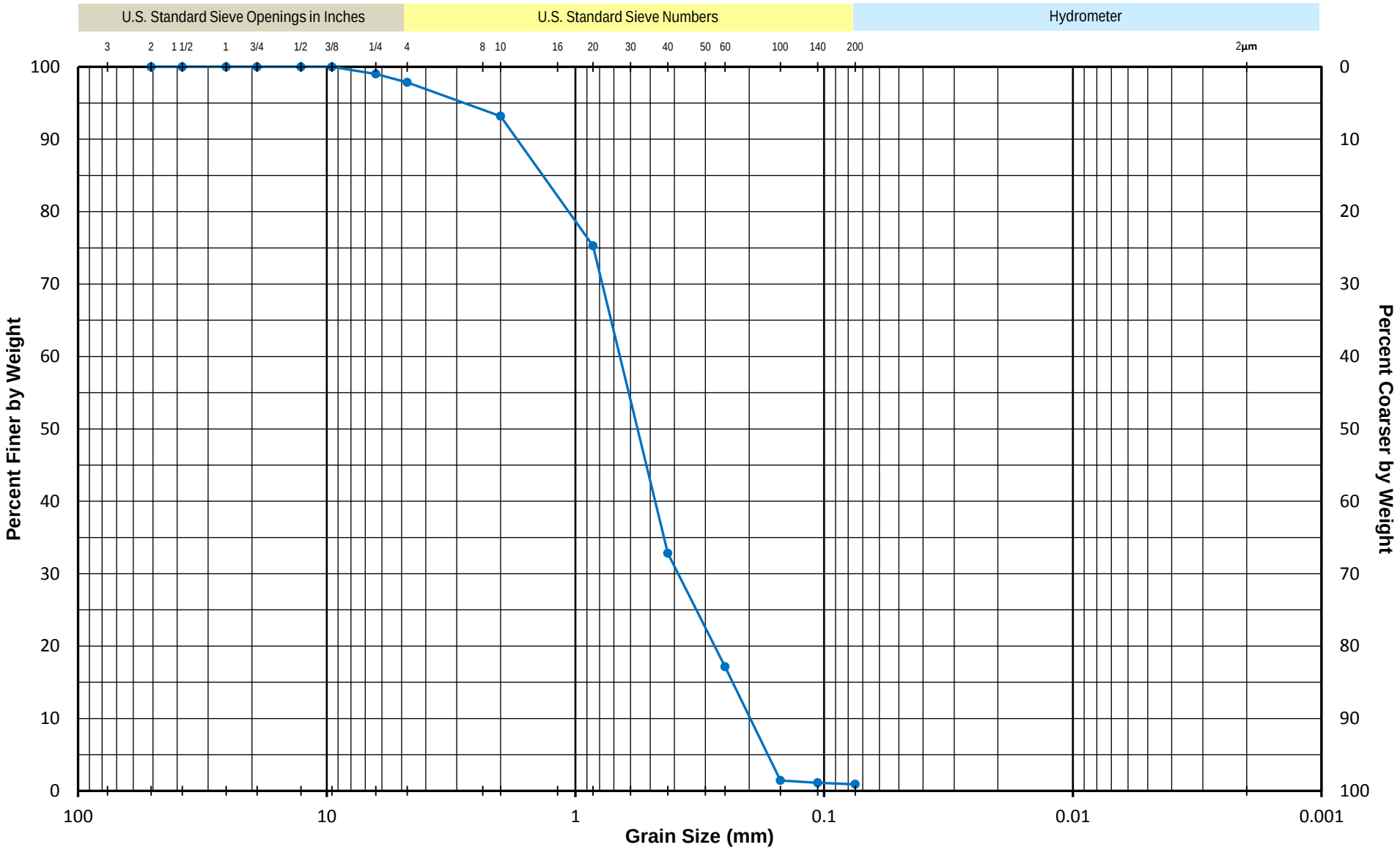


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC59 4-6	Dark Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

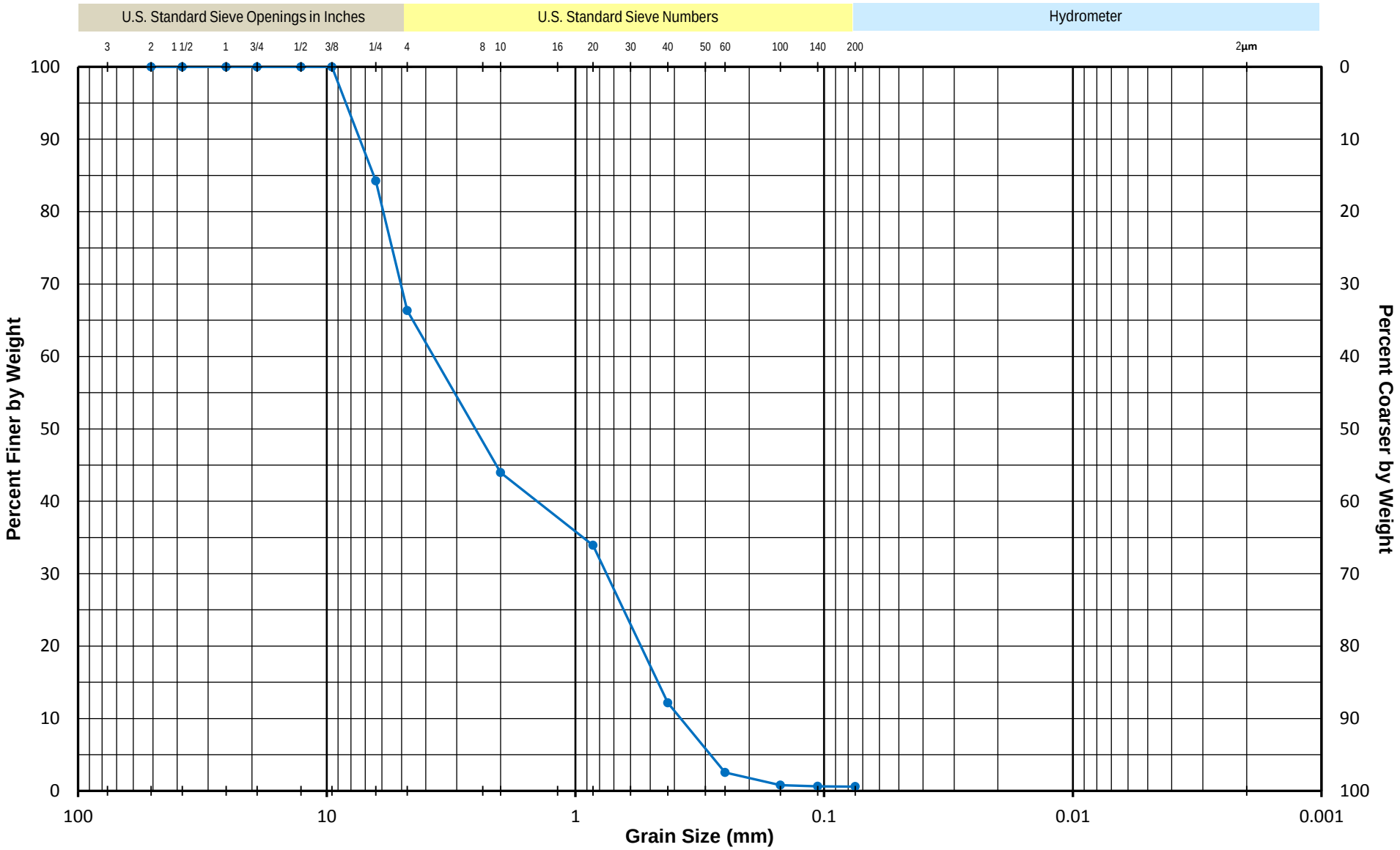


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC59 6-8	Very Dark Gray Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

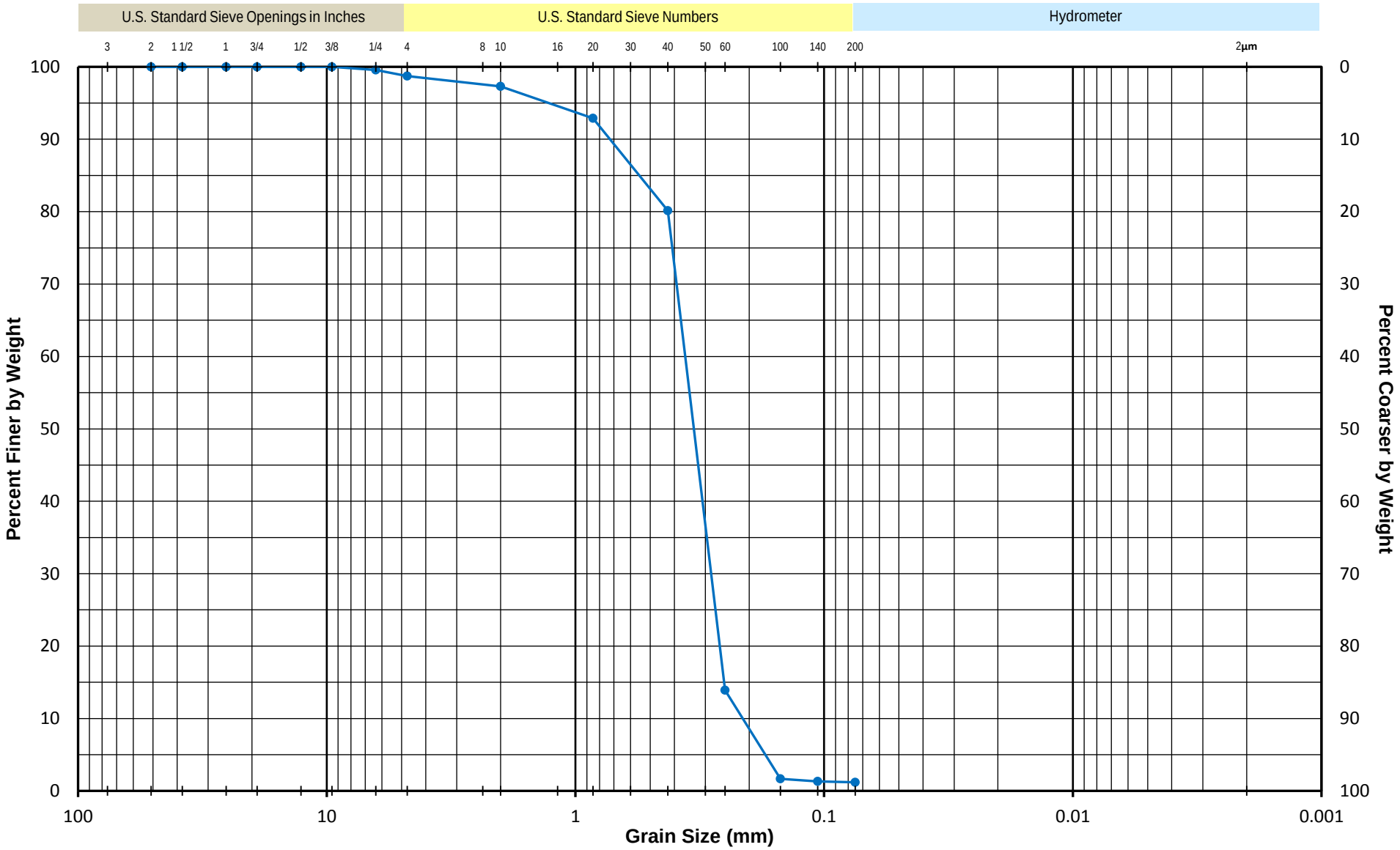


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC59 8-10	Very Dark Gray Medium to Coarse Sand with gravel

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

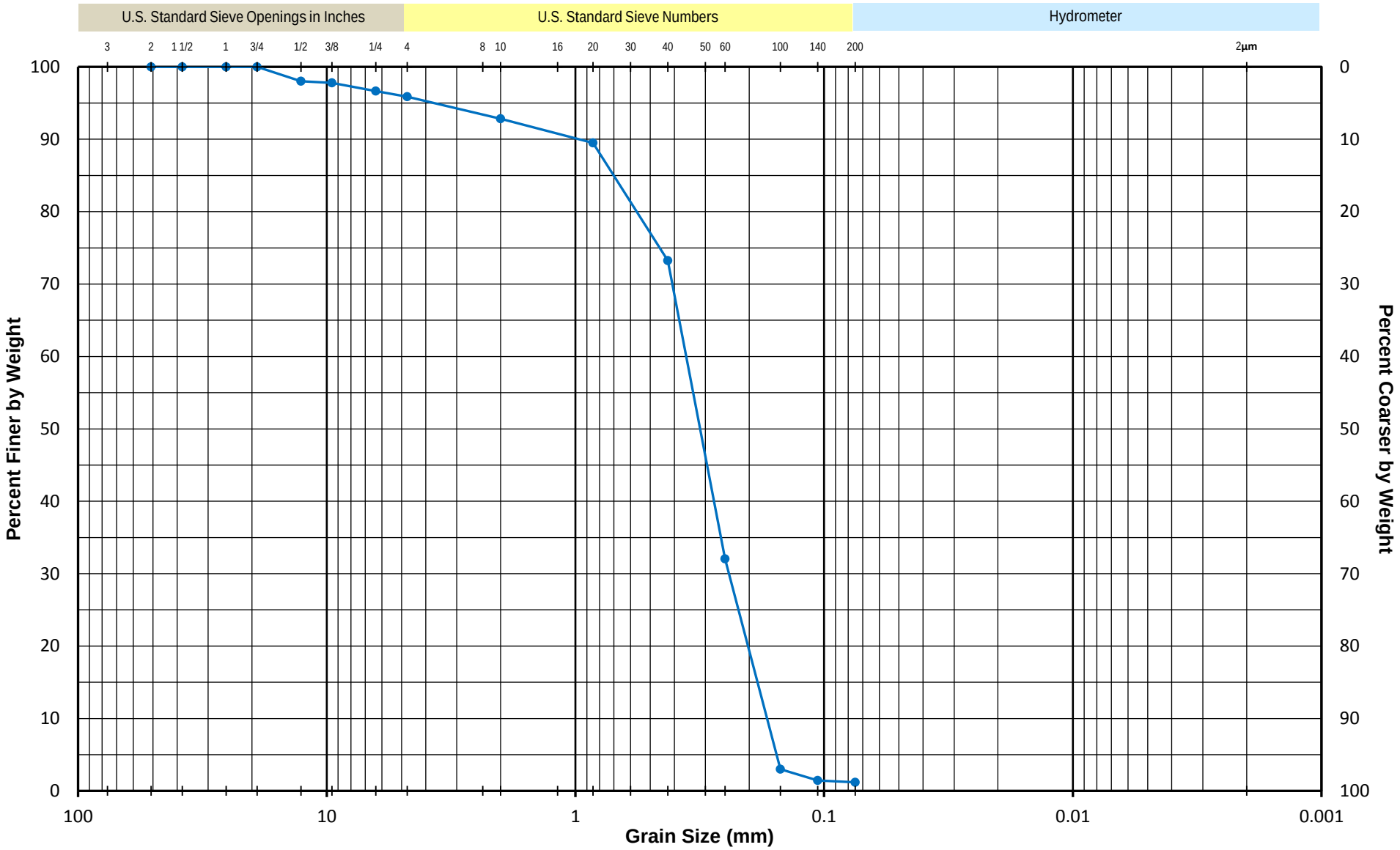


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC59 10-12	Dark Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

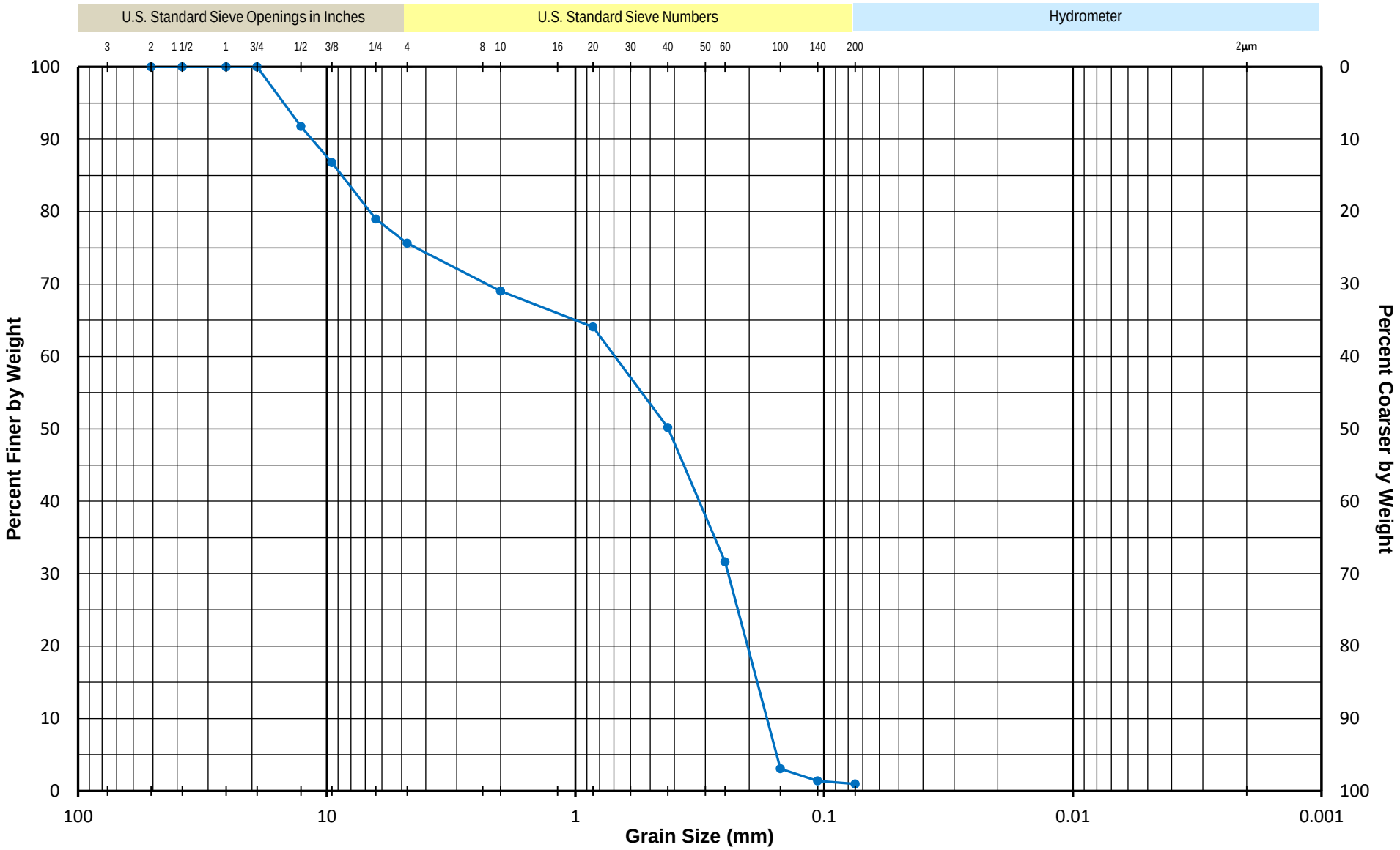


Test Depth	Core ID Section/Sample	Material Description
0.3 ft	VC60 0-2	Black and Brown Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

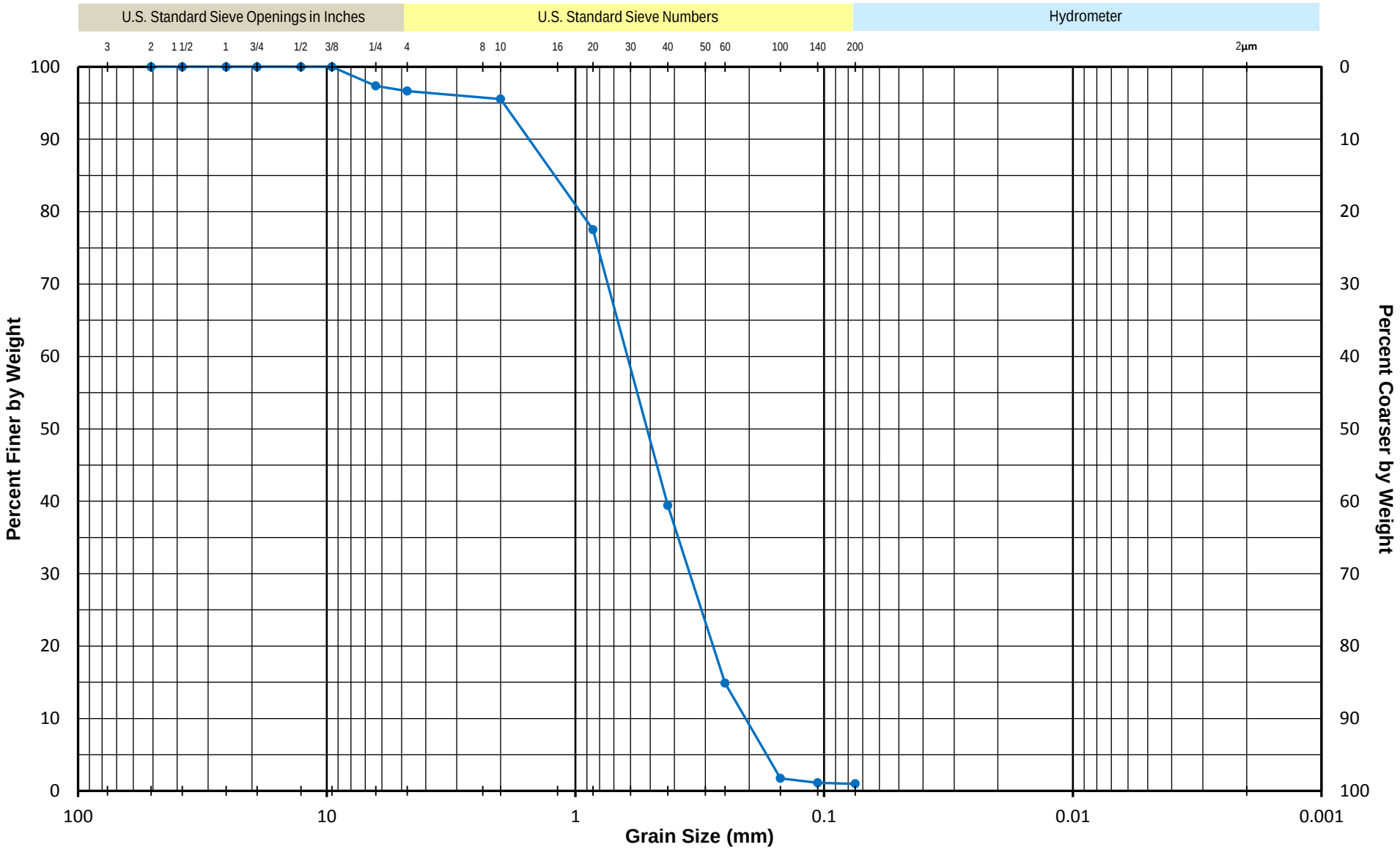


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC60 0-2	Black Fine to Medium Sand with gravel and glass debris

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

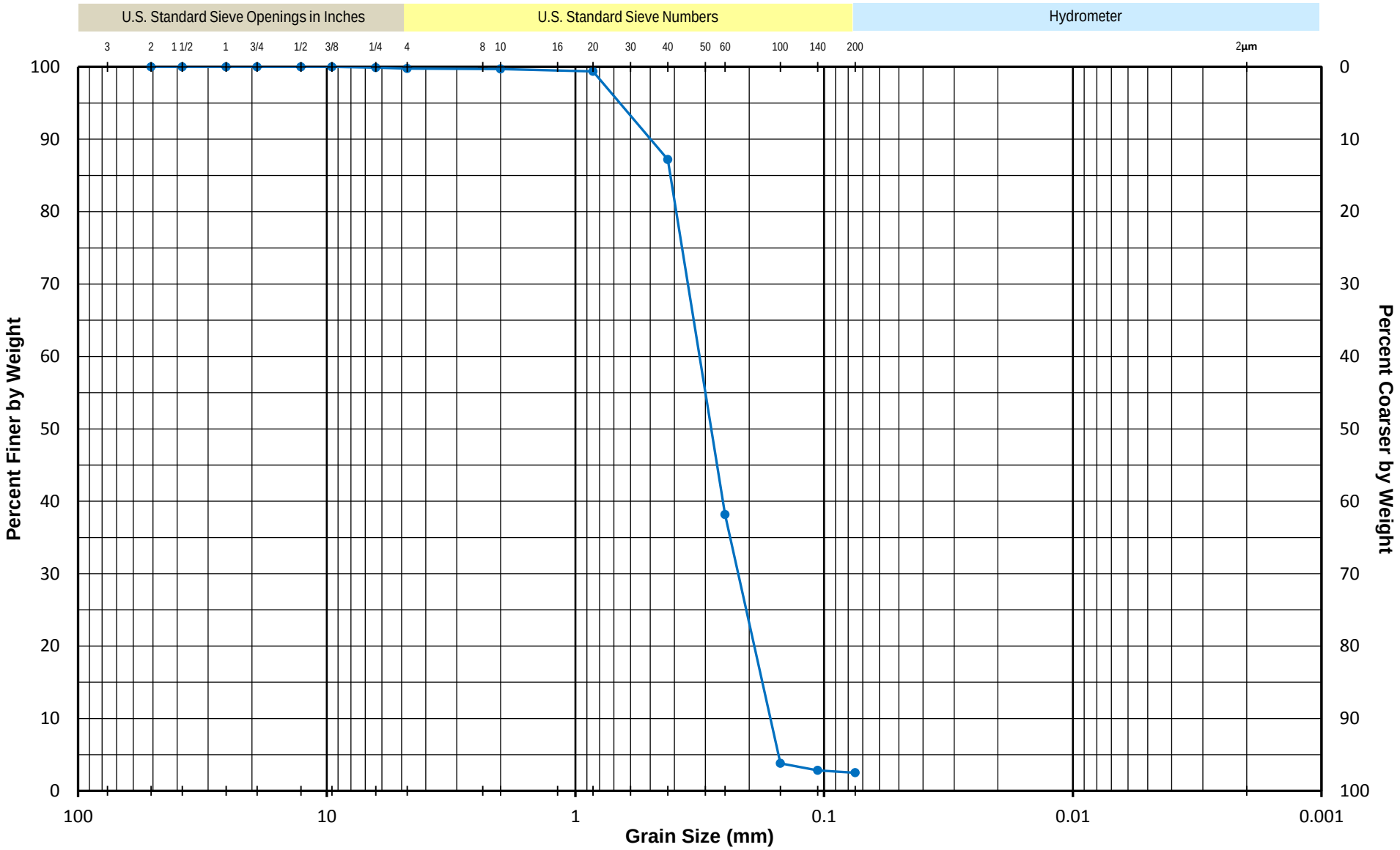


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC60 2-4	Grayish Brown Medium to Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

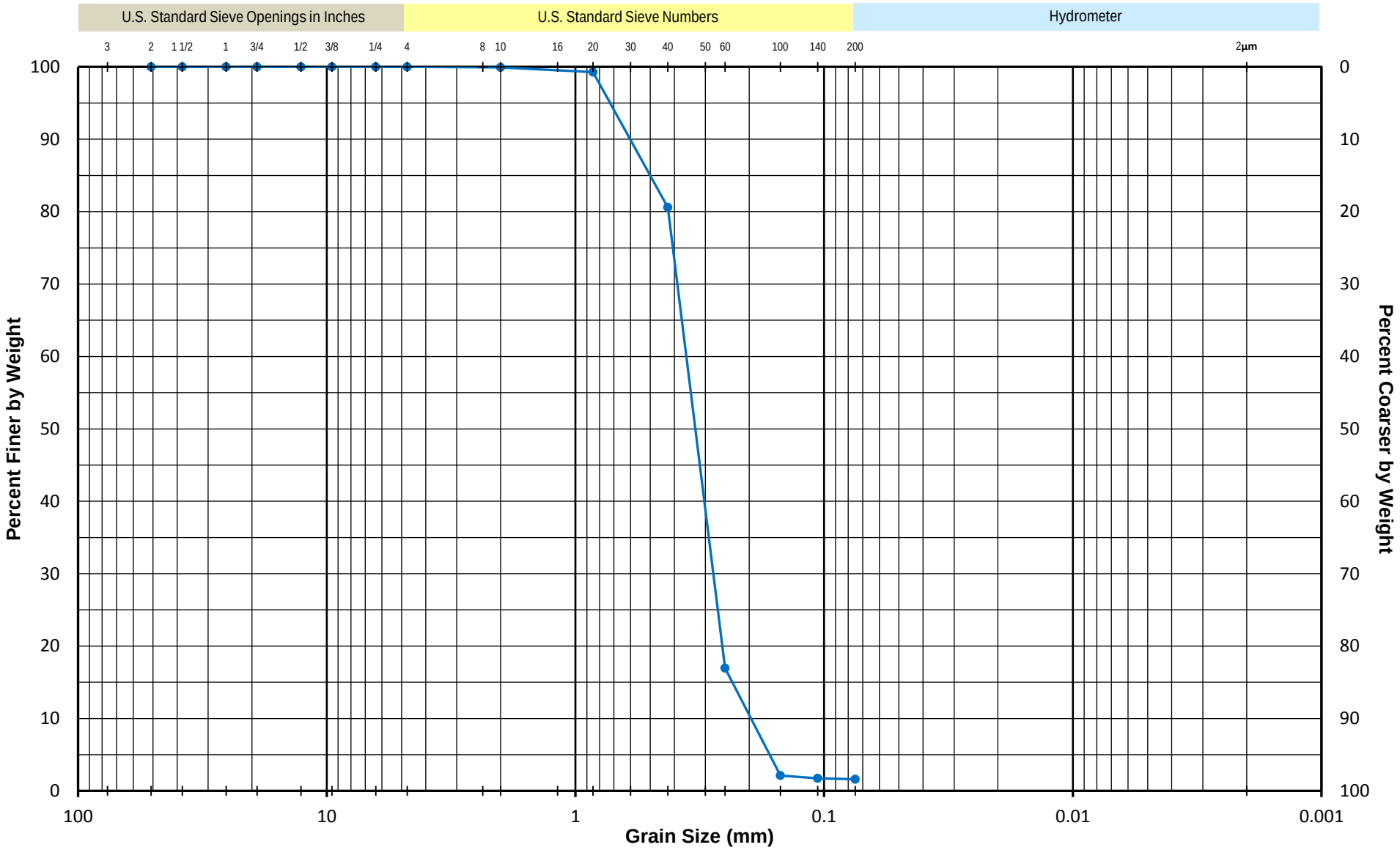


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC60 4-6	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

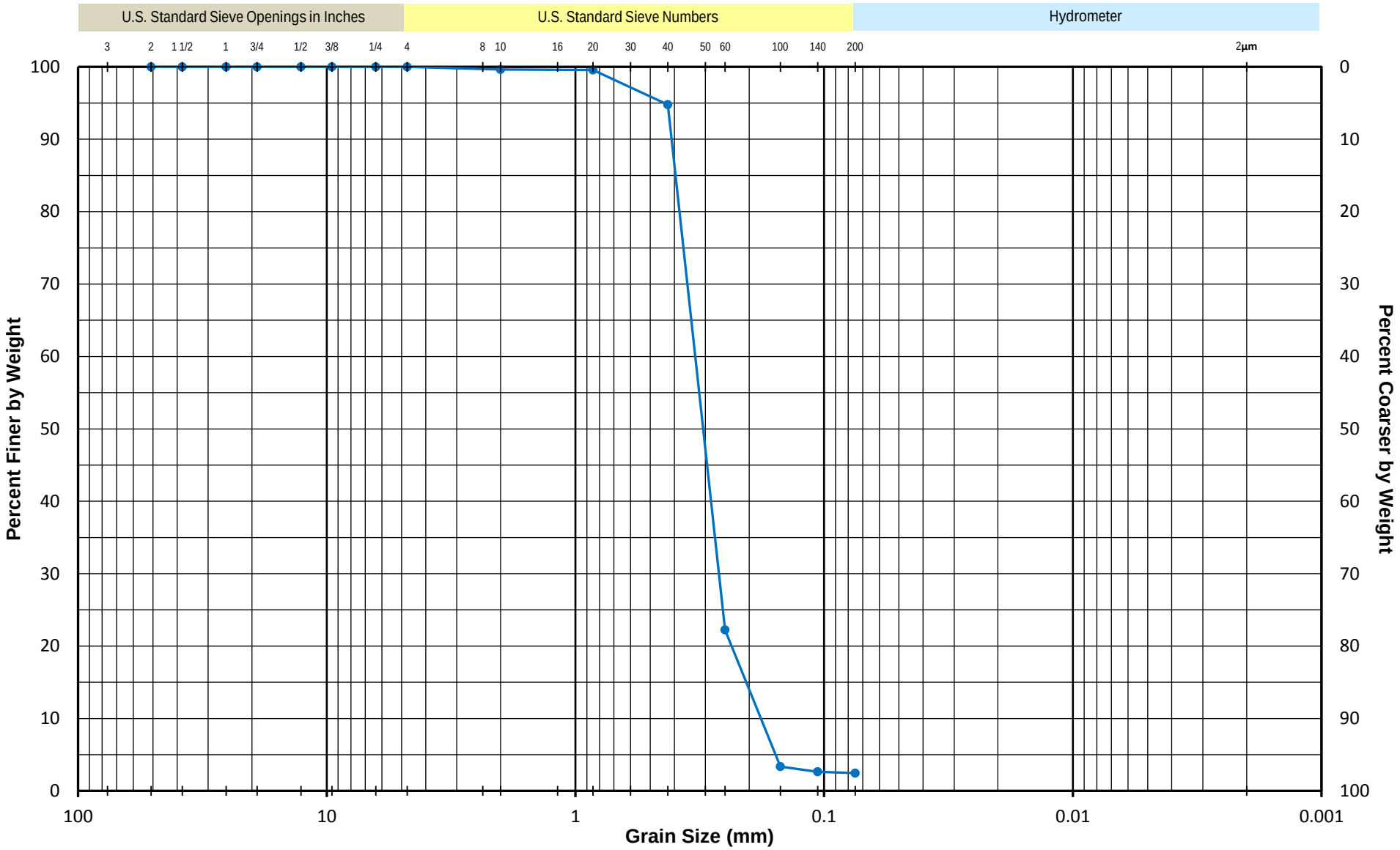


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC60 6-8	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

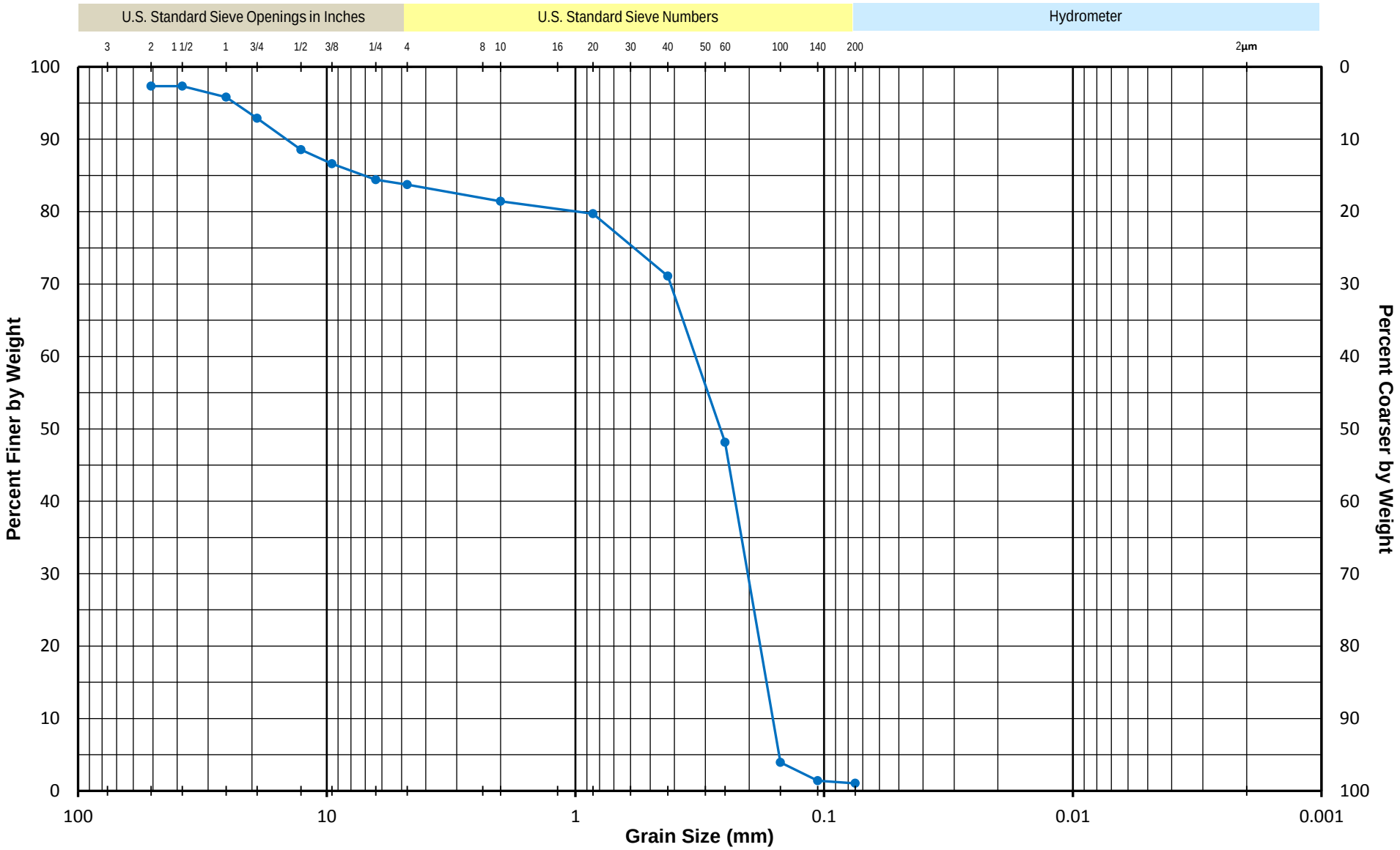


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC60 8-10	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

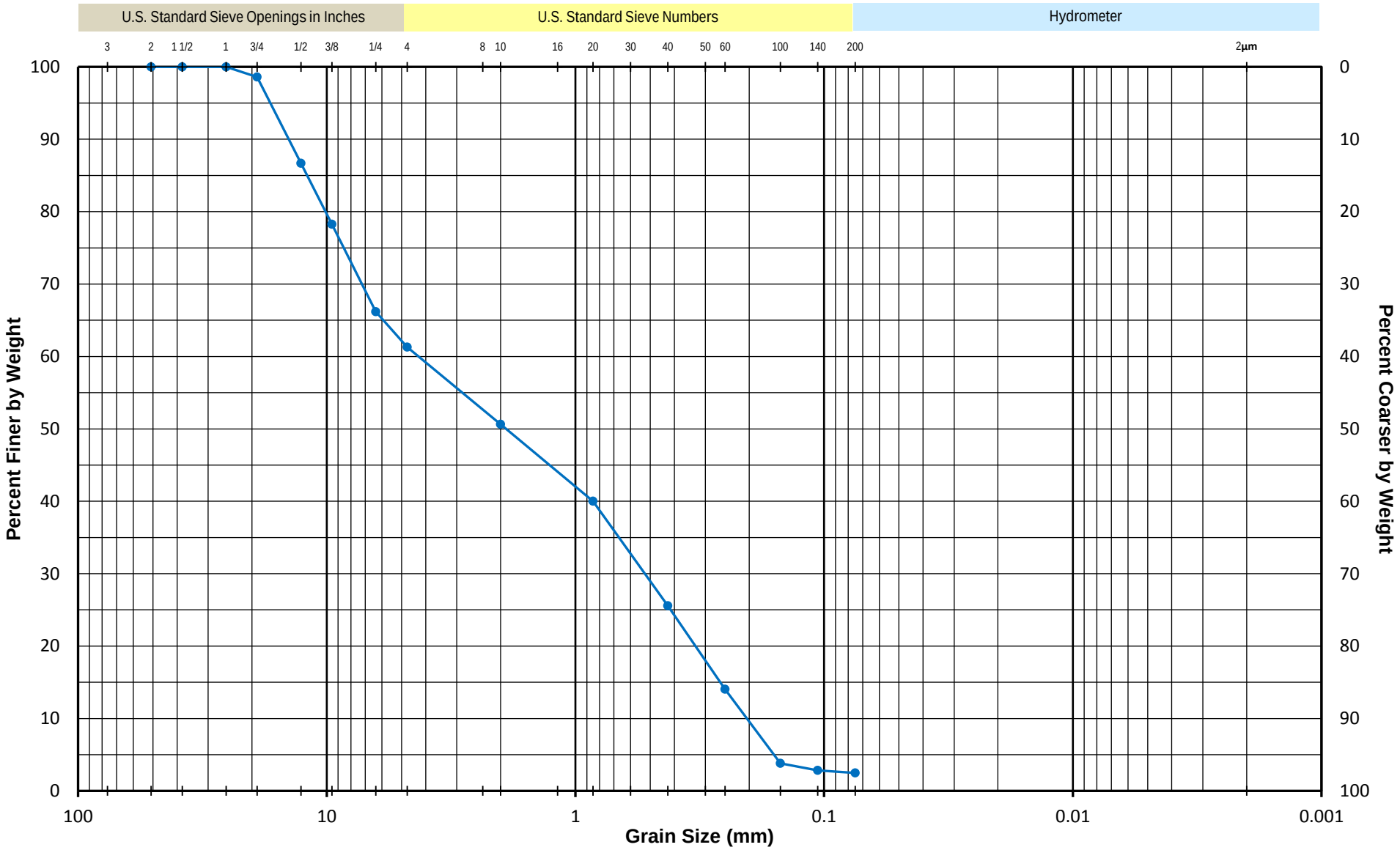


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC61 0-2	Very Dark Gray Fine to Medium Sand with gravel

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

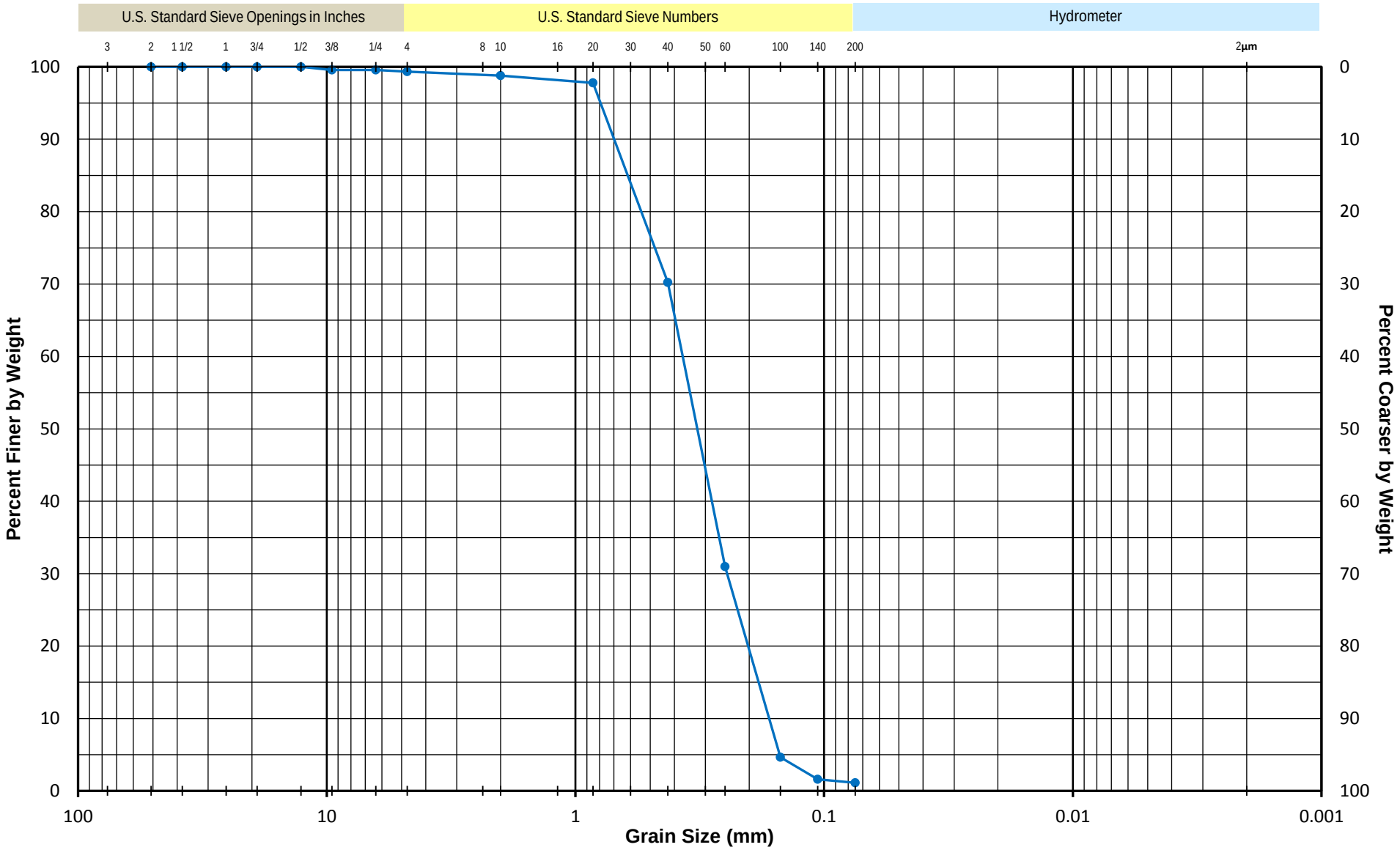


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC61 0-2	Black Fine to Coarse Sand with gravel and glass and metal debris

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

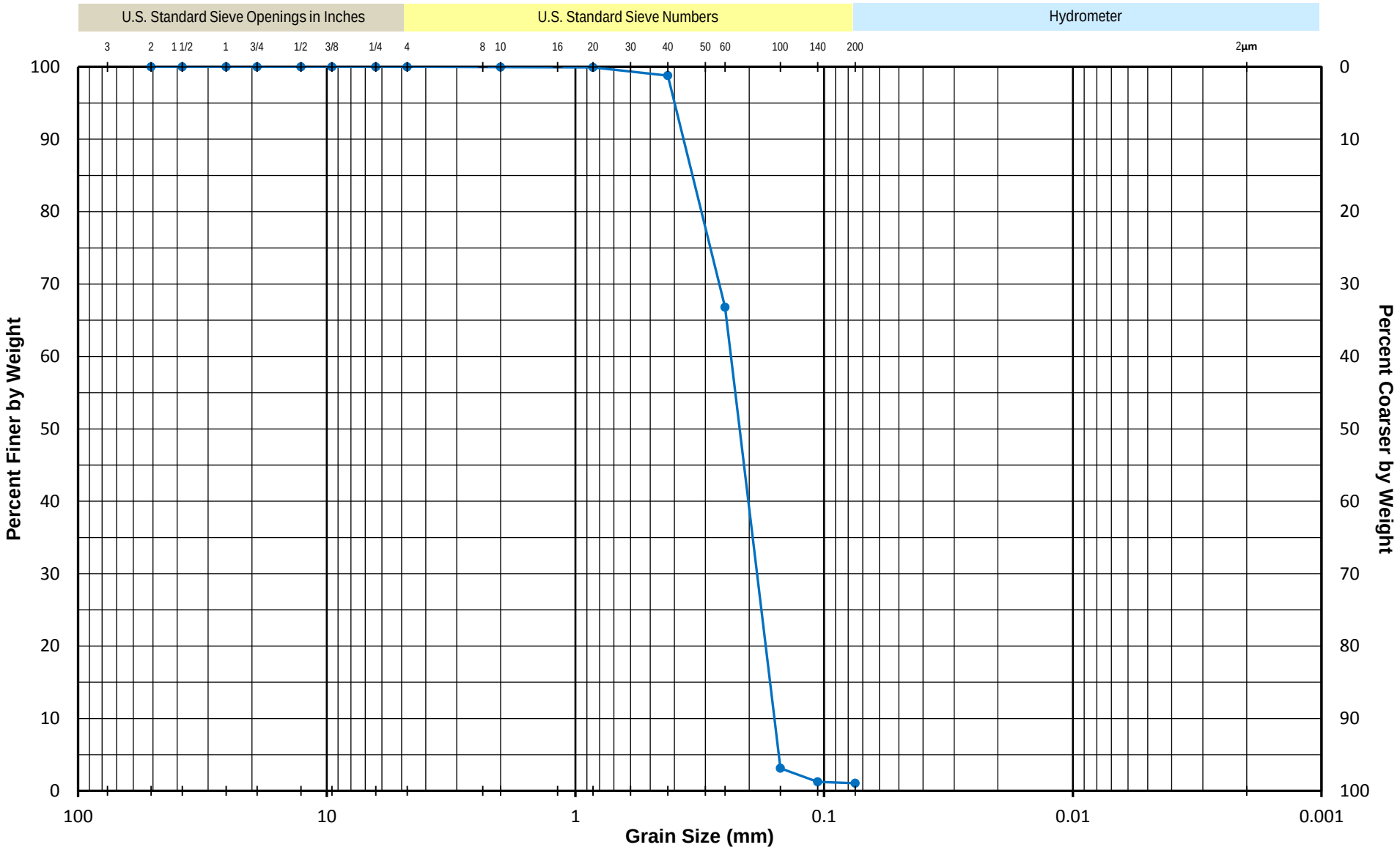


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC61 2-4	Very Dark Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

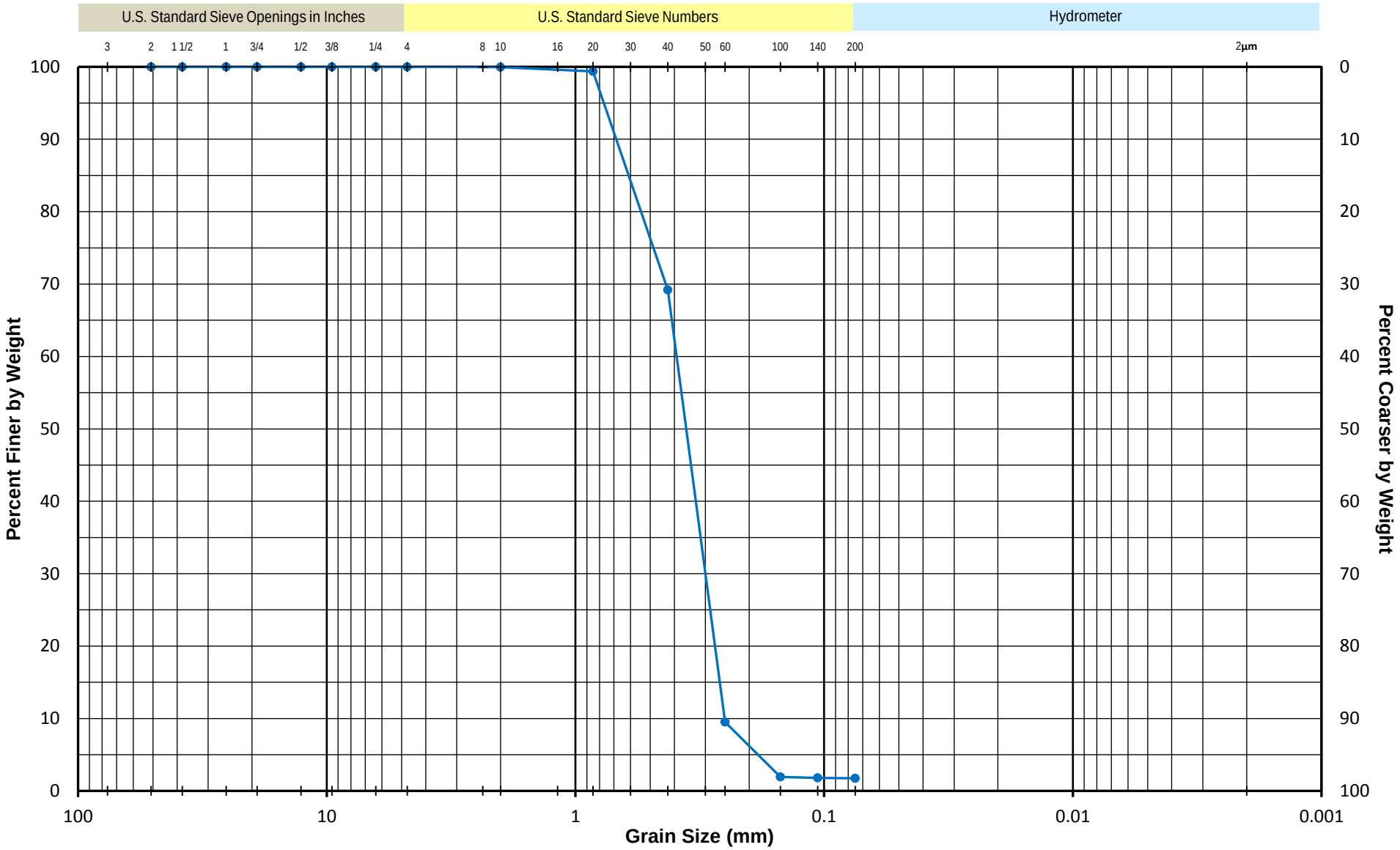


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC61 4-6	Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

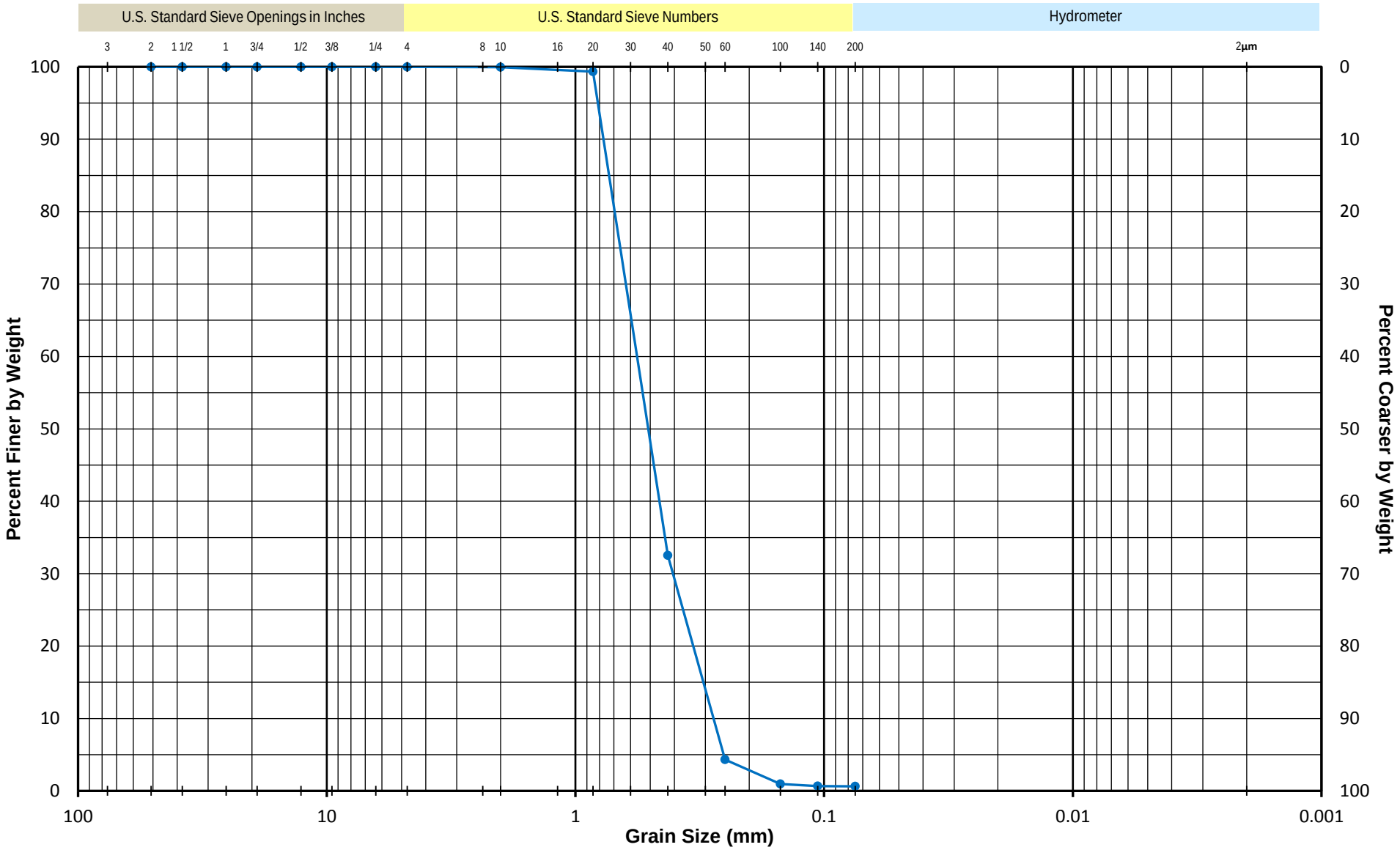


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC61 6-8	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

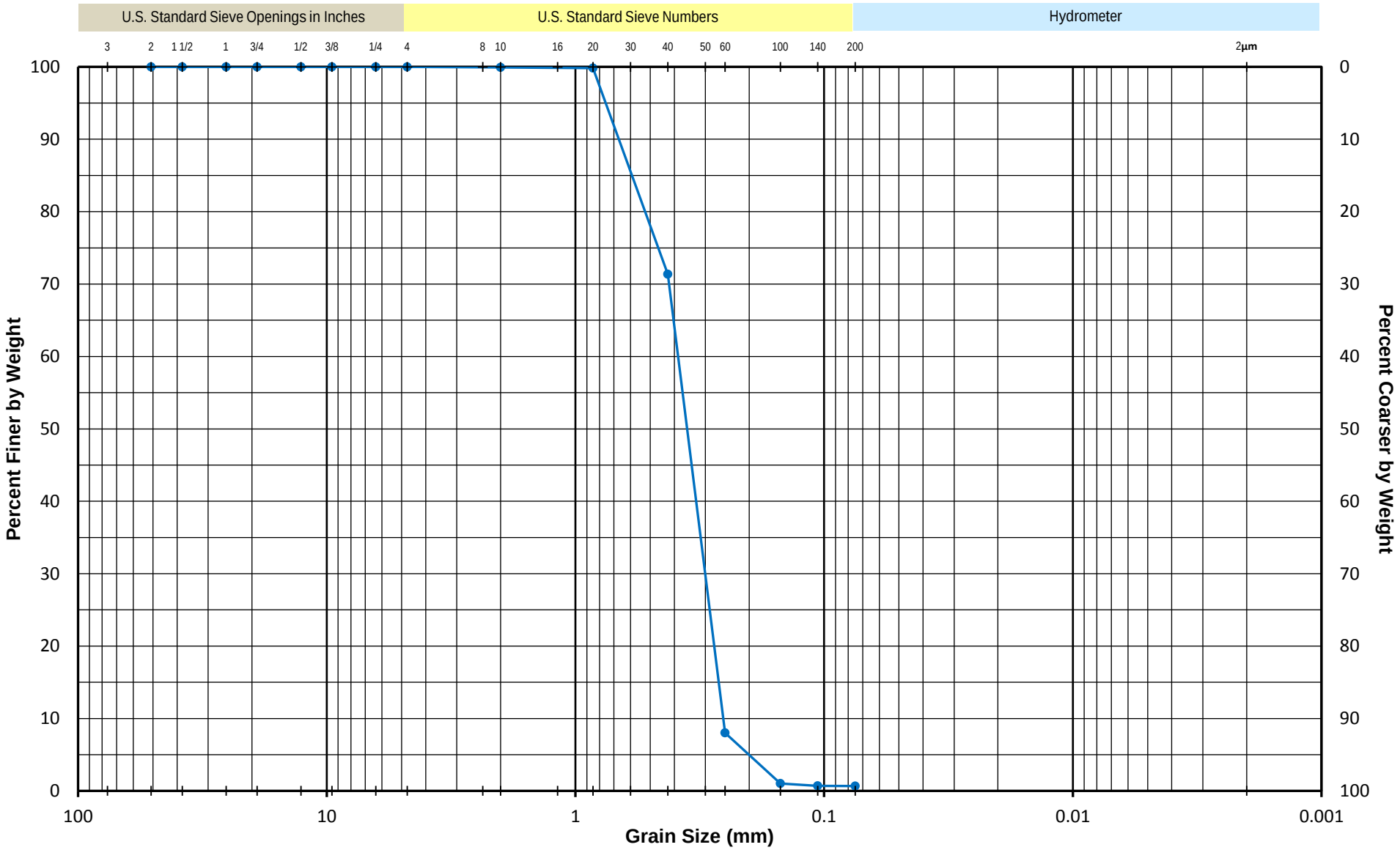


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC61 8-10	Gray Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

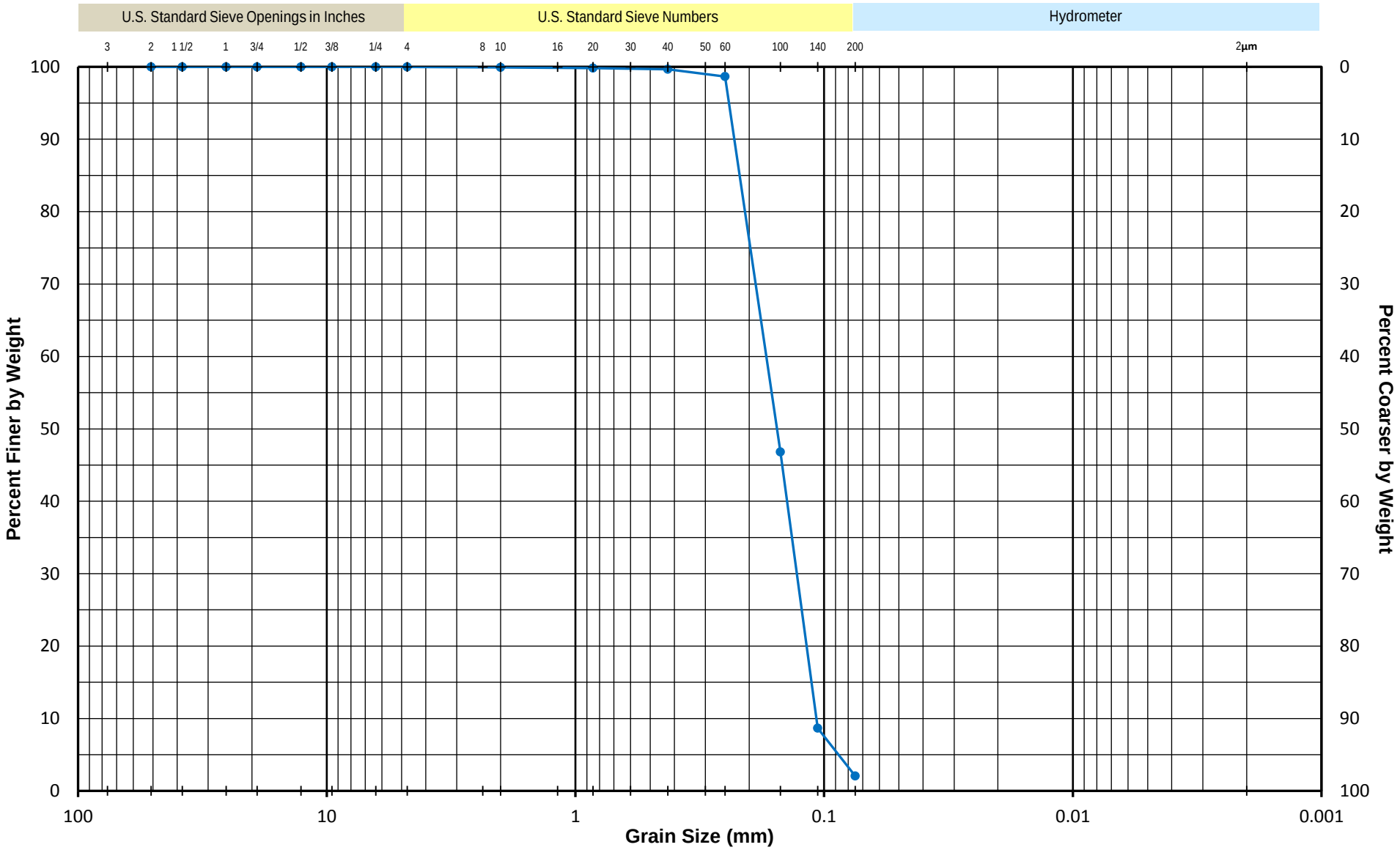


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC61 10-12	Dark Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

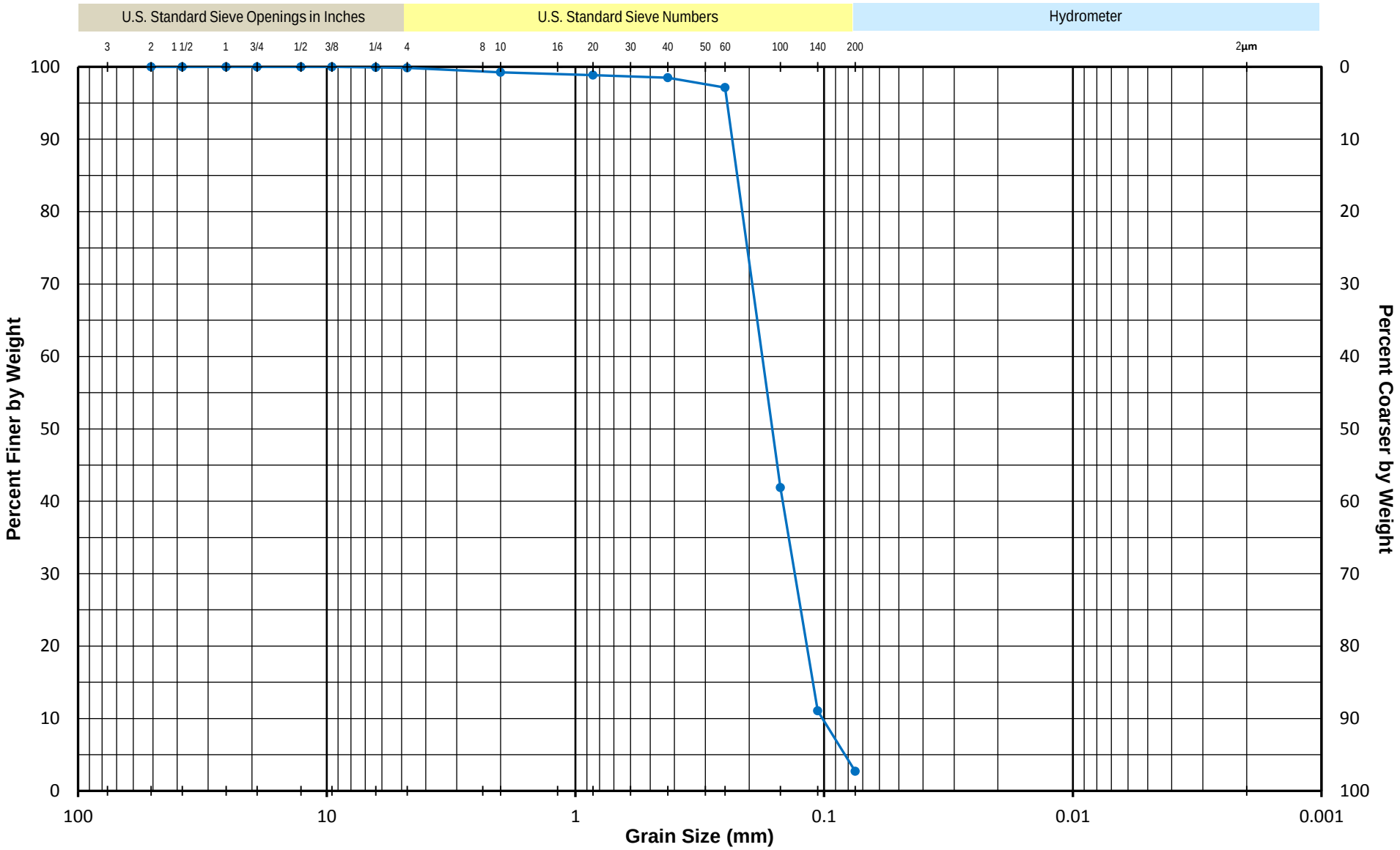


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC62 0-2	Very Dark Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

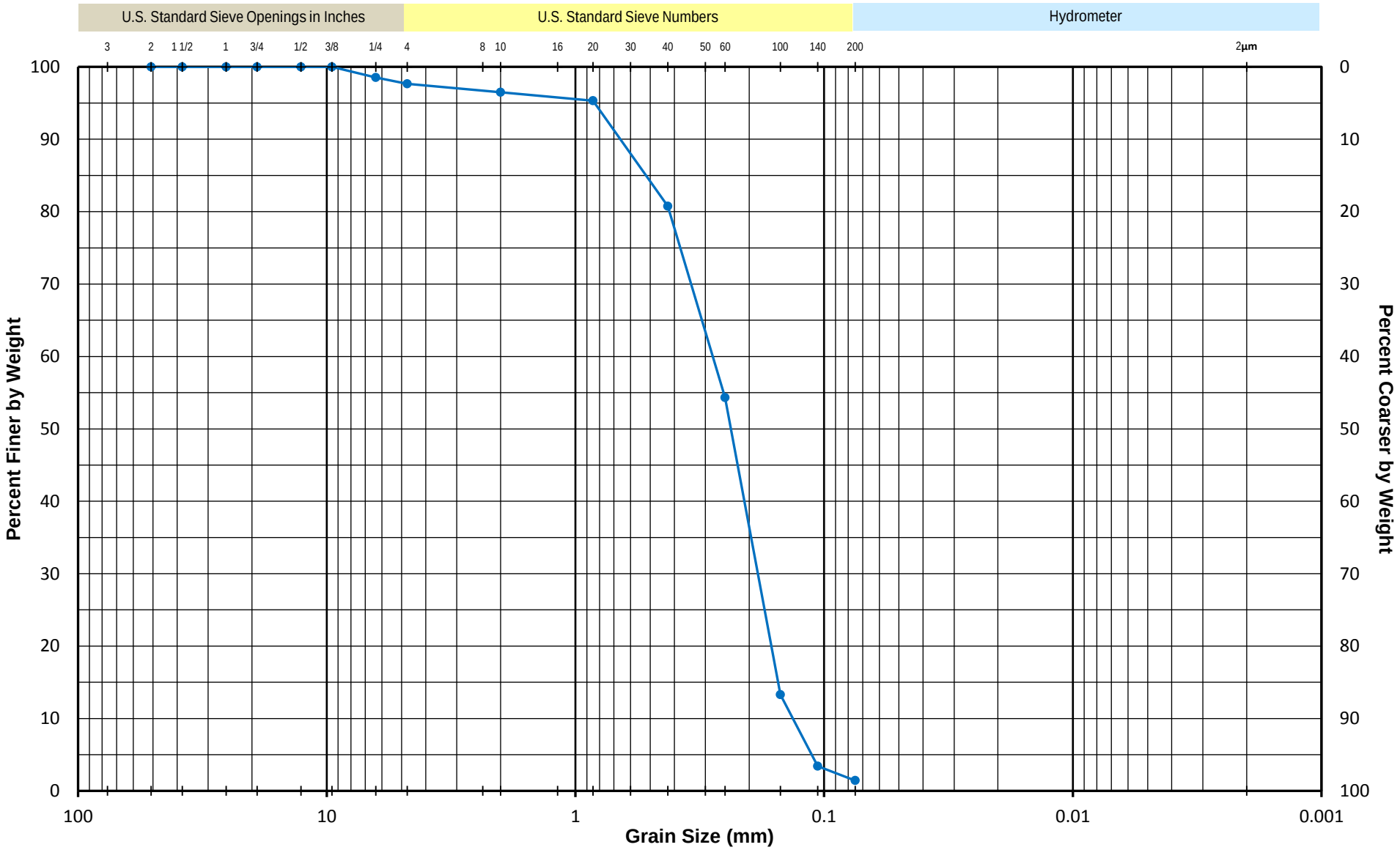


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC62 0-2	Black Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

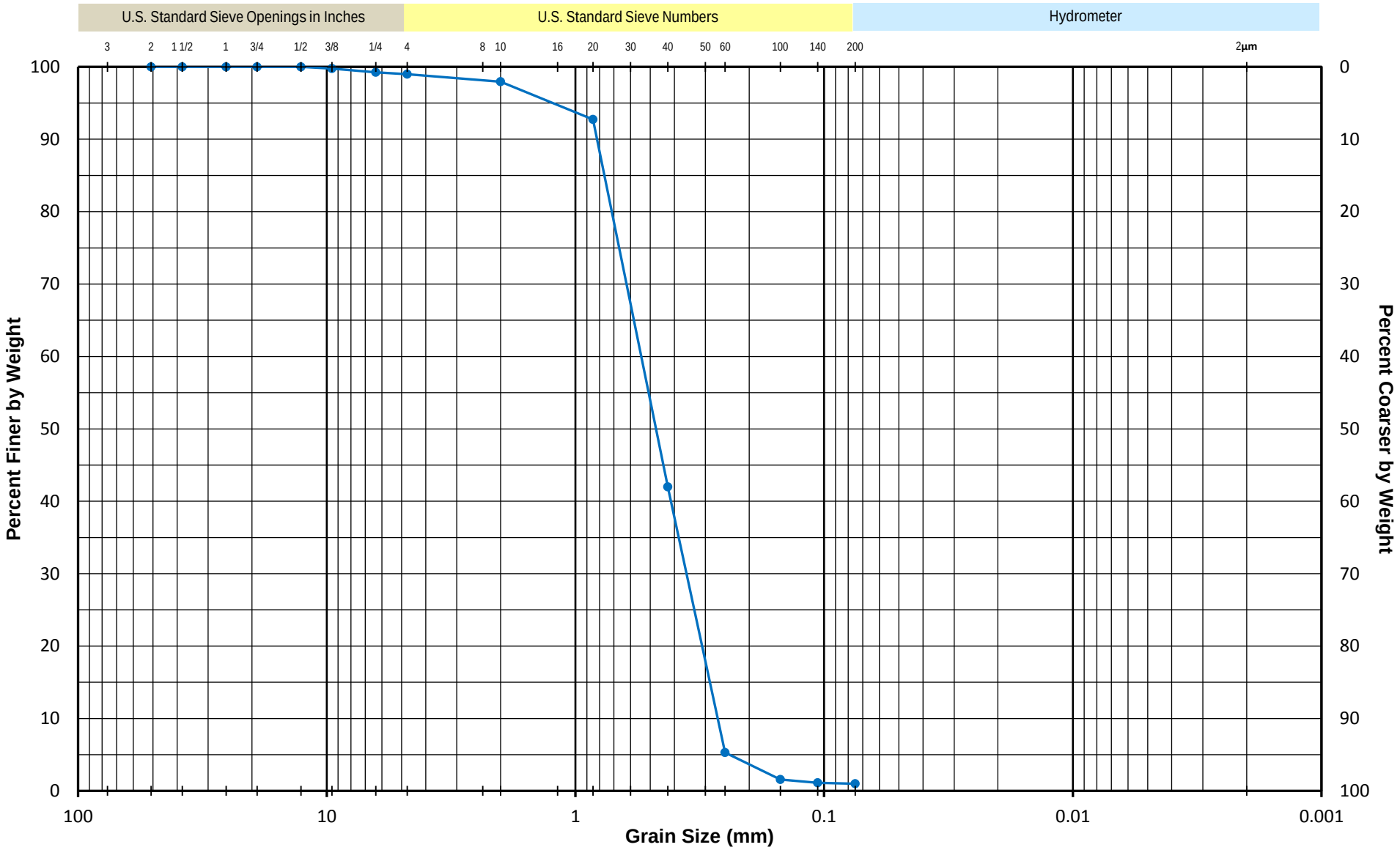


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC62 2-4	Very Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

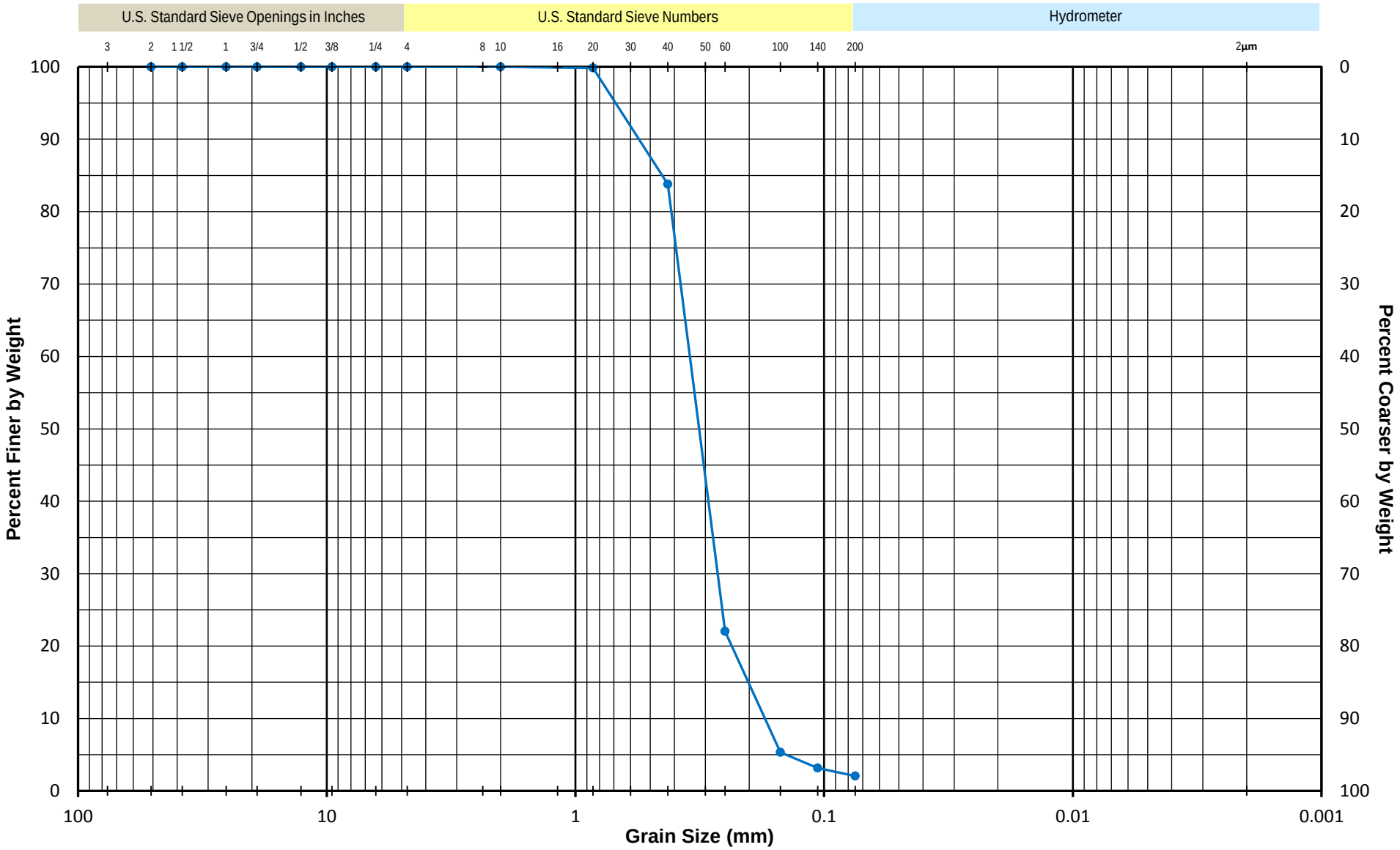


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC62 4-6	Grayish Brown Medium to Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

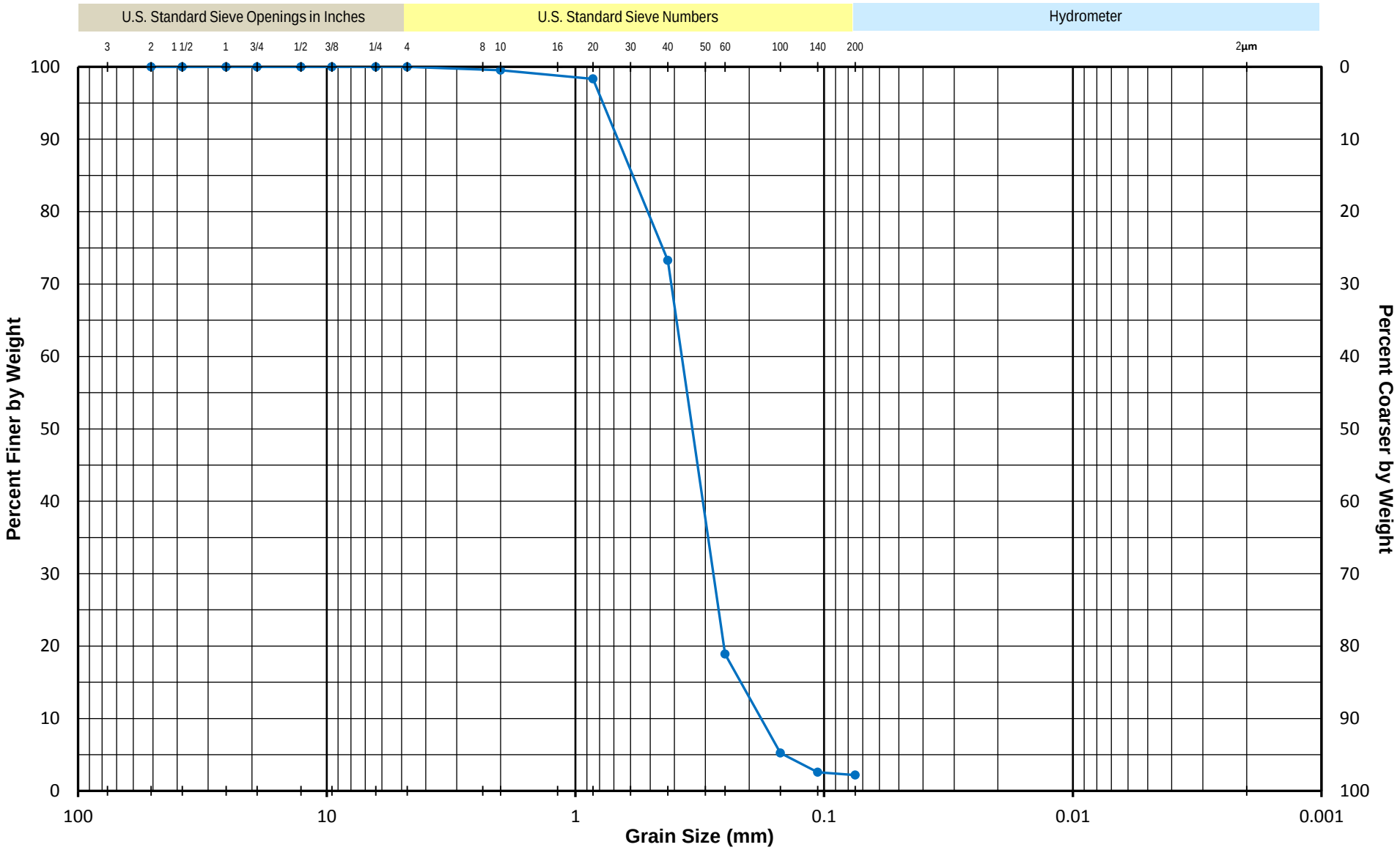


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC62 6-8	Gray Fine to Medium Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

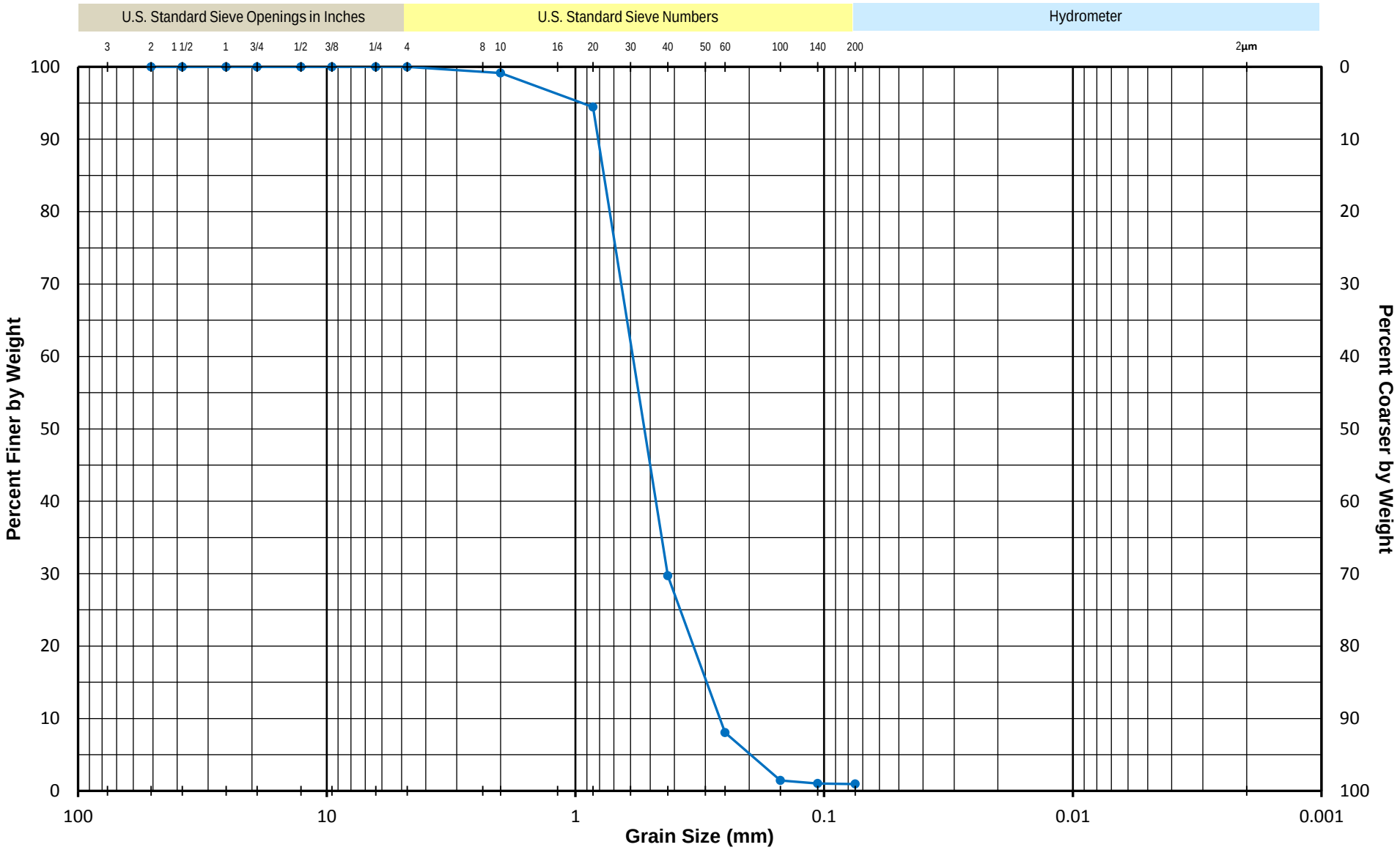


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC62 8-10	Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

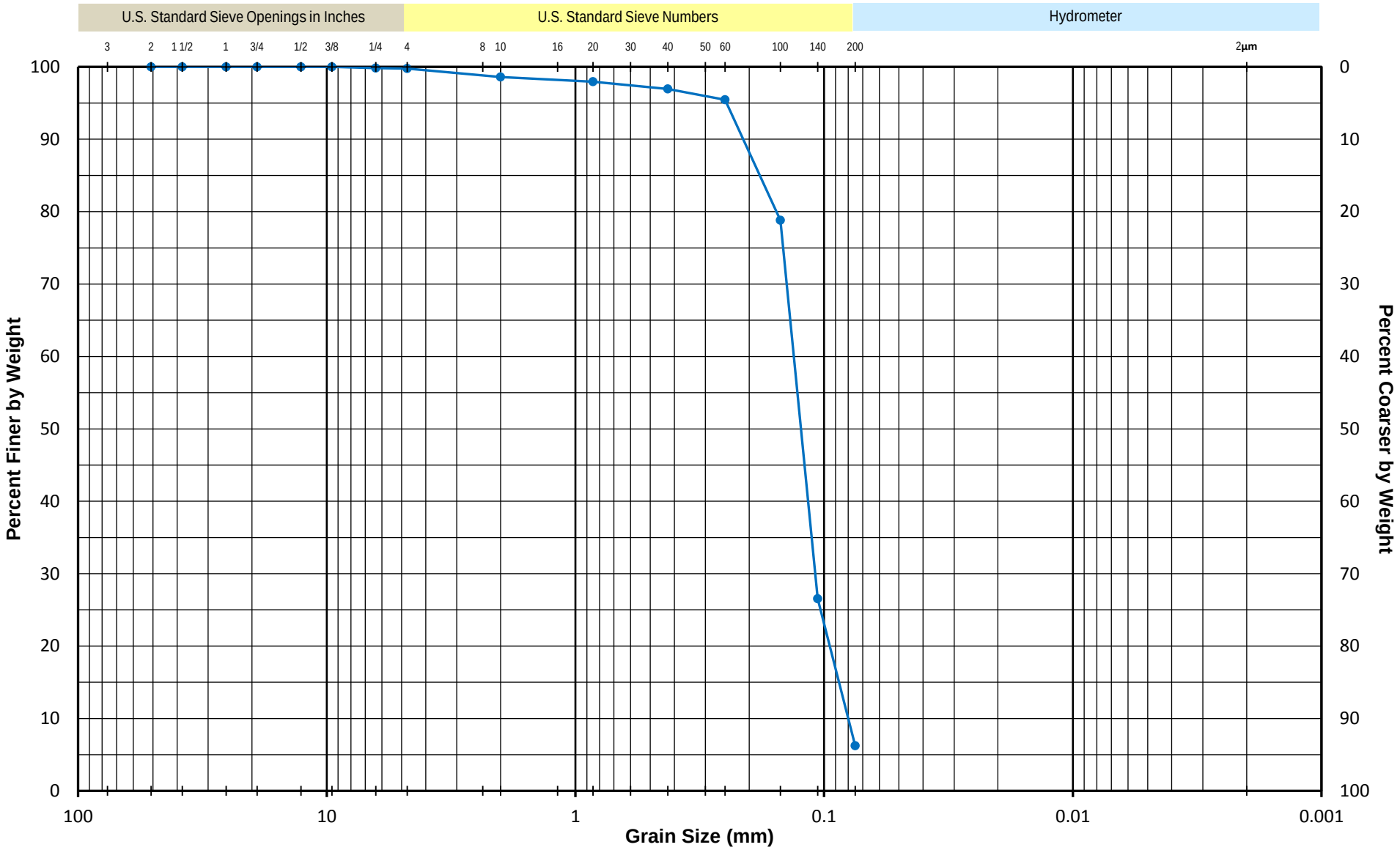


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC62 10-12	Grayish Brown Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

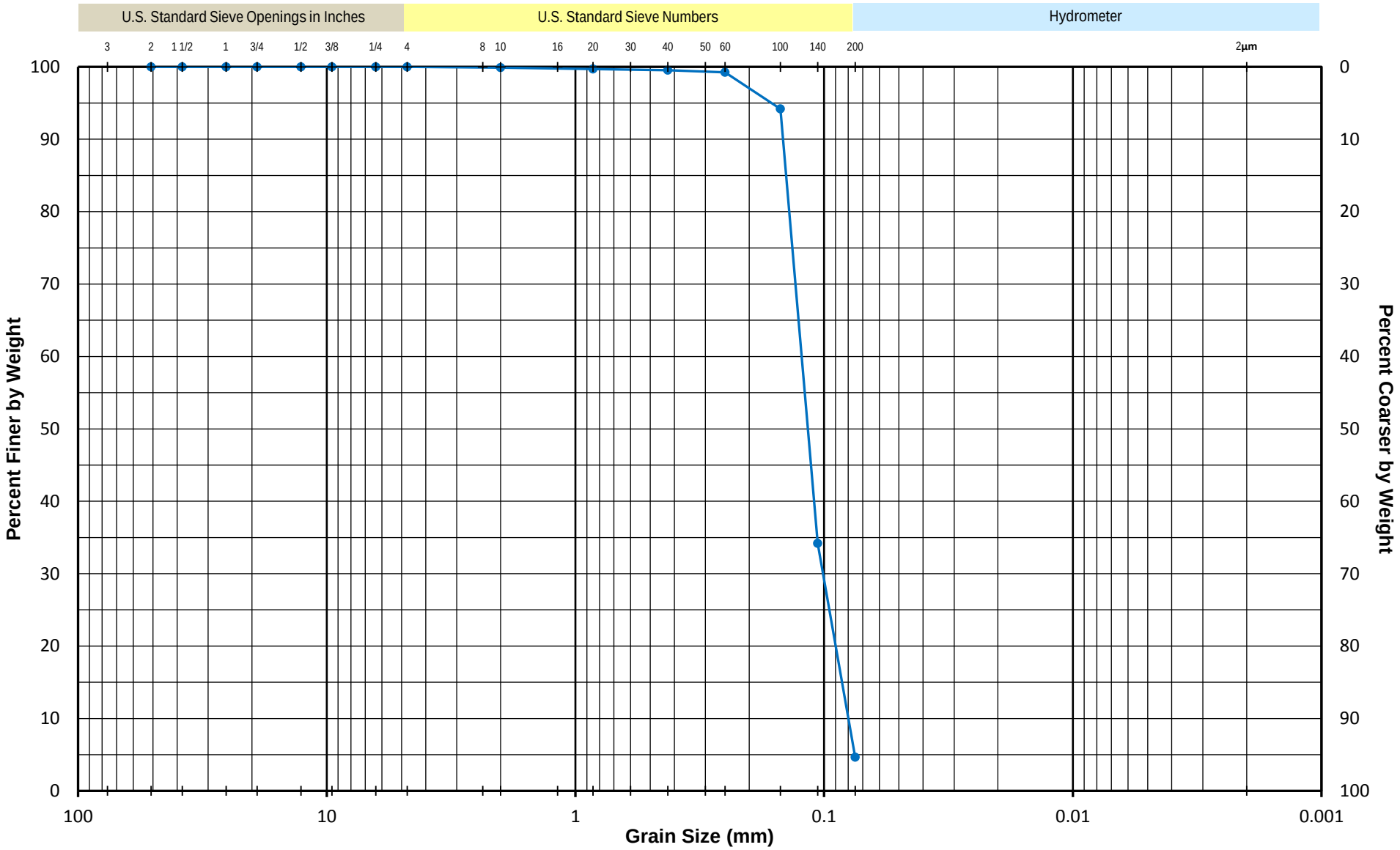


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC64 0-2	Black Very Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

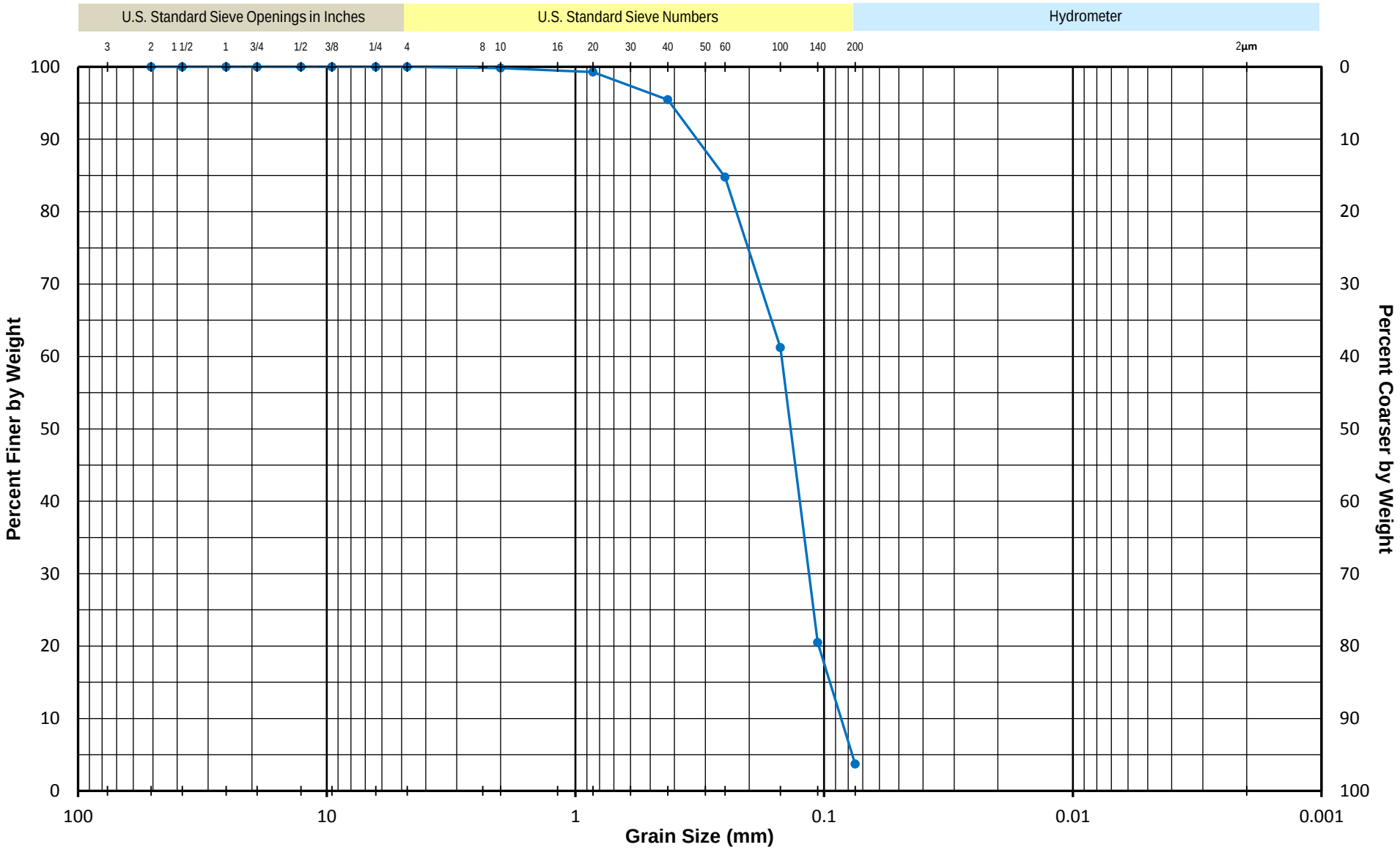


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC64 0-2	Black Very Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

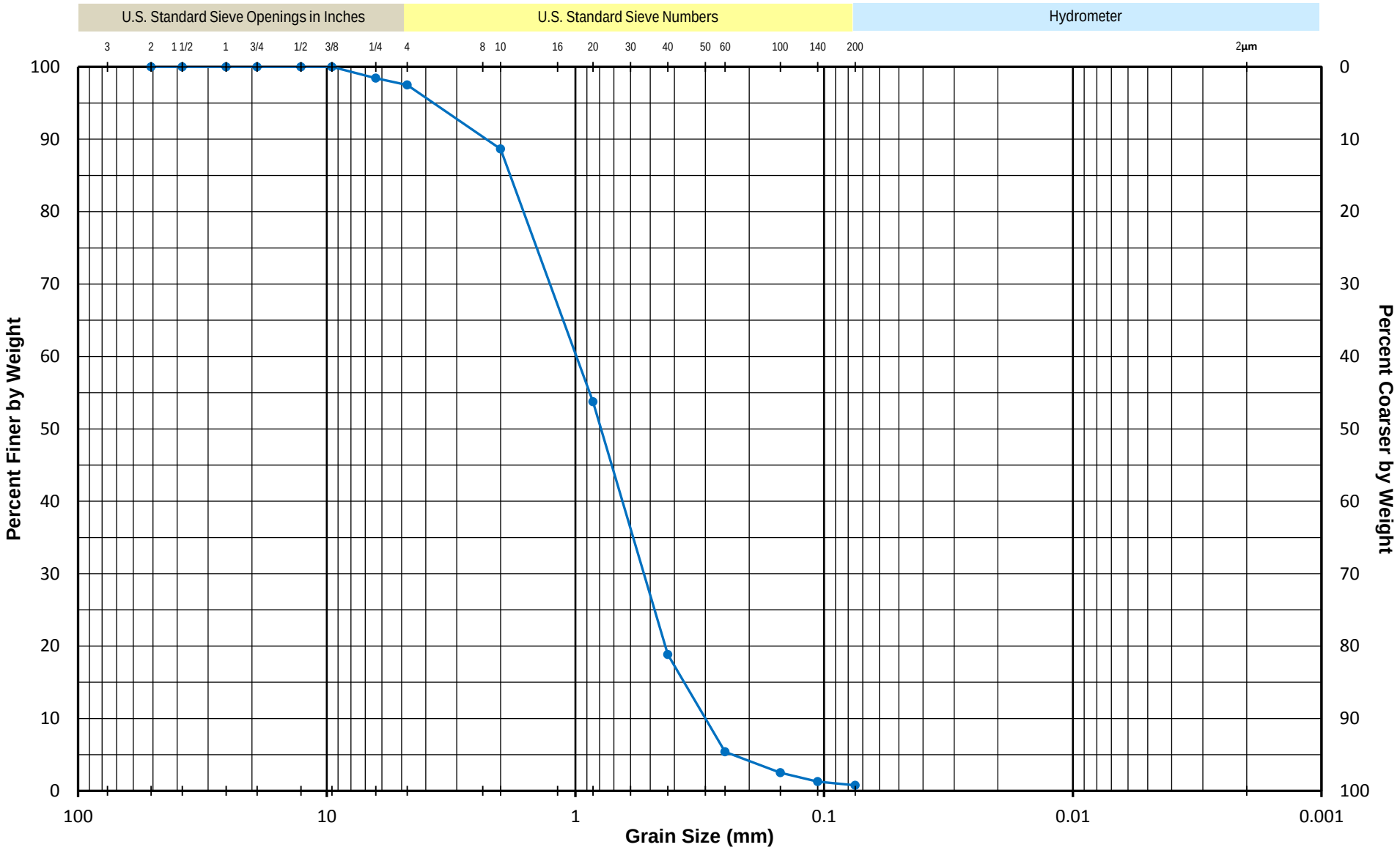


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC64 2-4	Very Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

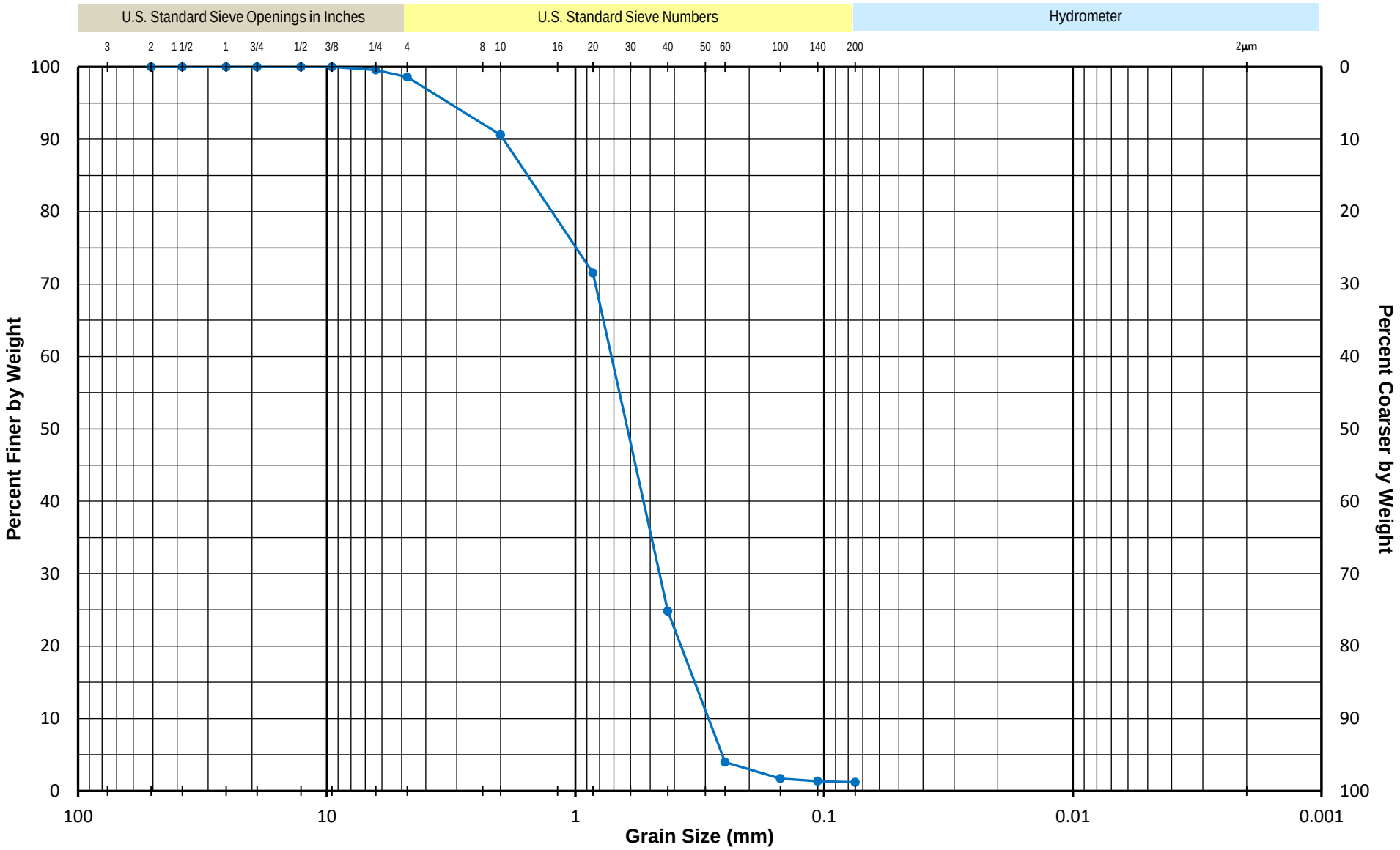


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC64 4-6	Brown Fine to Coarse Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

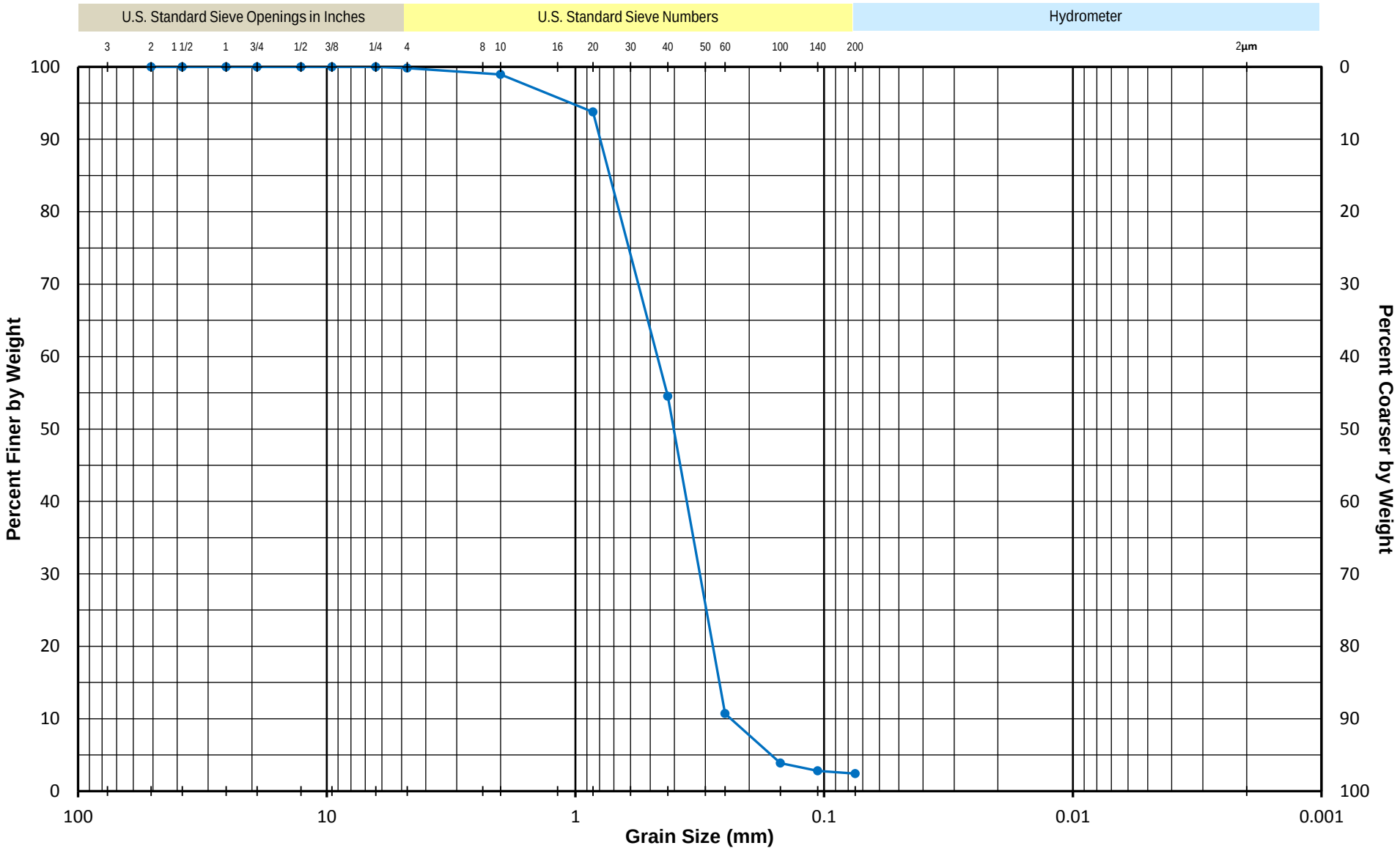


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC64 6-8	Brown Fine to Coarse Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

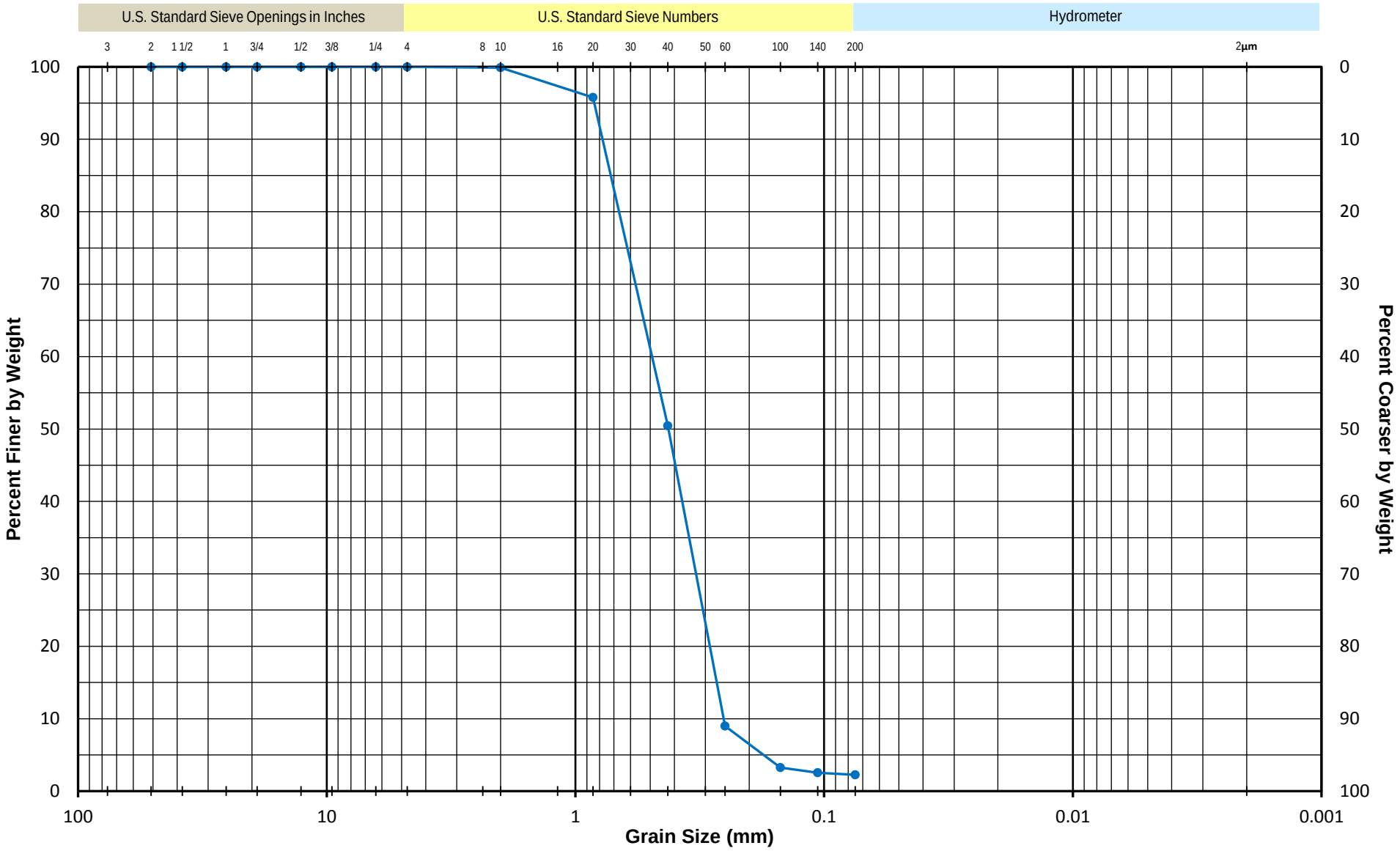


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC64 8-10	Dark Grayish Brown Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

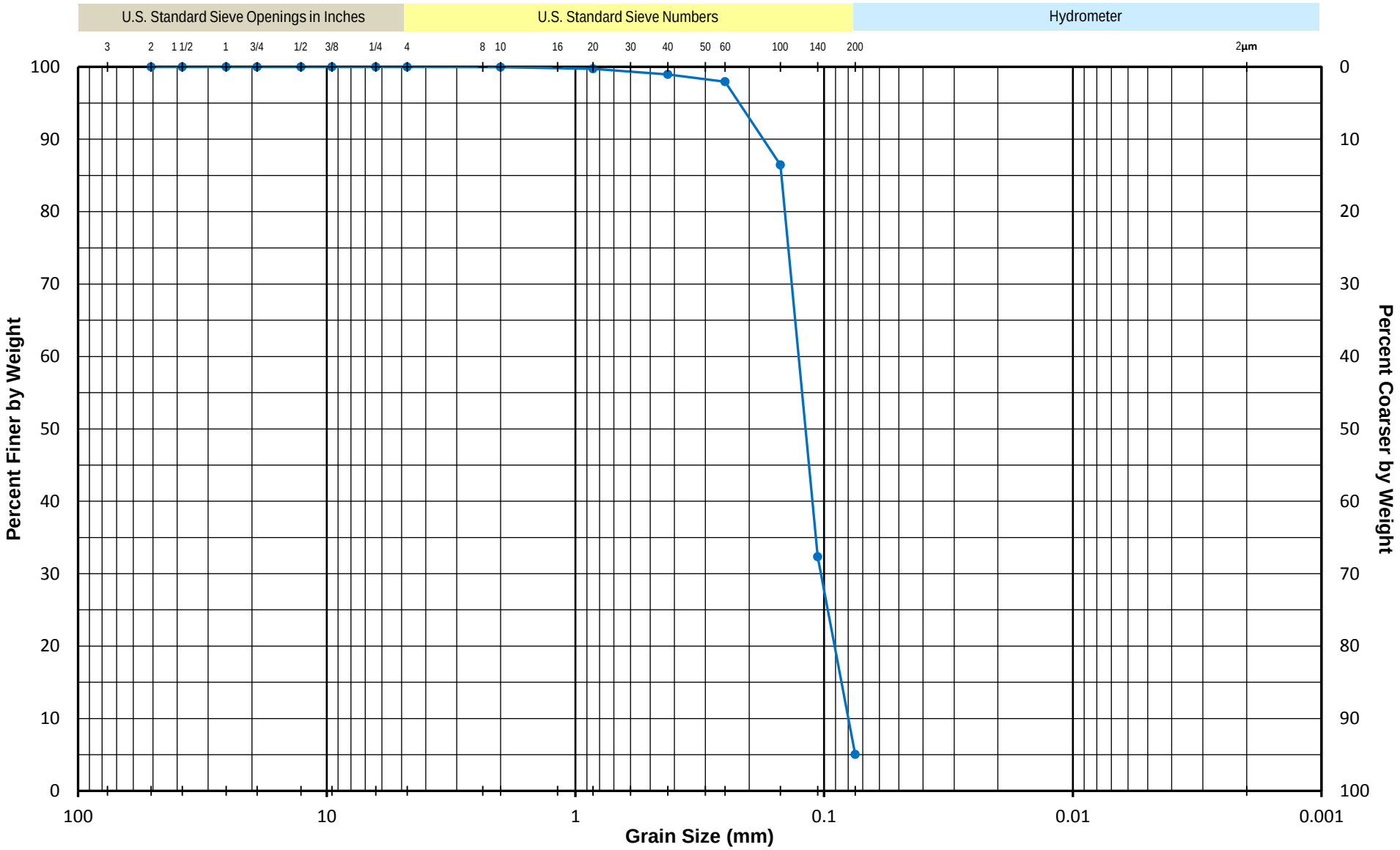


Test Depth	Core ID Section/Sample	Material Description
11.5 ft	VC64 10-12	Dark Grayish Brown Medium to Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

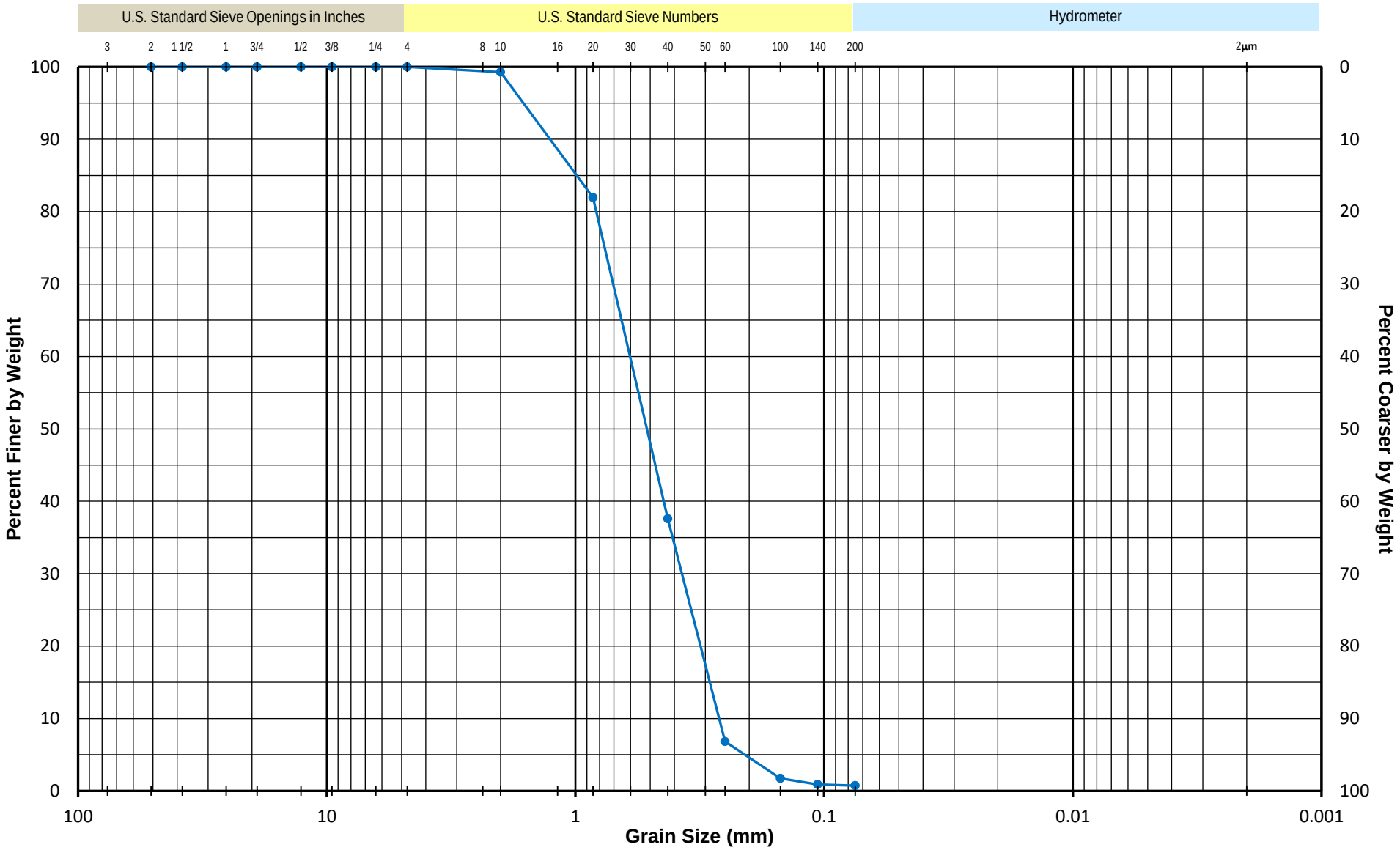


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC66 0-2	Very Dark Gray Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

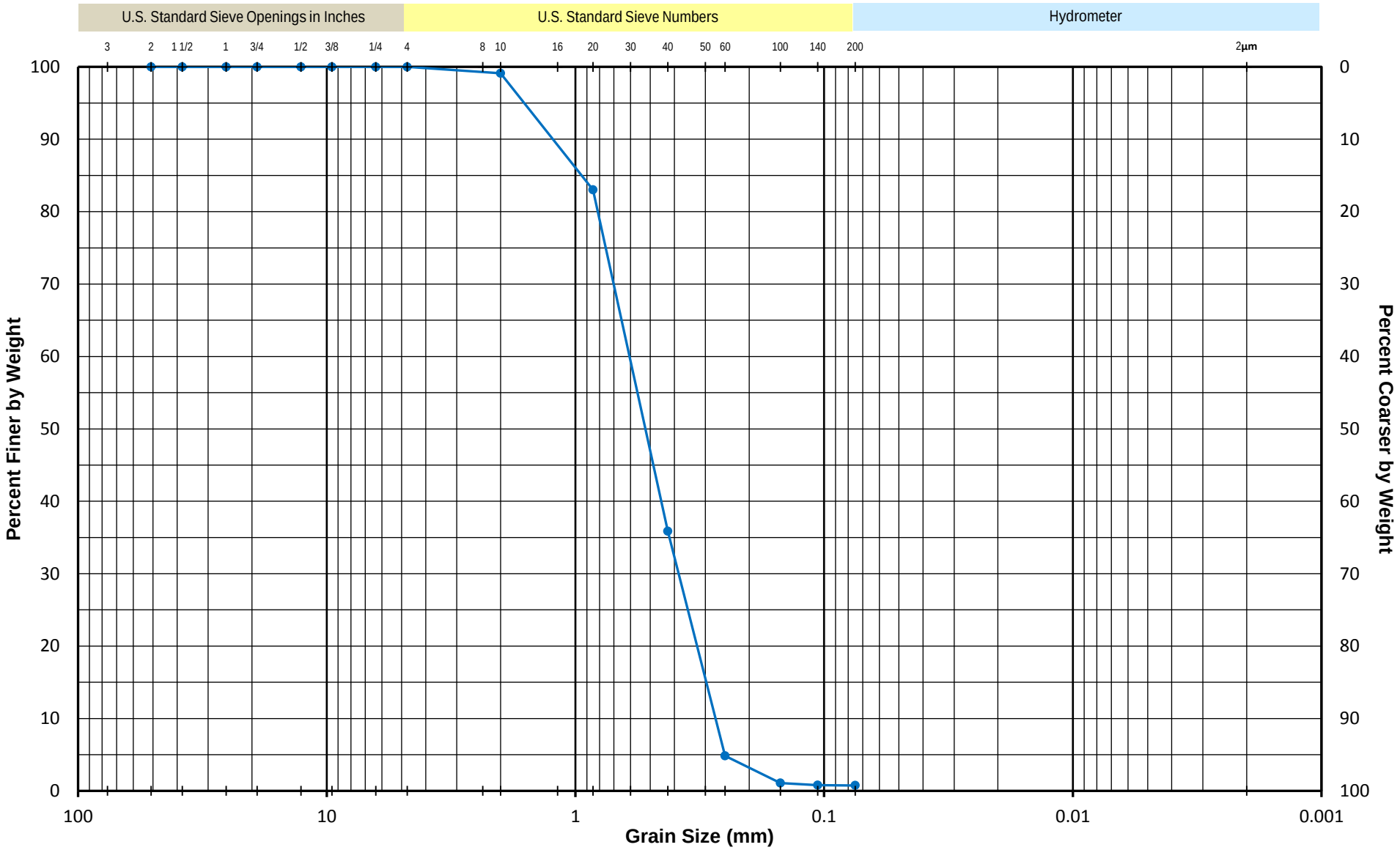


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC66 0-2	Dark Grayish Brown Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

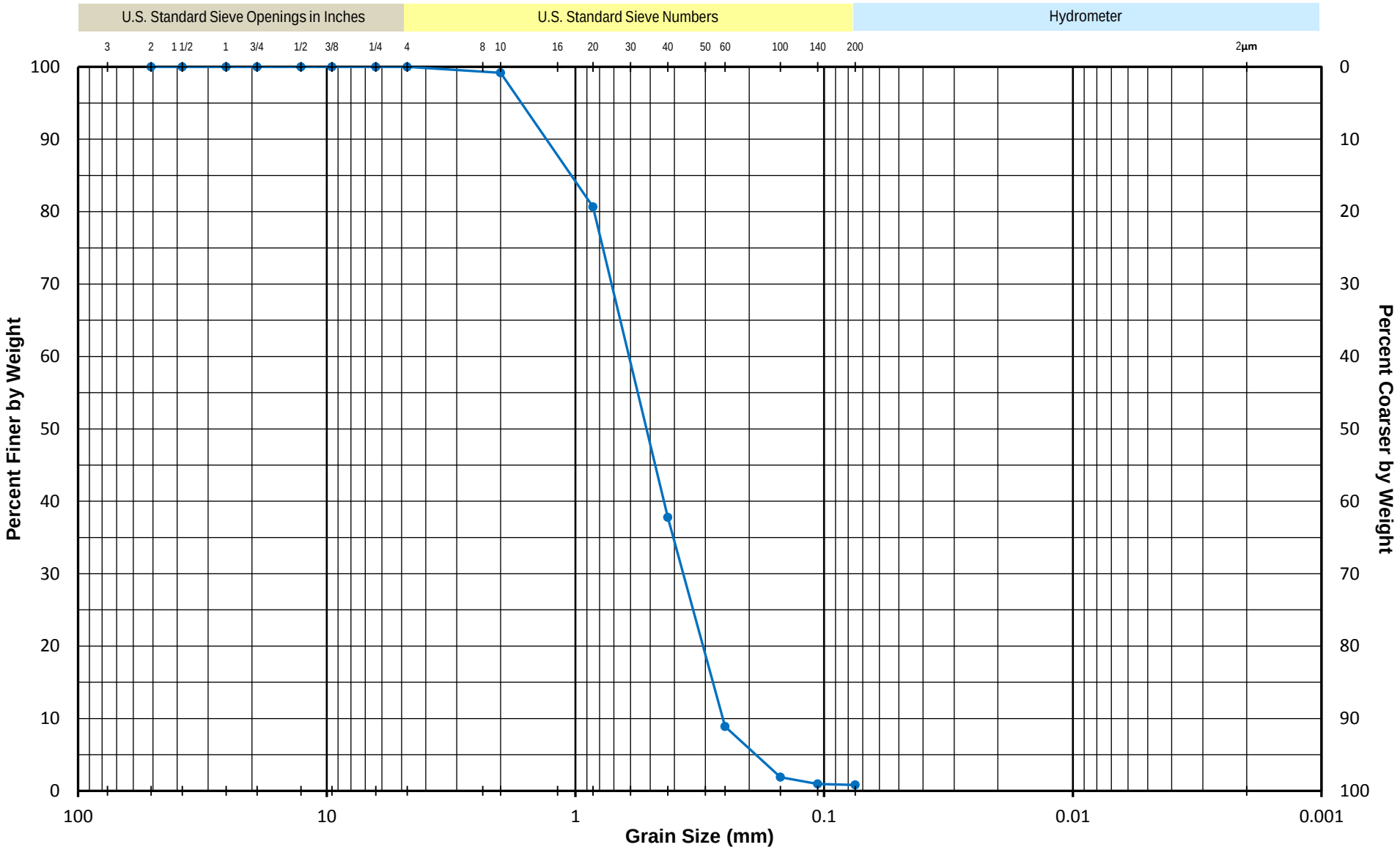


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC66 2-4	Dark Grayish Brown Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

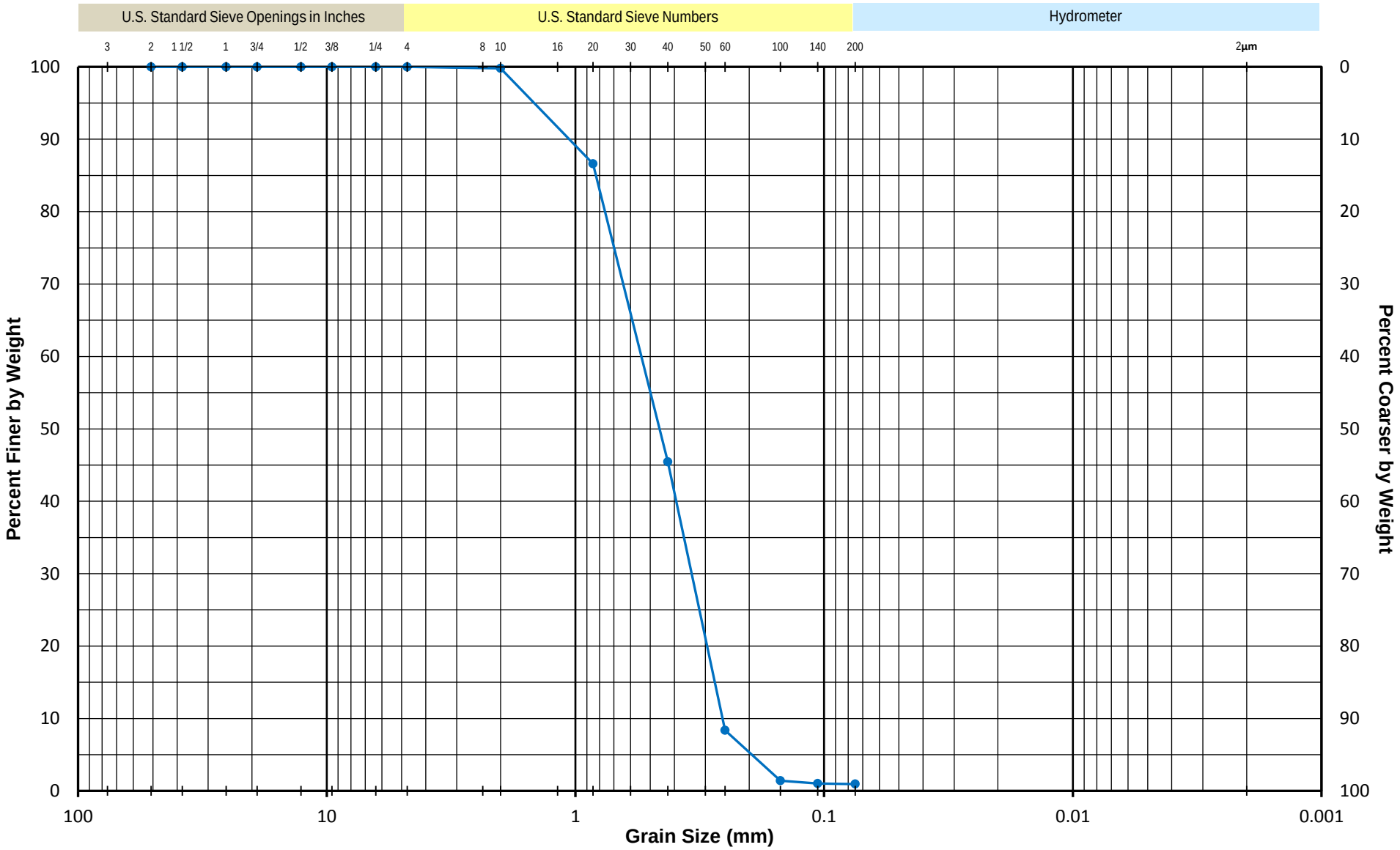


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC66 4-6	Dark Grayish Brown Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

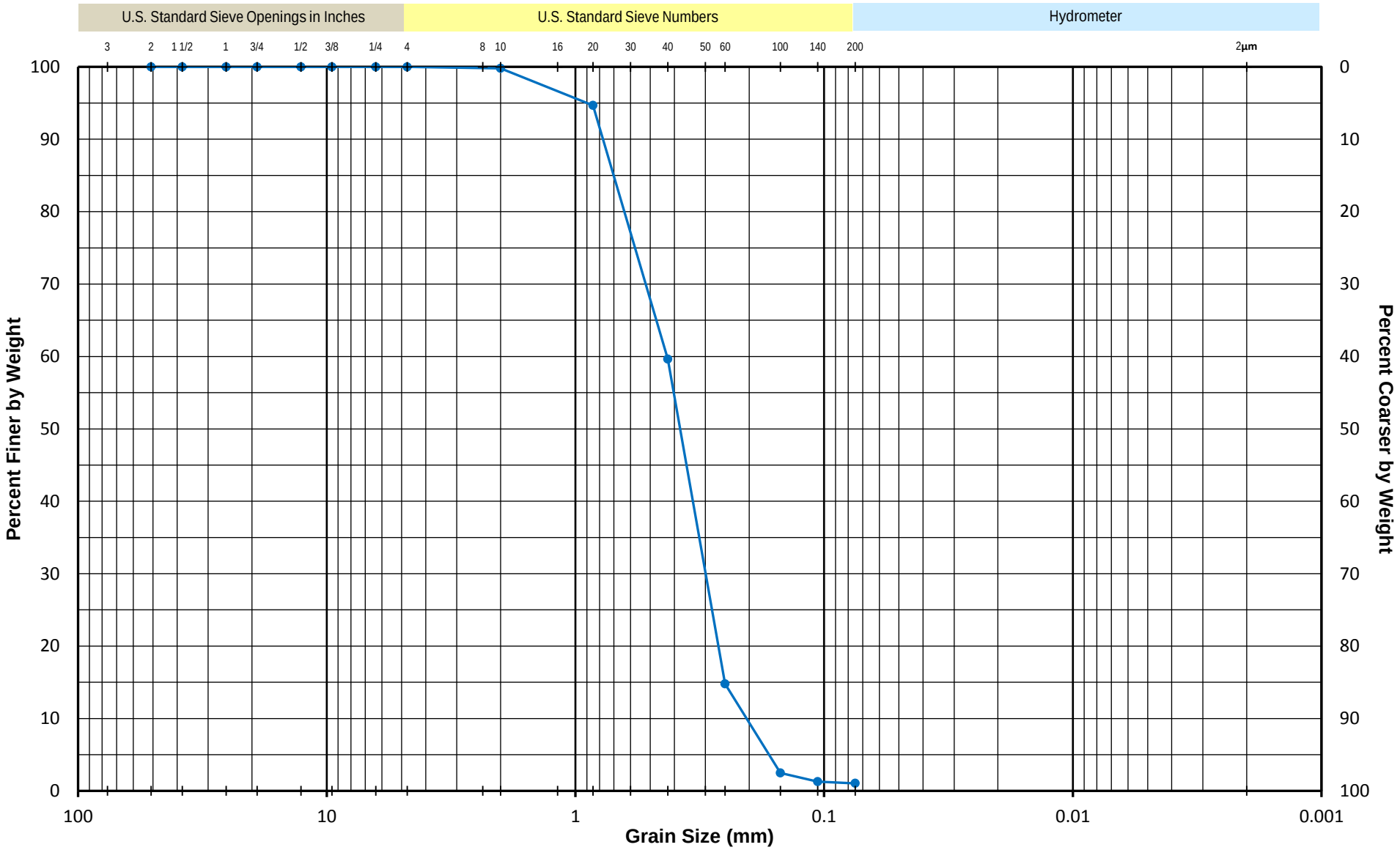


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC66 6-8	Dark Brownish Gray Medium to Fine Sand

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

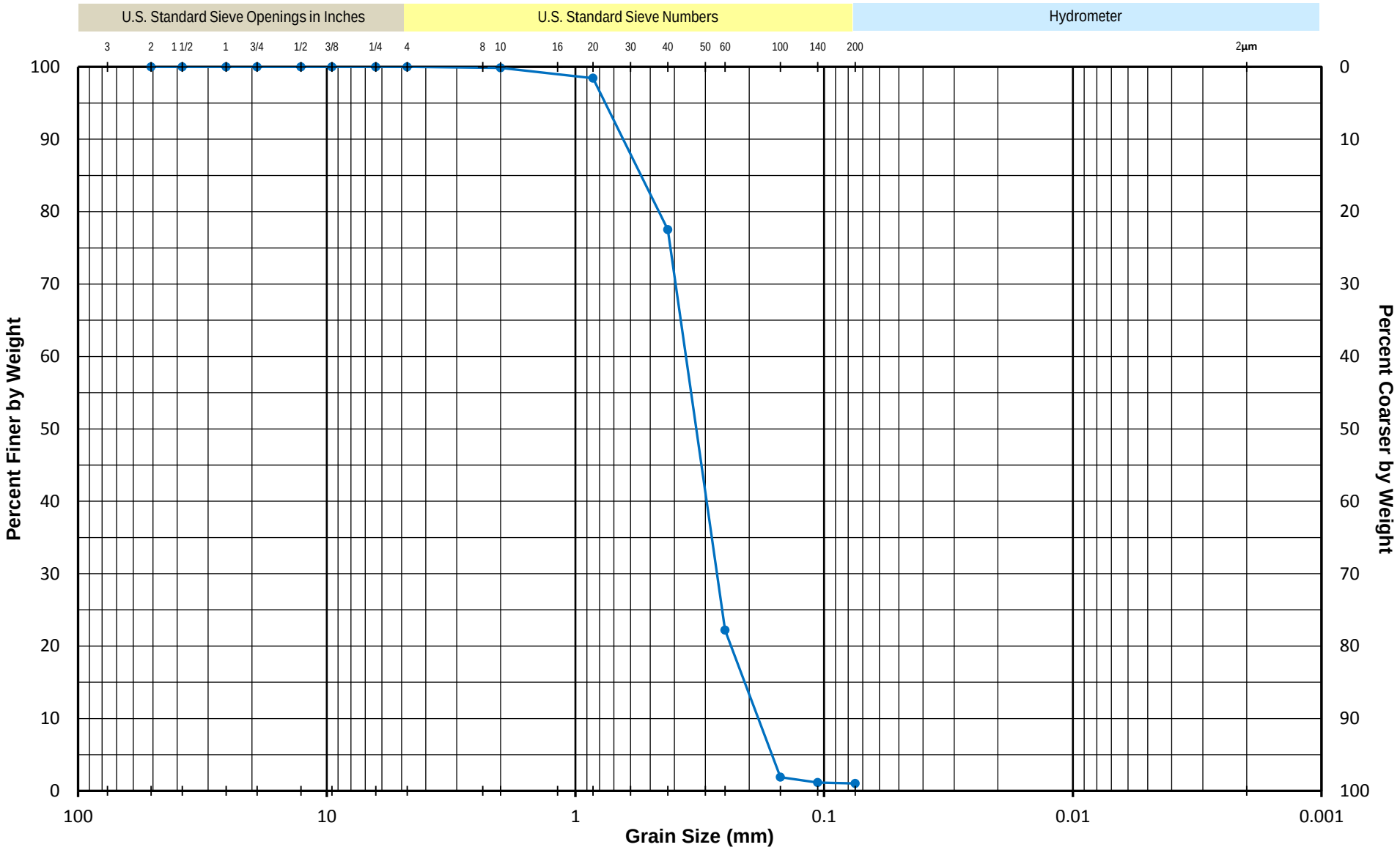


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC66 8-10	Dark Brownish Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

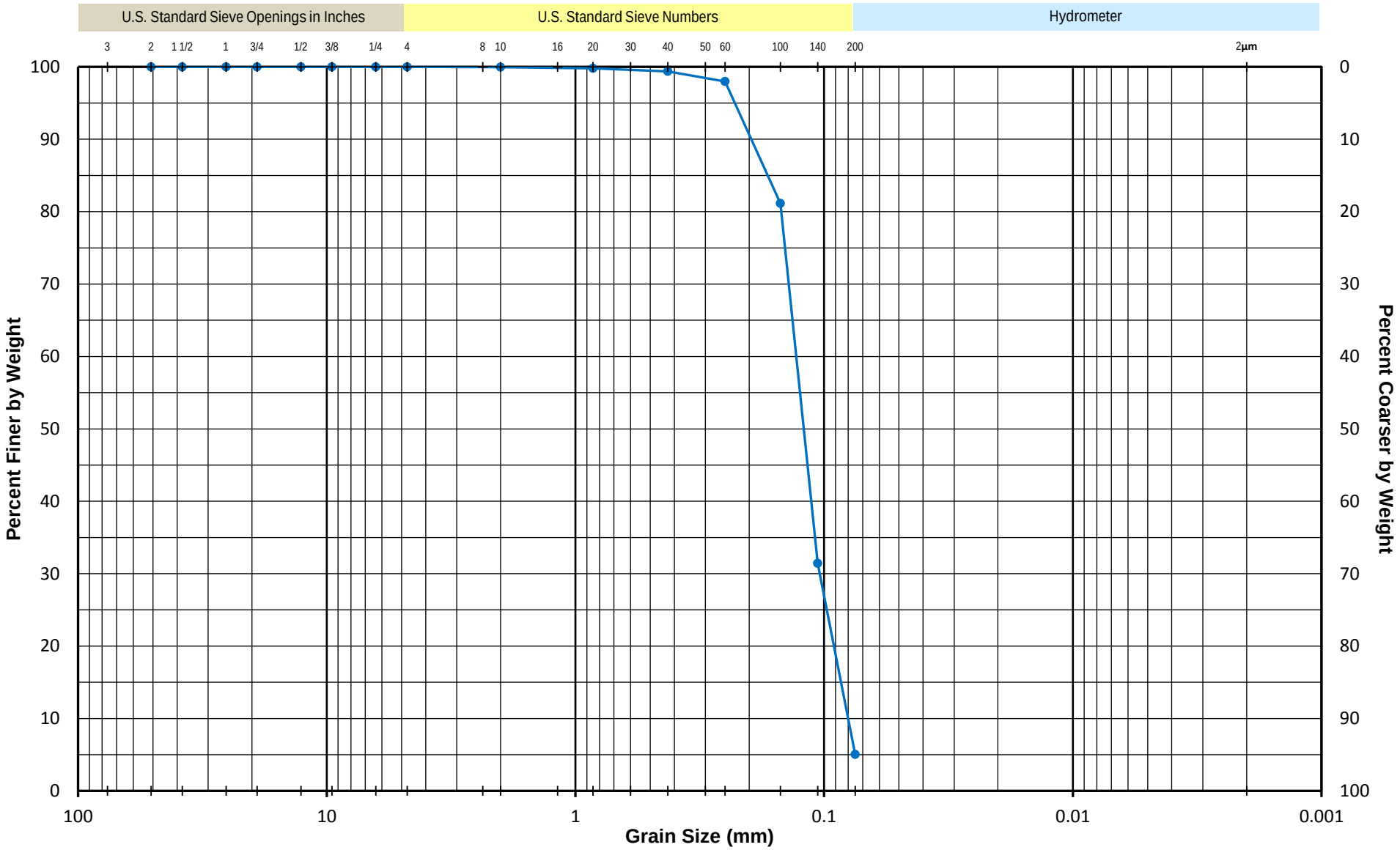


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC66 10-12	Very Dark Grayish Brown Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

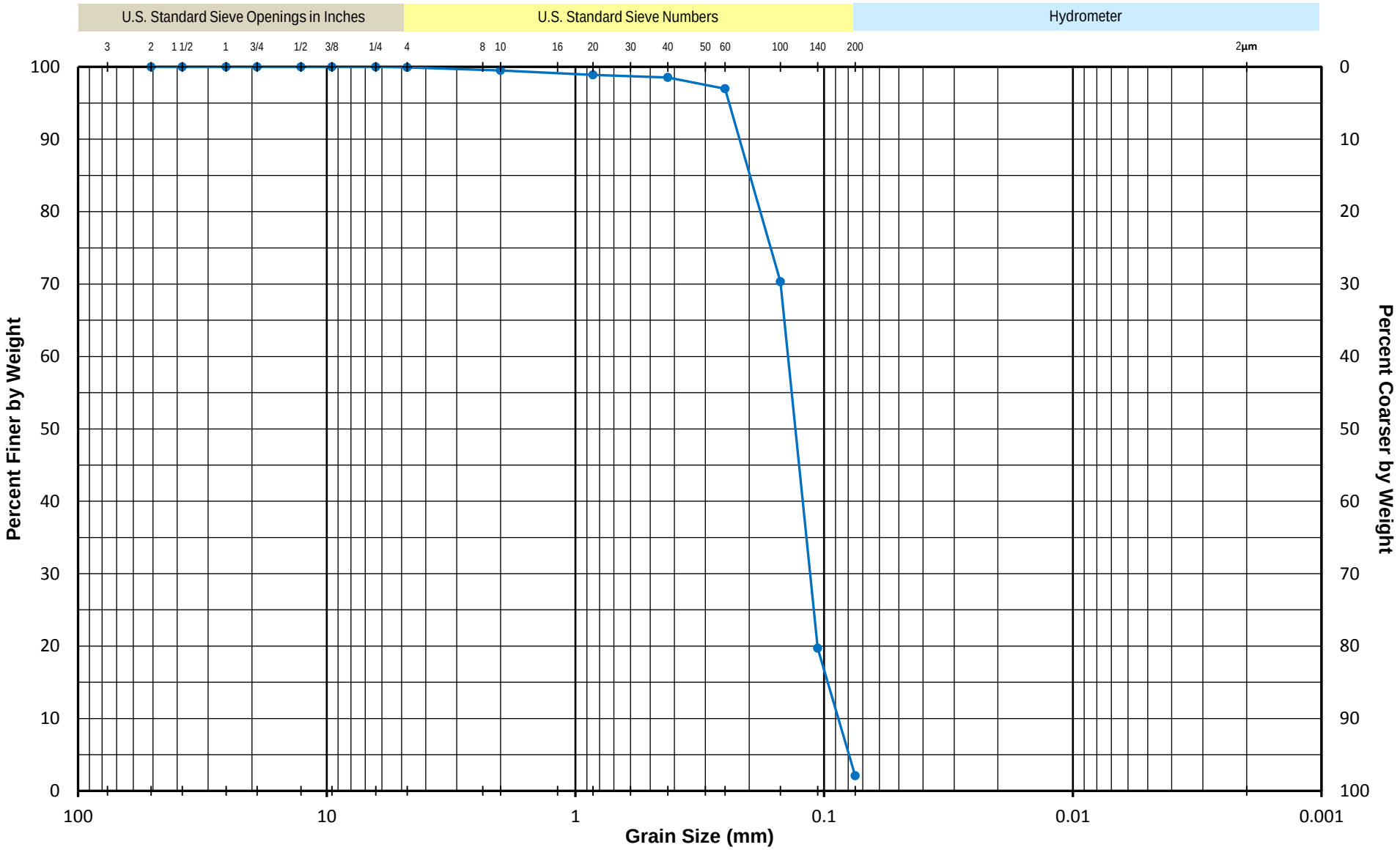


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC67 0-2	Very Dark Gray Fine Sand with silt, shell fragments, and organics

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

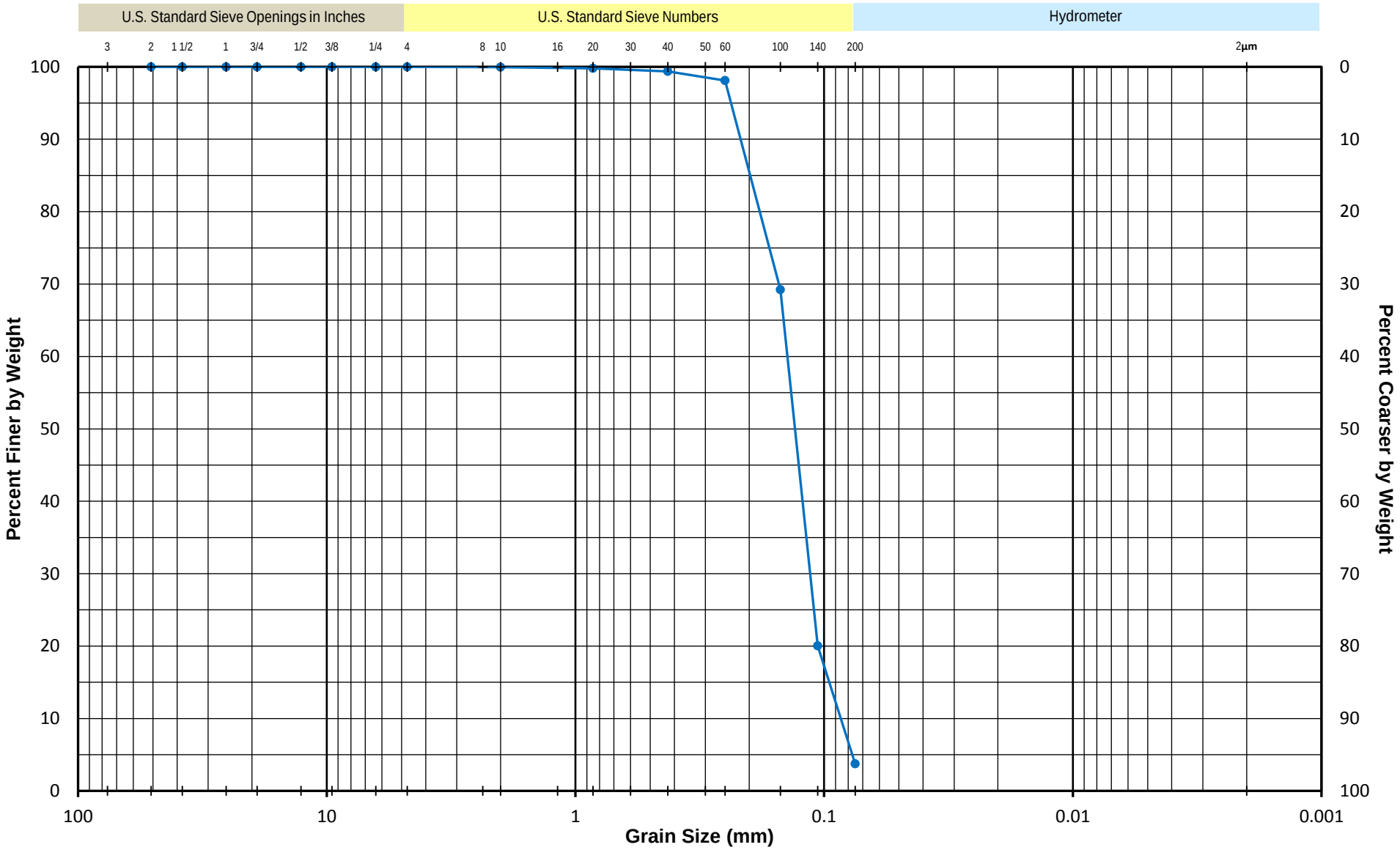


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC67 0-2	Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

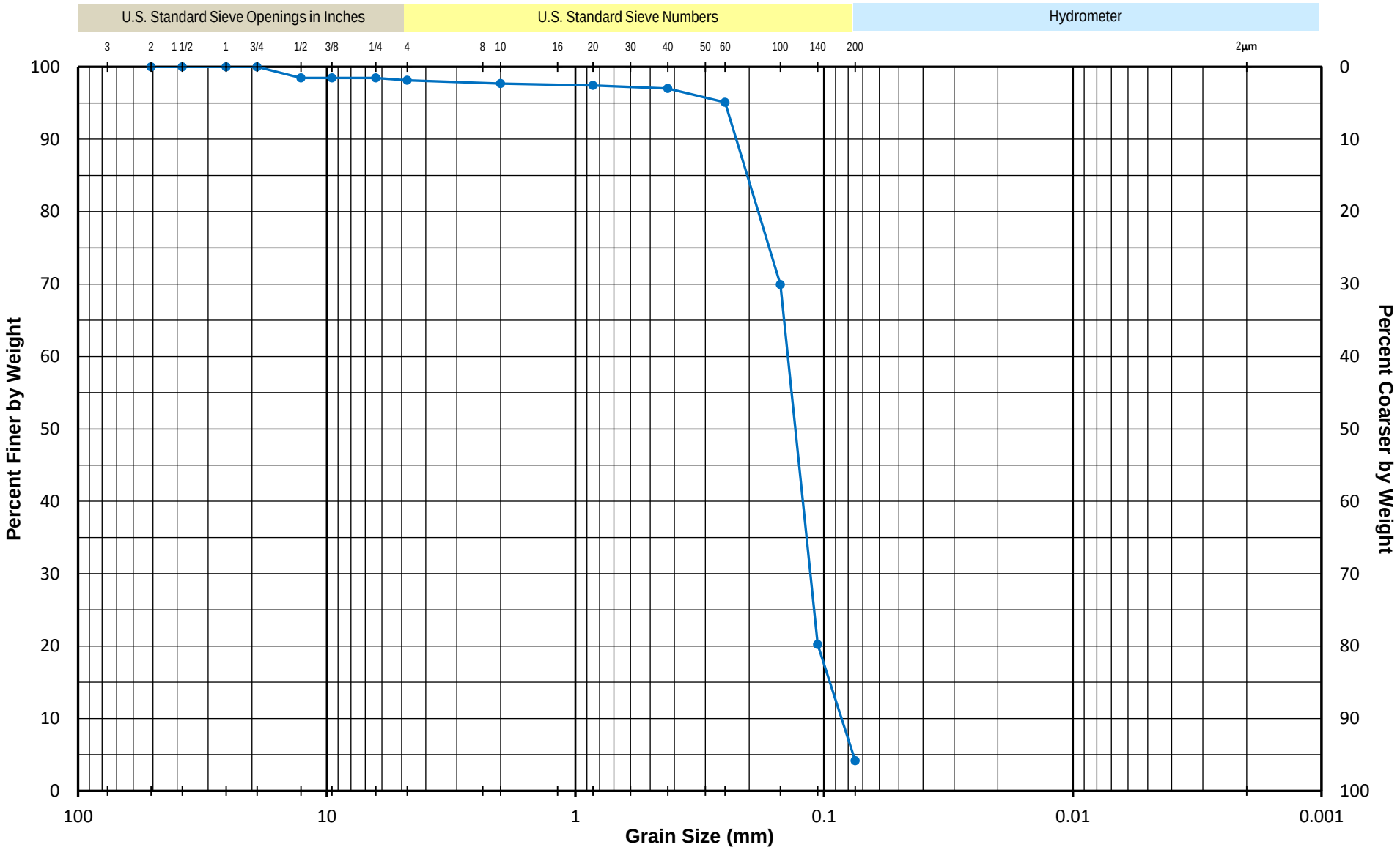


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC67 2-4	Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

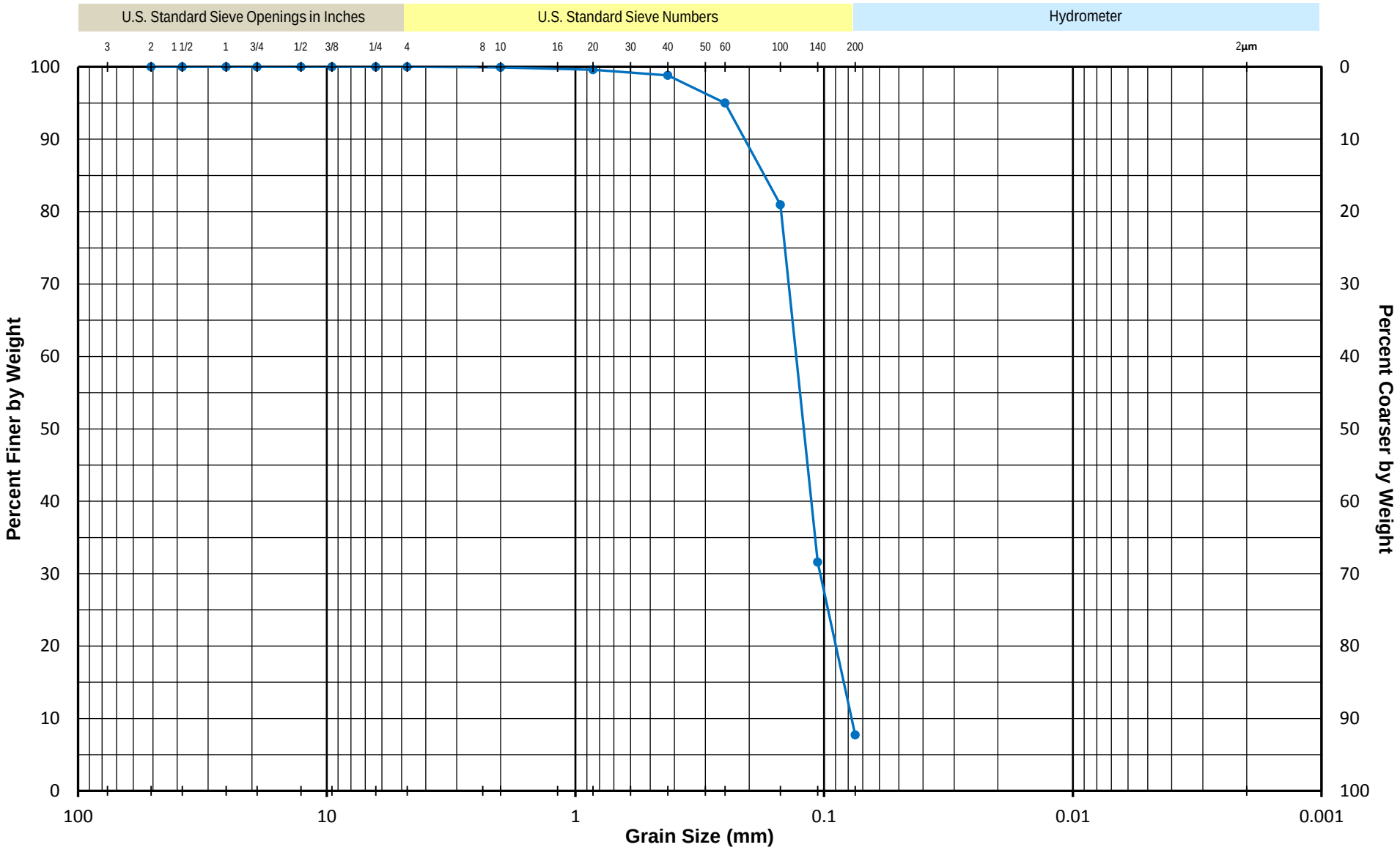


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC67 4-6	Very Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

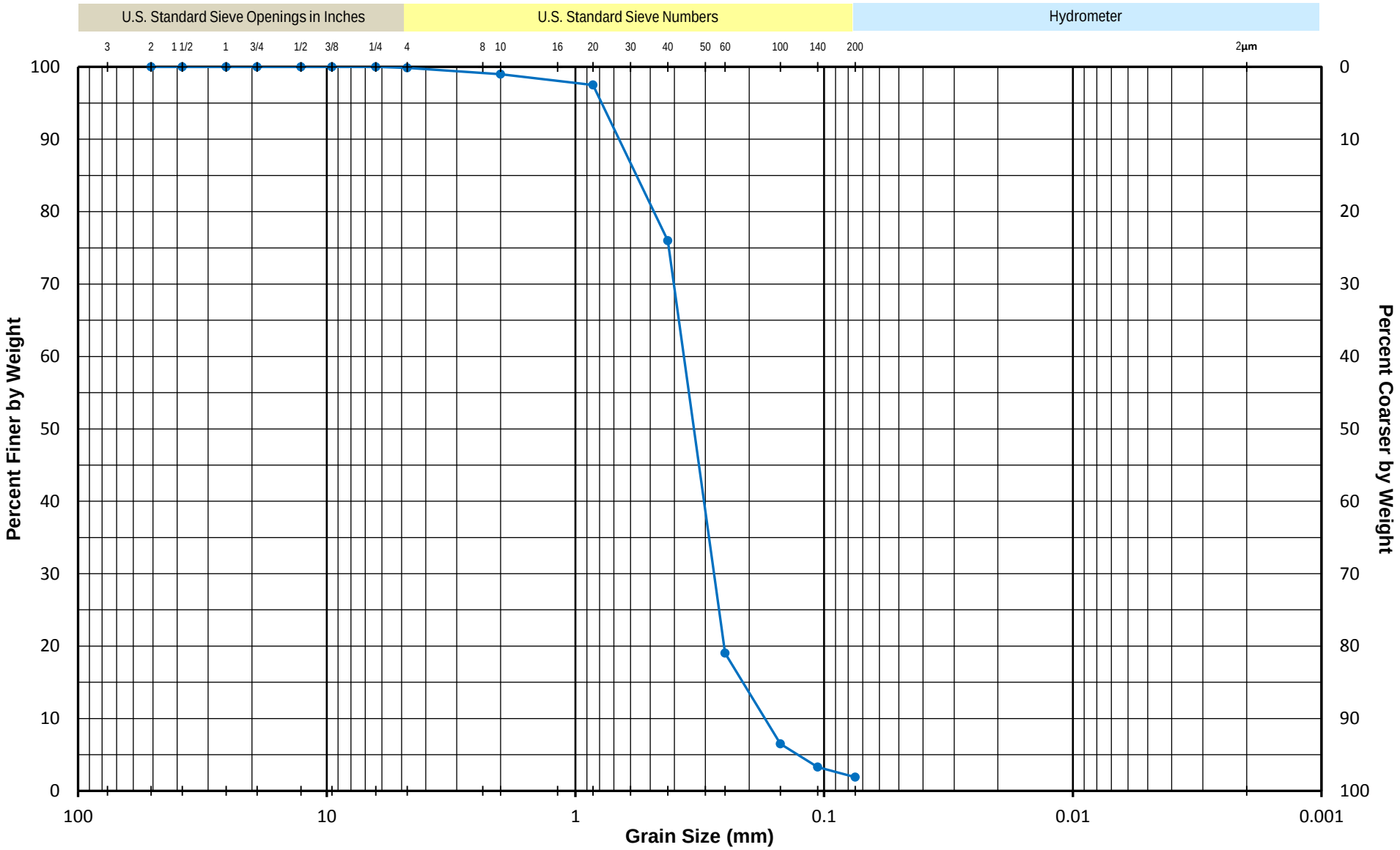


Test Depth	Core ID Section/Sample	Material Description
7.8 ft	VC67 6-8	Very Dark Gray Fine Sand with silt and shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

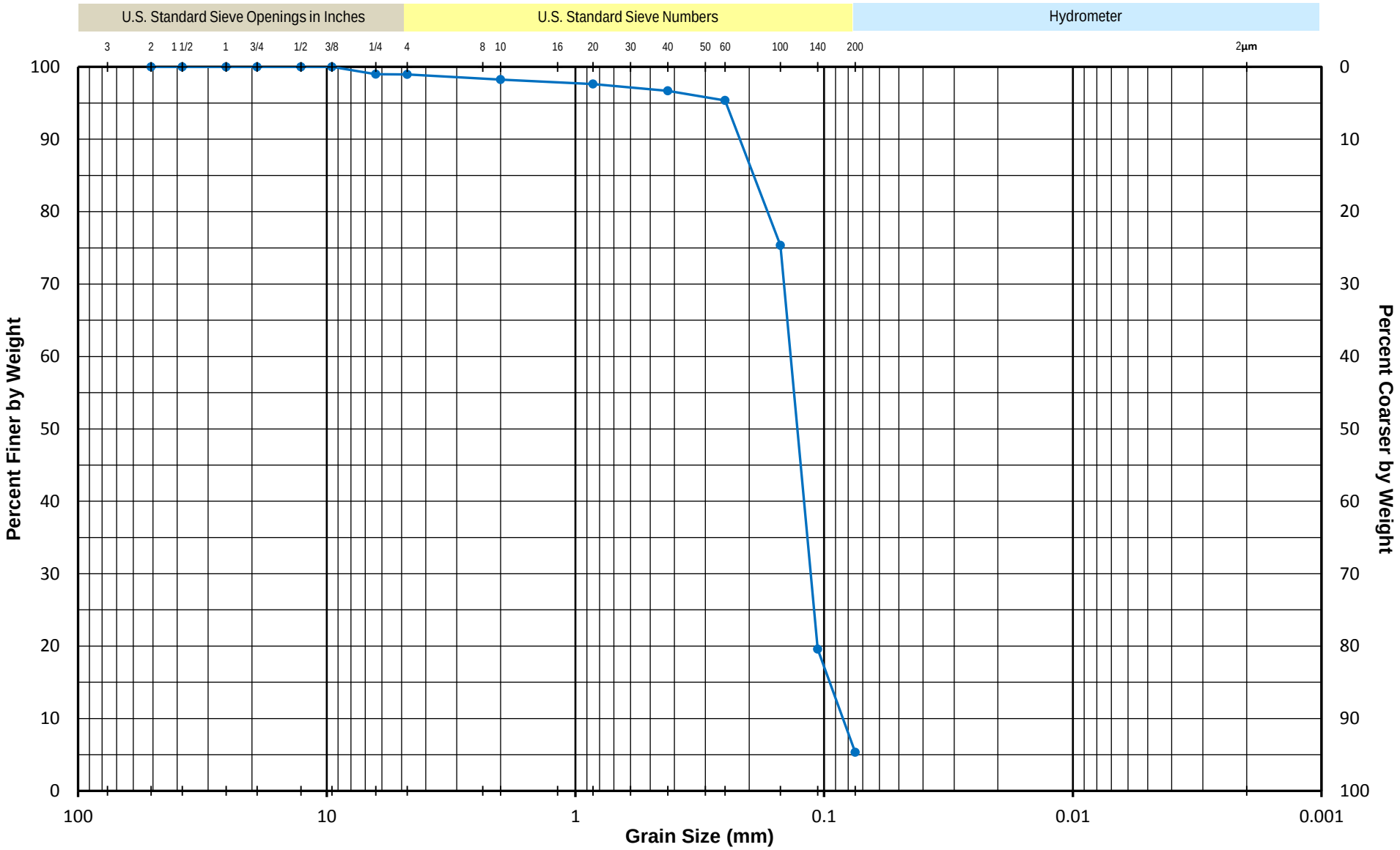


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC67 8-10	Very Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

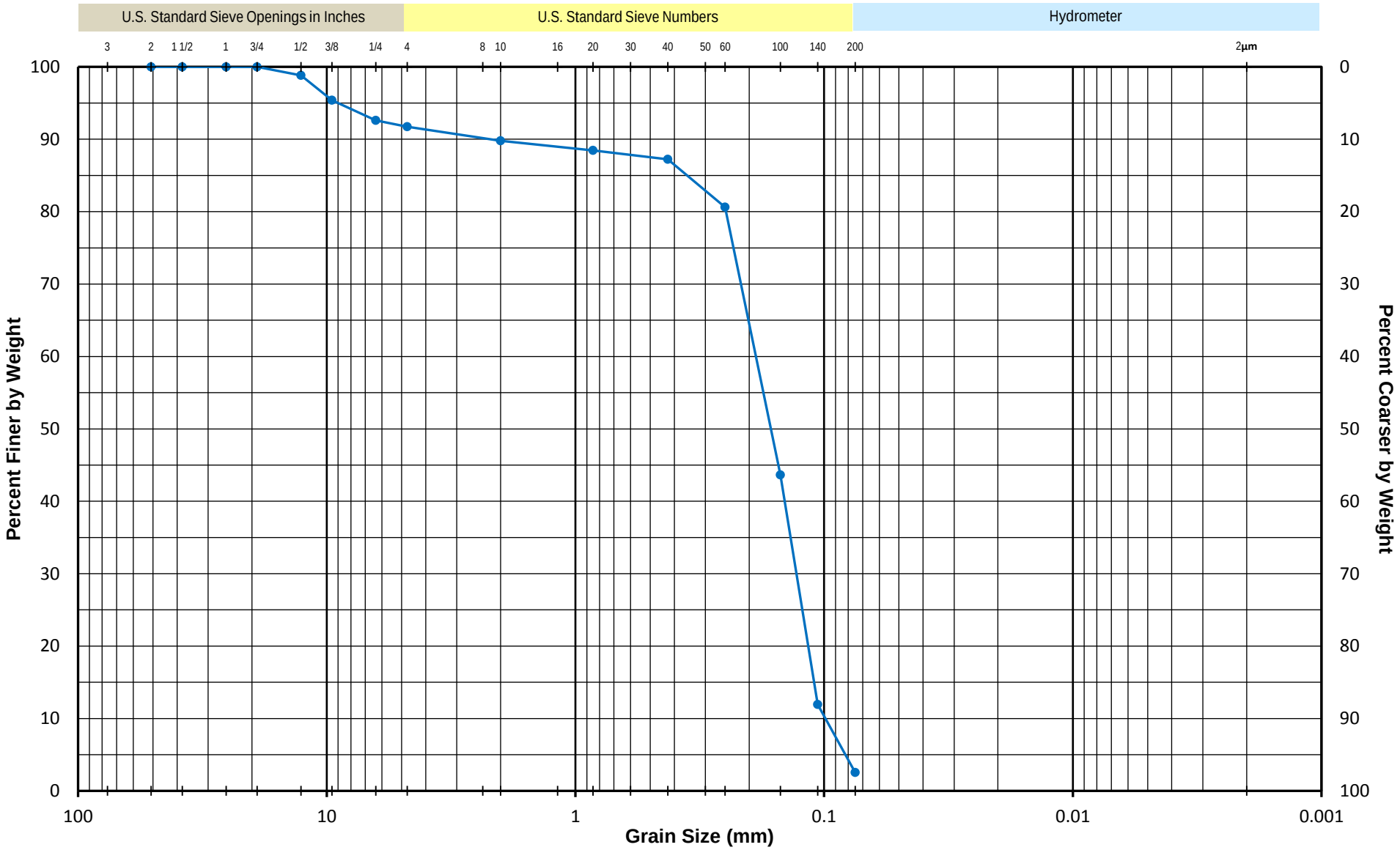


Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC67 10-12	Very Dark Gray Fine Sand with silt and shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

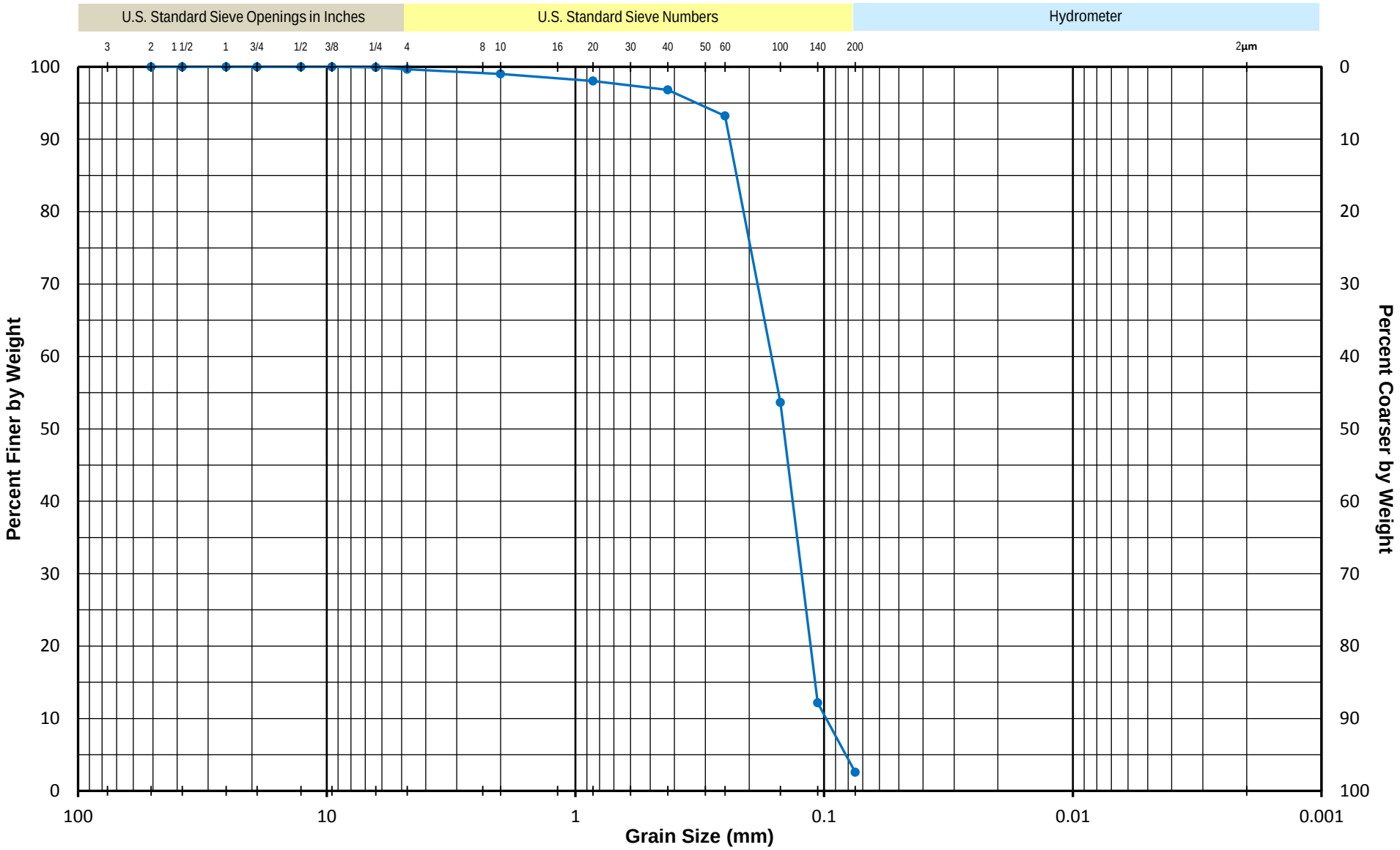


Test Depth	Core ID Section/Sample	Material Description
0.7 ft	VC68 0-2	Very Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

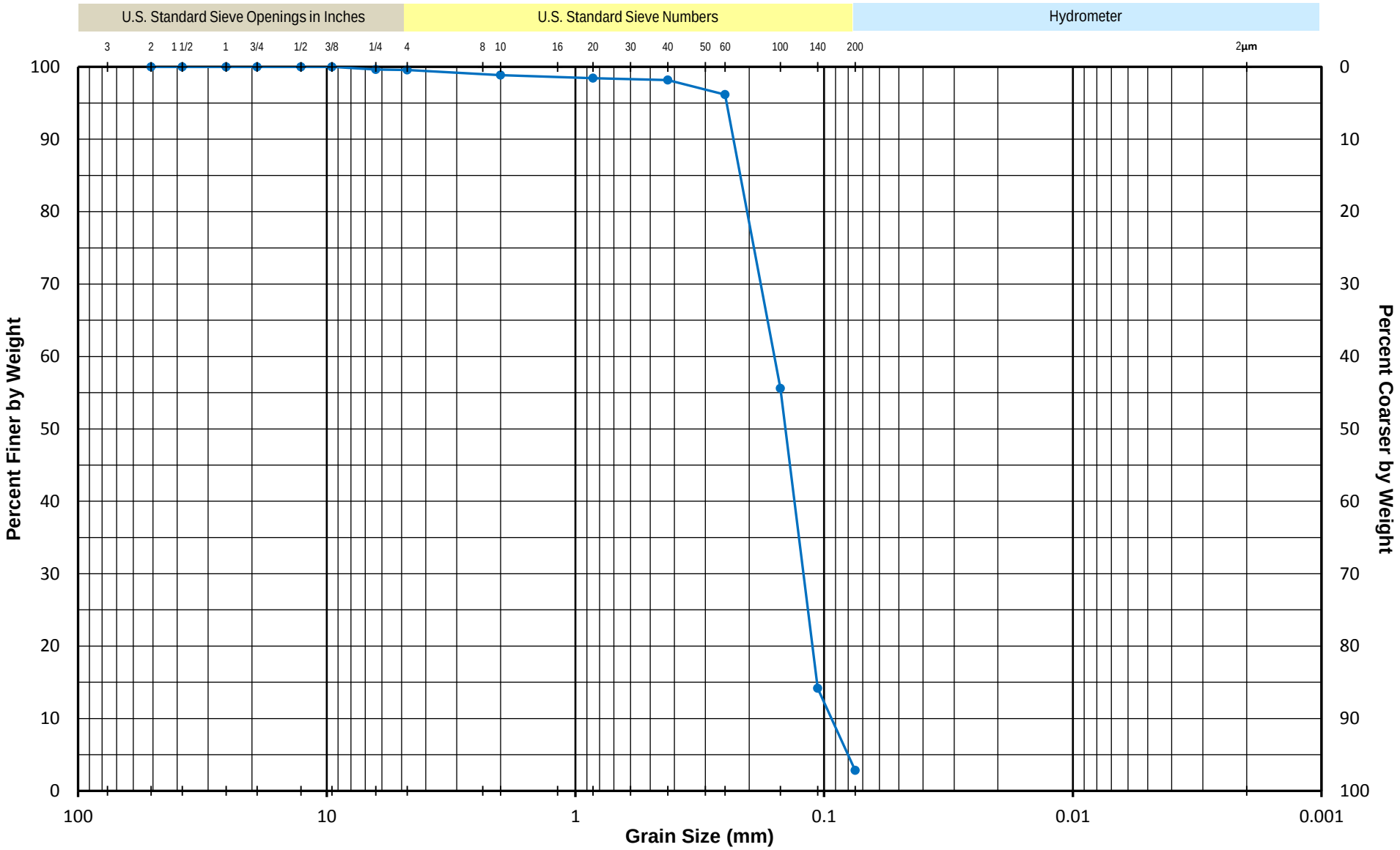


Test Depth	Core ID Section/Sample	Material Description
2.0 ft	VC68 0-2	Very Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

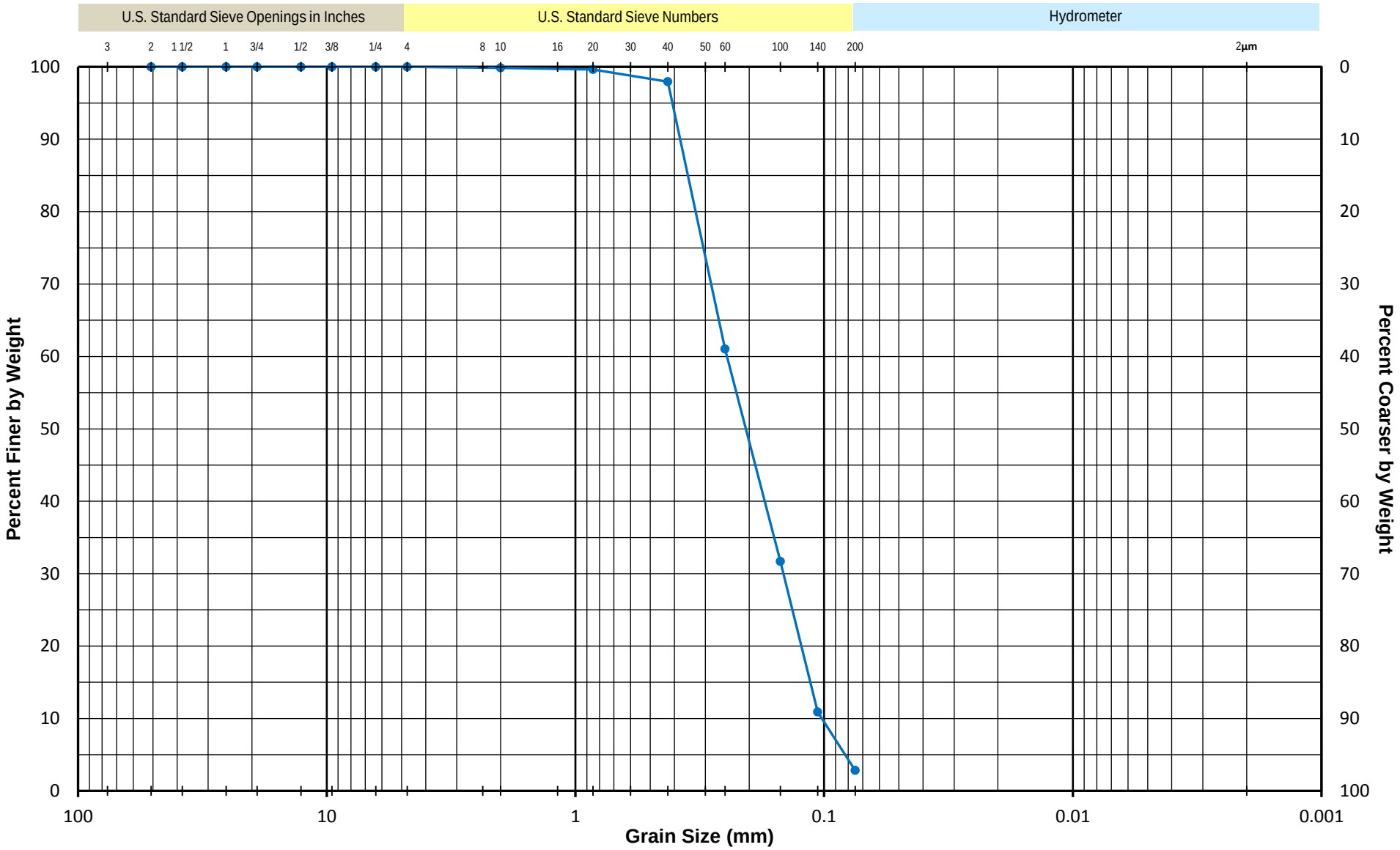


Test Depth	Core ID Section/Sample	Material Description
4.0 ft	VC68 2-4	Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

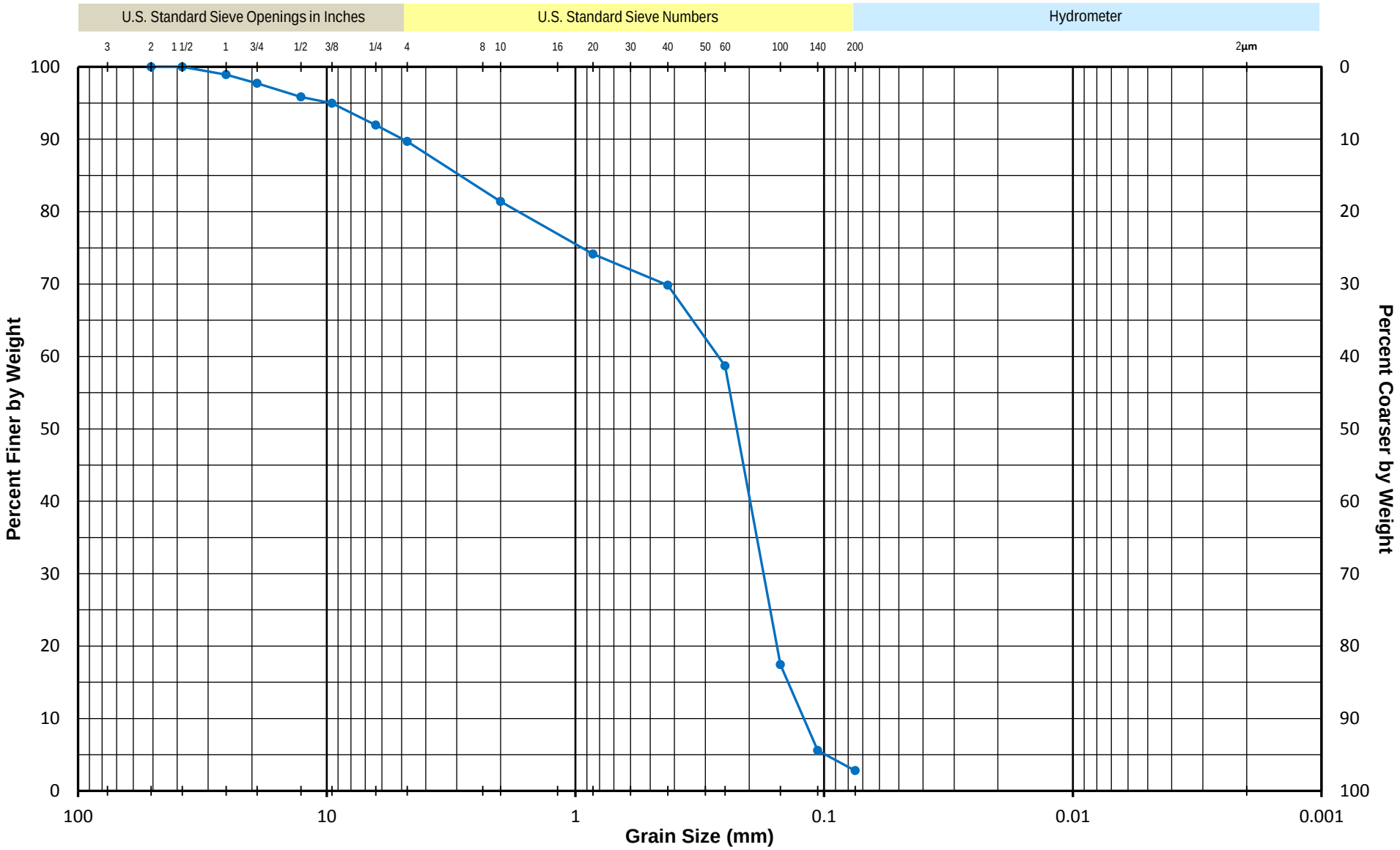


Test Depth	Core ID Section/Sample	Material Description
6.0 ft	VC68 4-6	Very Dark Gray Fine Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

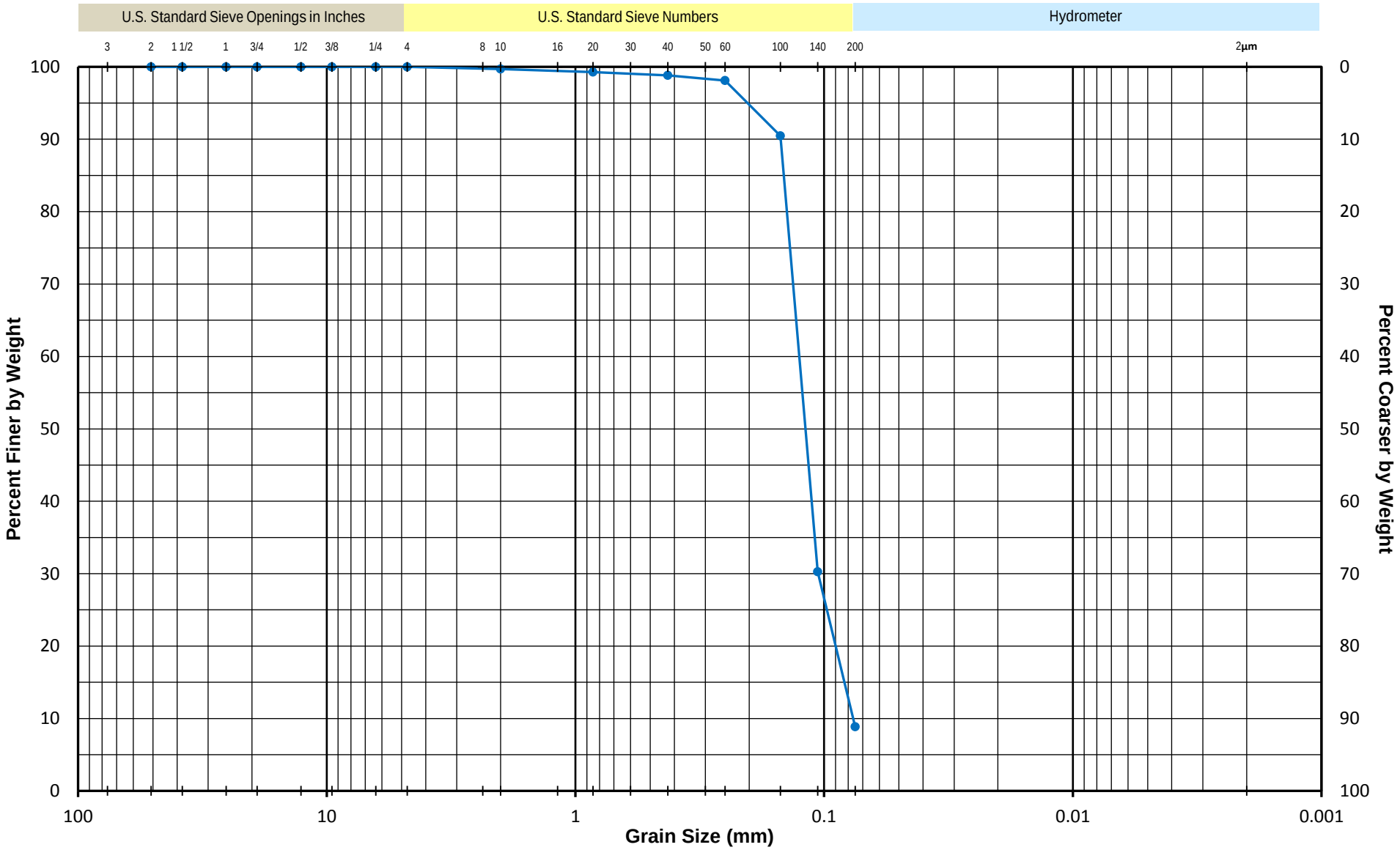


Test Depth	Core ID Section/Sample	Material Description
8.0 ft	VC68 6-8	Very Dark Gray Fine to Medium Sand with shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

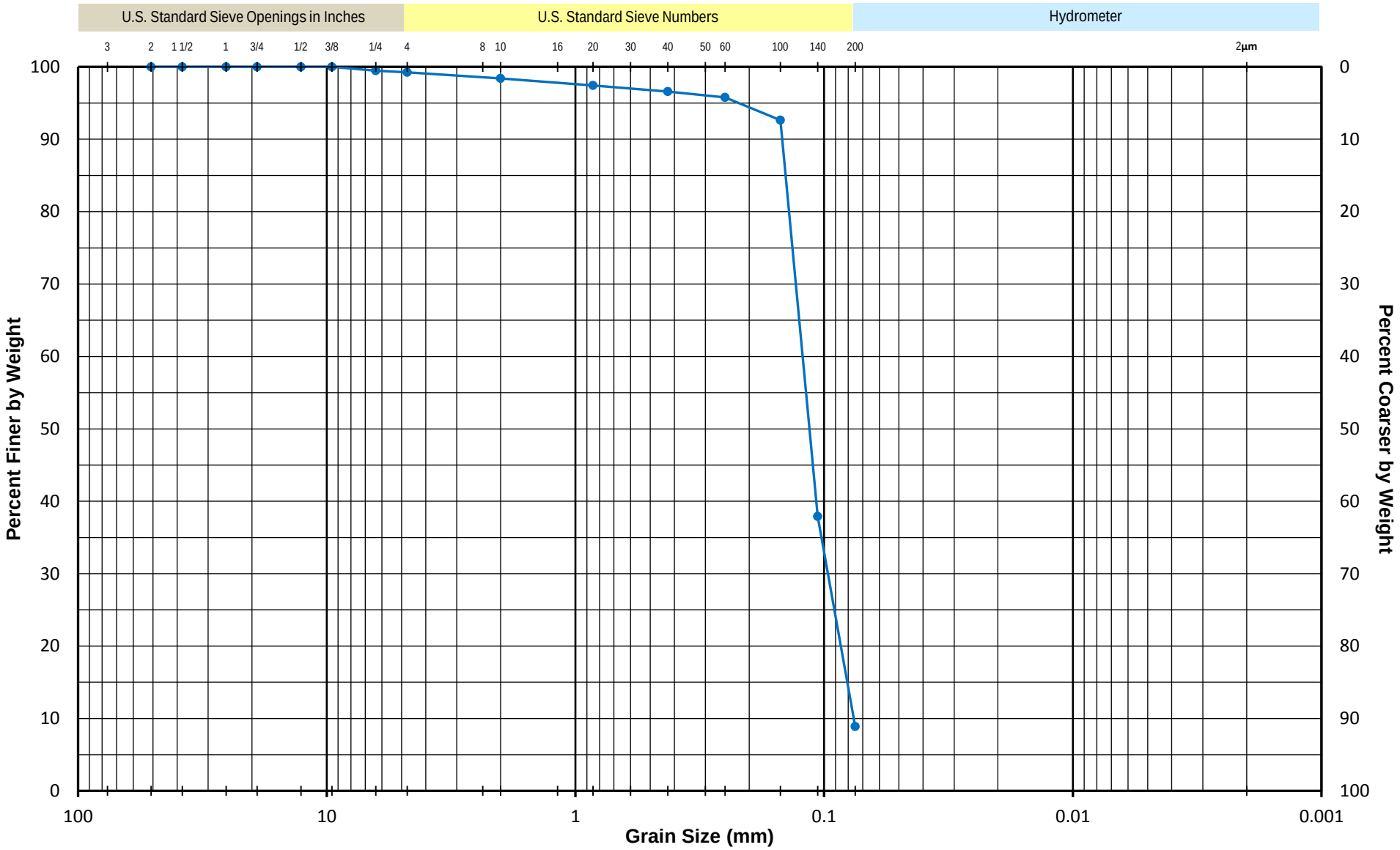


Test Depth	Core ID Section/Sample	Material Description
10.0 ft	VC68 8-10	Dark Gray Fine Sand with silt and shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II



Test Depth	Core ID Section/Sample	Material Description
12.0 ft	VC68 10-12	Dark Gray Fine Sand with silt and shell fragments

GRADATION CURVES

ASTM D-422, D-6913

Alpine - NESE II

B Core Logs

BORING NUMBER VC11

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.34
DRILLING METHOD Vibracore
CORE RECOVERY: 12.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:930281.95 Y:119832.47
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Very dark gray medium to fine Sand with Silt	1	100	0.9	112	71	135
		(SP) Dark brown coarse to fine Sand	2		1.4	105	63	127
		(SP) Dark brown medium to fine Sand	3		1.4	99	57	121
		(SP) Dark brown coarse to fine Sand	4		1.2	106	65	129
5		(SW) Dark brown fine to coarse Sand with Gravel	5		1.7 3.3	118	72	136
		(SP) Dark brown coarse to medium Sand with Gravel	6		4.5 5.7	122	68	132
		(SW) Brown coarse to medium Sand with Gravel	7		4.7 5	121	68	132
10		(SP) Brown medium to fine Sand	8		3.3	112	71	135
		(SP) Dark brown coarse to medium Sand	9		2.8	111	64	128
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.3 feet.

BORING NUMBER VC25

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.30
DRILLING METHOD Vibracore
CORE RECOVERY: 12.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:949132.07 Y:124379.23
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Very dark gray Sand with Silt and shell fragments	1	100	1.2	109	66	130
		(SP-SM) Dark gray Sand with Silt and shell fragments	2		1.1	106	65	129
		(SP) Brown coarse to medium Sand	3		1.7	121	73	137
			4		1.9	114	71	135
5		(SW) Dark brown coarse to medium Sand with Gravel	5		1.9	115	67	131
					6.4			
		(SP) Dark brown fine to medium Sand	6		13.9	113	67	131
					9.2			
		(SP) Dark brown medium to coarse Sand	7		11.3	122	73	137
10					7.6			
		(SW) Dark brown coarse to fine Sand	8		10.9	114	67	131
			9		9.7	111	67	131
15								

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
□ FINES CONTENT (%) □ 20 40 60 80

Bottom of borehole at 16.3 feet.

BORING NUMBER VC26

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.44
DRILLING METHOD Vibracore
CORE RECOVERY: 12.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:951144.82 Y:124410.16
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0	(SP) Very dark gray fine Sand with shell fragments	1	100	0.1	106	64	128
	(SW-SM) Very dark gray coarse to fine Sand with Silt	2		0.7	107	65	129
	(SW-SC) Very dark gray coarse to fine Sand with Clay	3		0.7	114	69	133
	(SW-SM) Very dark gray fine to coarse Sand with Silt	4		1.4	112	68	132
5	(SP) Very dark gray fine to coarse Sand with Gravel; Lense of strong brown Silty Sand with greenish gray Silt from 5.7-5.9'	5		0.7	122	73	137
		6		1.2			
	(SM) Strong brown Silty Sand	7		2.3	120	72	136
				5.4			
	(SP) Strong brown coarse to fine Sand	8		15.4	121	73	137
10				25			
	(SP) Dark brown fine to medium Sand	9		14.7	105	63	127
	(SP) Dark brown medium to fine Sand	10		13.8	109	65	129
15							

▲ SECONDS PER FOOT ▲ (20 40 60 80)
PL MC LL (20 40 60 80)
☐ FINES CONTENT (%) ☐ (20 40 60 80)

Bottom of borehole at 16.4 feet.

BORING NUMBER VC28

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.95
DRILLING METHOD Vibracore
CORE RECOVERY: 14.30
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:954827.41 Y:125889.14
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0	(SC) Black Clayey Sand with shell fragments	1	100	3.3	98	58	122
	(SC) Very dark gray Clayey Sand with shell fragments	2		1.9	98	59	123
		3		1.6	99	59	123
	(SP-SC) Very dark gray Gravelly Sand with Clay and shell fragments	4		1.6	107	64	128
5	(SM) Dark gray Silty fine to medium Sand	5		3.7	113	67	131
				2.1			
	(SP) Dark gray Silty medium to fine Sand	6		1.9	115	69	133
				5.1			
	(SP-SM) Dark gray fine to coarse Sand with Silt	7		4.4	112	67	131
10				7			
	(SP) Dark brown medium to coarse Sand	8		5.4	114	71	135
	(SW) Dark brown fine to medium Sand	9		4.7	105	65	129
	No Analysis						
15							

▲ SECONDS PER FOOT ▲ 20 40 60 80
PL MC LL 20 40 60 80
□ FINES CONTENT (%) □ 20 40 60 80

Bottom of borehole at 16.0 feet.

BORING NUMBER VC30

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/18/17 **COMPLETED** 5/18/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.76
DRILLING METHOD Vibracore
CORE RECOVERY: 12.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:958257.98 Y:127881.29
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / □ FINES CONTENT (%) □ 20 40 60 80
0		(SP-SM) Very dark gray Sand with Silt and shell fragments	1	100	1	101	62	126	
		(SM) Very dark gray Silty fine Sand with coarse Sand and shell fragments	2		1.8	100	61	125	
		(SP-SM) Very dark gray fine to medium Sand with Silt and shell fragments	3		1.8	107	64	128	
		(SM) Very dark gray Silty Sand with shell fragments	4		1.4	104	64	128	
5		(SP-SM) Very dark gray medium to fine Sand with Silt	5		0.9 1.8	117	71	135	
		(SP) Brown fine to coarse Sand with Gravel	6		2.3 6.1	118	71	135	
		(SP) Strong brown fine to medium Sand	7		8.2 11.8	102	62	126	
10		(SP) Brown fine to medium Sand	8		10.7	105	63	127	
		(SP) Dark brown fine to medium Sand	9		9.1	107	63	127	
15									

Bottom of borehole at 15.8 feet.

BORING NUMBER VC33

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/18/17 **COMPLETED** 5/18/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 15.21
DRILLING METHOD Vibracore
CORE RECOVERY: 12.10
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:963512.39 Y:130860.41
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Very dark gray fine to medium Sand	1	100	2.6	101	58	122
		(SP) Dark brown fine Sand	2		4	105	63	127
		(SP-SM) Very dark gray medium to fine Sand with Silt and shell fragments	3		7	111	68	132
		(SP) Dark gray medium to coarse Sand with Gravel	4		1.6	117	72	136
5		(SM) Brown and gray Silty fine Sand	5		1.4 1.9	107	64	128
		(SP) Brown fine to medium Sand	6		1.6 3.9	109	65	129
		(SP) Brown medium to coarse Sand with Gravel	7		4.9 4.4	112	65	129
10		(SP) Very dark grayish brown fine to medium Sand	8		2.3	110	67	131
		(SP) Very dark grayish brown fine to coarse Sand	9		3.9	113	68	132
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 15.2 feet.

BORING NUMBER VC34

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.77
DRILLING METHOD Vibracore
CORE RECOVERY: 14.40
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:965236.05 Y:131624.39
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Gray fine to medium Sand with shells; disturbed	1	100	1.1	95	50	114
		(SW) Very dark grayish brown fine to medium Sand with Gravel	2		1.4	110	66	130
		(SP) Very dark grayish brown fine to medium Sand	3		1.6	108	65	129
		(SP) Gray fine to medium Sand with shell fragments	4		3.3	103	64	128
5		(SP) Gray fine to medium Sand	5		2.1 2.6	109	65	129
		(SP-SM) Dark gray fine to medium Sand with Silt	6		2.3 3	108	64	128
		(SP) Very dark gray medium to fine Sand with shell fragments	7		6 10	109	62	126
10		(SP) Black fine to medium Sand	8		8.8	109	67	131
		(SP) Dark grayish brown medium to fine Sand	9		9.1	108	64	128
		No Analysis						
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.8 feet.

BORING NUMBER VC42

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/18/17 **COMPLETED** 5/18/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.37
DRILLING METHOD Vibracore
CORE RECOVERY: 15.20
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:974998.52 Y:130401.19
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Dark gray fine to medium Sand with shells	1	100	0.01	100	59	123
			2		0.1	104	62	126
		(SP) Gray fine to medium Sand	3		0.5	101	59	123
		(SP-SC) Black fine Sand with Clay seams and shell fragments, petrol odor	4		1.4	73	43	107
5		(SP-SC) Black fine to medium Sand with Clay seams and shell fragments, petrol odor	5		0.7	94	58	122
					1.2			
		(CH) Black Clay with Sand and shells	6		0.7	48	25	89
					0.9			
		(SP-SC) Black fine Sand with Clay pockets and shells	7		0.9	89	53	117
10					1.2			
		(SP) Gray fine Sand with shell fragments	8		3.9	102	61	125
			9		9.5	104	62	126
		No Analysis						
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
□ FINES CONTENT (%) □
20 40 60 80

Bottom of borehole at 16.4 feet.

BORING NUMBER VC43

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/18/17 **COMPLETED** 5/18/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.02
DRILLING METHOD Vibracore
CORE RECOVERY: 17.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:976721.34 Y:129338.52
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / □ FINES CONTENT (%) □ 20 40 60 80
0		(SP) Gray fine Sand	1	100	1.1	105	64	128	
			2		2	105	63	127	
			3		2	105	63	127	
			4		3	105	64	128	
			5		8.4	103	62	126	
5					12.7				
			6		17	103	62	126	
					16.6				
		(SP) Gray fine to medium Sand with shell fragments	7		15.9	104	63	127	
					18.7				
10		(SP) Gray fine Sand with shell fragments	8		20	102	62	126	
					16.4				
		(SP) Gray fine to medium Sand with shell fragments	9			108	63	127	
		No Analysis							
15									

Bottom of borehole at 17.0 feet.

BORING NUMBER VC46

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.23
DRILLING METHOD Vibracore
CORE RECOVERY: 12.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:980912.67 Y:124964.13
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / □ FINES CONTENT (%) □ 20 40 60 80
0		(SC) Dark olive gray Clayey Sand with large shells, air voids	1	100	1	94	57	121	
		(SP-SC) Dark olive gray Sand with Clay and large shell fragments; air voids	2		1.3	92	54	118	
		(SP) Gray fine to medium Sand with shell fragments	3		1.6	106	65	129	
		(SP) Gray fine to medium Sand	4		2.7	107	64	128	
			5		10.7	104	62	126	
5					13.9				
			6		13.4	105	63	127	
					13.9				
			7		13.6	103	61	125	
					16.4				
10			8		19.1	105	63	127	
					29.8				
		(SP) Dark gray fine to medium Sand	9			111	66	130	
15									

Bottom of borehole at 16.2 feet.

BORING NUMBER VC47

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.74
DRILLING METHOD Vibracore
CORE RECOVERY: 12.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:982399 Y:123622.76
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0	(SP) Black fine to medium Sand with shell fragments	1	100	1	105	63	127
	(SP-SC) Black Sand with Clay and shell fragments	2		1.1	87	52	116
	(SP) Dark gray fine to medium Sand with shell fragments	3		1.4	109	68	132
	(SP) Dark gray medium to fine Sand	4		1.4	103	64	128
5	(CL) Black Sandy Clay with shell, shell fragments, and organics	5		2	49	31	95
				1.4			
	(SP) Gray fine to medium Sand with shell fragments	6		1.1	103	62	126
				6.6			
		7		16	104	62	126
10				10.9			
	(SP) Dark gray fine to medium Sand with shell fragments	8		16.1	110	65	129
	(SP) Dark gray fine to medium Sand with shell fragments and medium Sand partings at 11.9 feet	9		11.1	103	63	127
15							

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.7 feet.

BORING NUMBER VC49

CLIENT Williams **PROJECT NAME** NESE Pipeline

PROJECT NUMBER 1794 **PROJECT LOCATION** Raritian Bay and Lower New York Harbor

DATE STARTED 5/27/17 **COMPLETED** 5/27/17 **WATER DEPTH** Not Recorded **HOLE SIZE** 3.5 inches

DRILLING CONTRACTOR Alpine Ocean Seismic **CORE PENETRATION:** 14.65

DRILLING METHOD Vibracore **CORE RECOVERY:** 12.00

LOGGED BY MK **CHECKED BY** CD **CORE LOCATION:** X:985603.77 Y:122065.18

NOTES **NAD 83, NY Long Island Lambert, ft**

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Light olive brown fine Sand with trace shell fragments; large vertical void 0-0.7 '	1	100	1			
		(SP) Grayish brown fine Sand	2		1	102	61	125
			3		1.2	105	59	123
			4		1.6	101	61	125
			5		3	108	57	121
5					5.8			
			6		6.6	106	58	122
					6.1			
			7		8.4	105	55	119
					8.7			
10		(SP) Gray fine Sand	8		7	104	59	123
			9		10.8	100	60	124

▲ SECONDS PER FOOT ▲ 20 40 60 80

PL MC LL 20 40 60 80

☐ FINES CONTENT (%) ☐ 20 40 60 80

Bottom of borehole at 14.7 feet.

BORING NUMBER VC50

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/4/17 **COMPLETED** 6/4/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.64
DRILLING METHOD Vibracore
CORE RECOVERY: 13.40
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:987393.88 Y:121657.18
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / □ FINES CONTENT (%) □ 20 40 60 80
0		(SP) Grayish brown fine to medium Sand with shell fragments	1	100	0.5	108	65	129	
		(SP) Gray fine Sand	2		5.4	105	65	129	
			3		4.9	103	62	126	
		(SP) Grayish brown fine to medium Sand	4		9.6	103	62	126	
5			5		24.6	110	66	130	
					25				
			6		19.9	110	66	130	
					17.8				
		(SP) Gray fine to medium Sand	7		24.1	105	63	127	
10					42.1				
		(SP) Dark gray fine Sand	8		40.5	105	64	128	
					47.5				
		(SP) Dark gray fine Sand with shell fragments	9			105	63	127	
		No Analysis							

Bottom of borehole at 13.6 feet.

BORING NUMBER VC51

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/4/17 **COMPLETED** 6/4/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 12.06
DRILLING METHOD Vibracore
CORE RECOVERY: 9.51
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:989313.17 Y:121658.87
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Dark gray fine to medium Sand with shell fragments	1	100	1.2	106	63	127
		(SP) Dark gray medium to fine Sand with shell fragments	2		3.3	108	64	128
		(SP) Dark gray fine to medium Sand	3		2.6	107	65	129
		(SP) Dark gray medium to fine Sand	4		8	109	65	129
5		(SP) Very dark gray fine Sand	5		21.1 63.2	109	67	131
		(SP-SM) Very dark gray fine to medium Sand with Silt and shell fragments	6		24.8 32.5	116	71	135
		(SP) Grayish brown fine to medium Sand	7		71.1	110	67	131
10			8		30.3	106	62	126

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 12.1 feet.

BORING NUMBER VC52

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/4/17 **COMPLETED** 6/4/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.52
DRILLING METHOD Vibracore
CORE RECOVERY: 13.50
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:991488.56 Y:122099.78
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Dark gray fine to medium Sand with shell fragments	1	100	2.6	107	65	129
			2		3	105	63	127
		(SP) Dark gray fine Sand with shell fragments	3		3.7	104	63	127
		(SP) Gray fine to medium Sand with shell fragments	4		10.8	102	62	126
		(SP) Gray fine Sand	5		24.6	100	59	123
5					50.3			
			6		36.3	102	62	126
					28.5			
			7		24.1	102	62	126
					20.4			
10			8		11.5	104	63	127
		(SP-SM) Gray fine to medium Sand with black Silty fine Sand seam	9		11.2			
		No Analysis				111	68	132

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 13.5 feet.

BORING NUMBER VC53

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/4/17 **COMPLETED** 6/4/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 10.91
DRILLING METHOD Vibracore
CORE RECOVERY: 11.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:992454.13 Y:122430.14
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Gray and brown fine to medium Sand	1	100	1.5			
		(SP) Grayish brown medium Sand with shell fragments	2		2.3	107	64	128
		(SP) Very dark gray fine to medium Sand with shell fragments	3		4.7	103	63	127
		(SP) Very dark gray fine to medium Sand	4		11.7	103	62	126
		(SP) Very dark gray fine Sand	5		29	105	64	128
5					30.4			
			6		33.2	105	64	128
					31.6			
			7		34.9	102	62	126
					29.3			
10			8		72.3	102	62	126

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 11.0 feet.

BORING NUMBER VC55

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/4/17 **COMPLETED** 6/4/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.33
DRILLING METHOD Vibracore
CORE RECOVERY: 14.30
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:997068.41 Y:124377.4
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Grayish brown medium to fine Sand	1	100	2.1	115	69	133
		(SP) Gray medium Sand	2		3.2	113	68	132
		(SP) Gray medium to fine Sand	3		3.5	111	66	130
			4		5.6	112	68	132
5		(SP) Gray fine to medium Sand	5		9.6	110	66	130
					14.3			
			6		17.3	111	67	131
					25.5			
		(SP) Pale olive medium to fine Sand	7		27.4	118	71	135
					20.6			
10		(SP) Pale olive medium to coarse Sand	8		16.4	124	74	138
		(SP) Pale olive medium to fine Sand	9		14.7	117	68	132
		No Analysis						

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 14.3 feet.

BORING NUMBER VC56

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/27/17 **COMPLETED** 5/27/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 9.27
DRILLING METHOD Vibracore
CORE RECOVERY: 12.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:998806.28 Y:125100.13
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Brown and gray medium to fine Sand with shell fragments	1	100	0.9	93	44	108
		(SP) Light yellowish brown medium Sand	2		0.9	105	60	124
		(SP) Light yellowish brown medium to fine Sand with Gravel	3		2.3	123	71	135
		(SP) Light brownish gray medium to coarse Sand with single 2" Gravel	4		17.8	111	68	132
5		(SP) Light brownish gray fine to medium Sand with single 2.5" Gravel	5		22.5	114	70	134
					9.4			
		(SP) Light brownish gray fine to medium Sand	6		31.6	108	64	128
					25.5			
		(SP) Gray and brownish gray fine to medium Sand	7		26.2	110	66	130
10					184.9			>>
		(SP) Brownish gray fine to coarse Sand	8			115	65	129
		(SP) Brownish gray medium to fine Sand	9			112	67	131

Bottom of borehole at 12.0 feet.

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

BORING NUMBER VC58

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/5/17 **COMPLETED** 6/5/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 12.01
DRILLING METHOD Vibracore
CORE RECOVERY: 13.50
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:1002375.54 Y:126300.99
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Black and olive brown fine to medium Sand with shell fragments	1	100	2.3			
		(SP) Gray medium to coarse Sand with Gravel	2		3.3	113	66	130
		(SP) Dark gray fine to medium Sand	3		4.7	106	64	128
		(SP) Dark gray medium to fine Sand	4		18	106	63	127
		(SP) Dark gray fine to coarse Sand	5		19.7	116	68	132
5					15			
		(SW) Dark gray fine to coarse Sand with Gravel	6		12.2	127	76	140
					13.6			
		(SP) Dark gray medium to fine Sand	7		13.3	115	70	134
					19.2			
10			8		72.5	111	68	132
					83.5			
		(SP) Dark gray fine to medium Sand	9			112	68	132
		No Analysis						

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 13.5 feet.

BORING NUMBER VC59

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/27/17 **COMPLETED** 5/27/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 14.20
DRILLING METHOD Vibracore
CORE RECOVERY: 12.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:1004351.94 Y:127031.76
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(GP) Black coarse Gravel, cemented Sand and shell	1	100	2.2	109	66	130
		(SP) Dark brownish gray fine to coarse Sand with shell, shell fragments, and organics	2					
			3		5.9	106	63	127
		(SP) Dark gray medium to fine Sand with shell fragments						
		(SP) Dark gray fine to medium Sand with shell fragments	4		8.7	111	67	131
			5		13.1	107	65	129
5			6		20.4	106	66	130
					22.7			
		(SP) Dark gray fine to medium Sand	7		23.9	108	66	130
					25.3			
		(SP) Very dark gray medium to fine Sand	8		24.8	117	71	135
					32.7			
10		(SP) Very dark gray medium to coarse Sand with Gravel	9		34.4	130	75	139
					40.3			
		(SP) Dark gray fine to medium Sand	10			109	66	130

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 14.2 feet.

BORING NUMBER VC60

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/5/17 **COMPLETED** 6/5/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 12.52
DRILLING METHOD Vibracore
CORE RECOVERY: 11.80
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:1006143.21 Y:127792.69
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Black and brown fine to medium Sand with shell fragments	1	100	0.2			
		(SW) Black and brown fine to medium Sand with Gravel and ceramic and glass debris	2		2.6	108	62	126
		(SW) Black fine to medium Sand with Gravel and glass debris	3		3.5	98	53	117
		(SP) Grayish brown medium to fine Sand with shell fragments	4		9.6	112	68	132
			5		14.3	116	71	135
5					15.9			
		(SP) Gray fine to medium Sand	6		35.1	110	67	131
					57.1			
			7		52.4	111	68	132
					56.6			
10			8		46.6	109	67	131
					66.7			

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 12.5 feet.

BORING NUMBER VC61

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/5/17 **COMPLETED** 6/5/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.60
DRILLING METHOD Vibracore
CORE RECOVERY: 15.50
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:1007440.11 Y:128281.66
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SW) Very dark gray fine to medium Sand with Gravel	1	100	3	100	57	121
		(SP) Very dark gray fine to medium Sand	2		3	94	53	117
		(SP) Black medium to coarse Sand with Gravel and glass debris	3			95	45	109
		(SW) Black fine to coarse Sand with Gravel, glass and metal debris, and piece of concrete 0.5' long	4		3	99	52	116
		(SP) Black medium Sand with Gravel	5		13.1	107	63	127
5		(SP) Very dark gray fine to medium Sand	6		40.7	107	63	127
					15			
		(SP) Gray fine Sand	7		14	104	63	127
					15.7			
		(SP) Gray fine to medium Sand	8		15.4	108	66	130
10					15.2			
		(SP) Gray medium to fine Sand	9		19.4	110	65	129
		(SP) Dark gray fine to medium Sand	10		17.8	105	63	127
		No Analysis						
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 15.5 feet.

BORING NUMBER VC62

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/5/17 **COMPLETED** 6/5/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.40
DRILLING METHOD Vibracore
CORE RECOVERY: 14.50
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:1009876.68 Y:129212.16
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Very dark gray fine Sand	1	100	2.6	100	60	124
			2		3.9	101	61	125
		(SP) Black fine Sand with shell fragments	3		4.2	102	62	126
		(SP) Very dark gray fine Sand with trace shell fragments	4		8.4	103	63	127
5		(SP) Very dark gray fine to medium Sand with shell fragments	5		17.1	104	62	126
					17.3			
		(SP) Grayish brown medium to fine Sand with shell fragments	6		16.8	110	67	131
					29.5			
		(SP) Gray fine to medium Sand	7		32.3	111	68	132
10					18			
		(SP) Dark gray fine to medium Sand with shell fragments	8		32.8	110	64	128
		(SP) Grayish brown medium to fine Sand	9		27.8	112	68	132
		No Analysis						

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 14.5 feet.

BORING NUMBER VC64

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/5/17 **COMPLETED** 6/5/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.32
DRILLING METHOD Vibracore
CORE RECOVERY: 12.60
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:1013567.2 Y:130638.38
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Black very fine Sand with shell fragments	1	100	2.5	96	57	121
		(SP) Black very fine Sand	2		3.5	97	56	120
		(SP) Black very fine Sand with shell fragments	3		4	101	62	126
		(SP) Dark gray very fine Sand with shell fragments	4		5.1	100	61	125
5		(SP) Very dark gray fine Sand with shell fragments	5		7.3	105	64	128
					9.4			
		(SP) Brown fine to coarse Sand with shell fragments	6		9.8	119	69	133
					12.2			
			7		25	117	71	135
					30.9			
10		(SP) Dark grayish brown fine to medium Sand with shell fragments	8		40	112	67	131
		(SP) Dark grayish brown medium to fine Sand with shell fragments	9		53.6	109	66	130

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 13.3 feet.

BORING NUMBER VC66

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/5/17 **COMPLETED** 6/5/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.25
DRILLING METHOD Vibracore
CORE RECOVERY: 13.40
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:1017706.23 Y:131248.68
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / ☐ FINES CONTENT (%) ☐ 20 40 60 80
0		(SP) Very dark gray fine Sand	1	100	2.9	101	62	126	
		(SP) Very dark gray and dark gray fine Sand with shell fragments	2		3.1	103	64	128	
		(SP) Dark grayish brown medium to fine Sand	3		4	110	67	131	
			4		6.8	112	66	130	
			5		8.4	110	67	131	
5					8.2				
			6		7.7	111	67	131	
					7.7				
		(SP) Dark brownish gray medium to fine Sand	7		9.1	108	65	129	
					17.1				
10		(SP) Dark brownish gray fine to medium Sand with shell fragments	8		19.4	109	66	130	
		(SP) Very dark grayish brown fine to medium Sand with shell fragments	9		19	105	64	128	
		No Analysis							

Bottom of borehole at 13.4 feet.

BORING NUMBER VC67

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/5/17 **COMPLETED** 6/5/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.61
DRILLING METHOD Vibracore
CORE RECOVERY: 14.50
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:1019751.72 Y:130933.32
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP-SM) Very dark gray fine Sand with Silt, shell fragments, and organics	1	100	2.6	96	57	121
		(SP) Very dark gray fine Sand	2		3.7	103	63	127
		(SP) Dark gray fine Sand with shell fragments	3		5.8	106	66	130
			4		9.4	94	53	117
			5		14.5	102	63	127
5					18			
		(SP) Very dark gray fine Sand with shell fragments	6		17.3	102	61	125
					14.7			
		(SP-SM) Very dark gray fine Sand with Silt and shell fragments	7		12.9	100	62	126
10					18.5			
		(SP) Very dark gray fine to medium Sand with shell fragments	8		20.1	105	63	127
		(SP-SM) Very dark gray fine Sand with Silt and shell fragments	9		18.7	104	65	129
		No Analysis						

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 14.5 feet.

BORING NUMBER VC68

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 6/5/17 **COMPLETED** 6/5/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.62
DRILLING METHOD Vibracore
CORE RECOVERY: 14.80
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:1021691.55 Y:130413.2
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Very dark gray fine Sand with shell fragments	1	100	2.4	104	63	127
			2			103	63	127
					3.5			
			3		5.4	103	63	127
		(SP) Dark gray fine Sand with shell fragments	4		9.4	101	61	125
			5		13.3	104	63	127
5					14.5			
		(SP) Very dark gray fine Sand with shell fragments	6		10.5	105	64	128
					8.4			
		(SP) Very dark gray fine to medium Sand with shell fragments	7		13.3	109	66	130
					15.2			
10		(SP-SM) Dark gray fine Sand with Silt and shell fragments	8		17.8	103	63	127
					16.9			
			9			102	63	127
		No Analysis						

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 14.8 feet.

BORING NUMBER VC88

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.19
DRILLING METHOD Vibracore
CORE RECOVERY: 13.90
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:913271.52 Y:110239.53
NOTES Chemical Core
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Dark gray fine to medium Sand with trace Gravel and shell fragments			2.1			
					3			
					2.9			
		(SM) Dark gray Silty Sand with trace shell fragments and odor			1.4			
		(CH) Dark gray fat Clay with trace shell fragments			0.9			
5					0.2			
					1.2			
					1.4			
					1.4			
10					1.6			
		No Analysis						
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 16.2 feet.

BORING NUMBER VC89

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 16.42
DRILLING METHOD Vibracore
CORE RECOVERY: 13.50
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:913441.45 Y:110357.24
NOTES Chemical Core
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / ☐ FINES CONTENT (%) ☐ 20 40 60 80
0		(SM) Dark gray Silty fine Sand with trace shell fragments and Gravel up to 1.2', black lamination from 1.4-1.6'			1				
					1.3				
		(CH) Dark gray fat Clay with trace shell fragments			1.4				
					0.7				
5					0.9				
					0.7				
					0.9				
					0.9				
					0.7				
10					0.9				
		No Analysis							
15									

Bottom of borehole at 16.4 feet.

BORING NUMBER VC90

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 17.97
DRILLING METHOD Vibracore
CORE RECOVERY: 14.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:913628.93 Y:110439.42
NOTES Chemical Core
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SW) Dark gray fine to medium Sand with trace Gravel and shell fragments			1.5			
		(SW) Brown medium Sand with some Gravel			1.8			
		(CL) Dark gray Sandy Clay with trace Gravel and shell fragments, brown lamination at 2.4'			1.9			
		(CH) Dark gray fat Clay with trace shell fragments			0.9			
5					0.9			
					0.9			
					1.2			
					0.9			
					0.9			
10					1.2			
		No Analysis						
15								

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 18.0 feet.

BORING NUMBER VC91

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 11.46
DRILLING METHOD Vibracore
CORE RECOVERY: 10.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:913775.32 Y:110502.77
NOTES Chemical Core
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80
0		(SW) Dark gray fine Sand with Gravel and shells			2				
		(SW) Brown fine to medium Sand with Gravel and laminations of dark gray Sand			2.1				
					2.1				
		(CH) Dark gray fat Clay with trace shells			0.9				
5					1.2				
					1.2				
					1.2				
					0.9				
					1.2				
10					1.2				

PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 11.5 feet.

BORING NUMBER VC92

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/19/17 **COMPLETED** 5/19/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.72
DRILLING METHOD Vibracore
CORE RECOVERY: 12.30
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:913988.35 Y:110601.69
NOTES Chemical Core
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / ☐ FINES CONTENT (%) ☐ 20 40 60 80
0		(SM) Dark gray Silty Sand with trace shell fragments			1				
					1				
					1.1				
					1.2				
5					1.6				
					1.4				
					1.4				
					1.6				
					1.6				
10					1.6				
		No Analysis							

Bottom of borehole at 13.7 feet.

BORING NUMBER VC93_Chemical

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/20/17 **COMPLETED** 5/20/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.43
DRILLING METHOD Vibracore
CORE RECOVERY: 13.00
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:911477.71 Y:110573.91
NOTES Chemical Core
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)	▲ SECONDS PER FOOT ▲ 20 40 60 80 / PL MC LL 20 40 60 80 / ☐ FINES CONTENT (%) ☐ 20 40 60 80
0		(SP) Dark brown fine Sand with trace Gravel			0.2				
					1.5				
					2.1				
		(SP) Light gray fine Sand with lamination of light brown fine Sand with trace shells and Gravel from 4.6-4.8'			1.6				
5					1.9				
		(SP) Light brown fine Sand with lamination of black fine Sand			7				
					13				
					14.6				
					7.2				
10					6				
		No Analysis							

Bottom of borehole at 13.4 feet.

BORING NUMBER VC93_Physical

CLIENT Williams
PROJECT NAME NESE Pipeline
PROJECT NUMBER 1794
PROJECT LOCATION Raritian Bay and Lower New York Harbor
DATE STARTED 5/20/17 **COMPLETED** 5/20/17
WATER DEPTH Not Recorded **HOLE SIZE** 3.5 inches
DRILLING CONTRACTOR Alpine Ocean Seismic
CORE PENETRATION: 13.59
DRILLING METHOD Vibracore
CORE RECOVERY: 11.50
LOGGED BY MK **CHECKED BY** CD
CORE LOCATION: X:911469.61 Y:110567.68
NOTES
NAD 83, NY Long Island Lambert, ft

DEPTH (ft)	GRAPHIC LOG	MATERIAL DESCRIPTION	SAMPLE NUMBER	SAMPLE RECOVERY %	PENETRATION SECONDS PER FOOT	DRY UNIT WT. (pcf)	SUBMERGED UNIT WT. (pcf)	TOTAL UNIT WT. (pcf)
0		(SP) Dark grayish brown and very dark gray fine Sand	1	100	0.2			
		(SP) Dark olive gray fine Sand	2		1	97	55	119
		(SP) Dark olive gray and gray medium to fine Sand	3		1.9	95	51	115
		(SP) Dark grayish brown fine to medium Sand	4		0.9	106	64	128
5		(SM) Light brownish gray Silty fine Sand with dark yellowish brown seams	5		2.6	91	50	114
		(SP-SM) Light brownish gray fine Sand with Silt	6		10.9	91	49	113
		(SM) Light brownish gray Silty fine Sand with Clay seam and dark yellowish brown seams	7		9.1	101	61	125
		(SP) Light brownish gray fine Sand with gray seam with mica	8		6.5	93	54	118
		(SP-SM) Light brownish gray fine Sand with Silt and gray seams with mica	9		5.6	96	55	119
		(SP-SM) Light brownish gray fine Sand with gray Silt seams with mica and dark yellowish brown seams	10		6	97	56	120
10		(SP) Light brownish gray fine Sand with gray seams with mica	11			98	55	119
		No Analysis						

▲ SECONDS PER FOOT ▲
20 40 60 80
PL MC LL
20 40 60 80
☐ FINES CONTENT (%) ☐
20 40 60 80

Bottom of borehole at 13.6 feet.

TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC

NORTHEAST SUPPLY ENHANCEMENT PROJECT

ENVIRONMENTAL REPORT

J-4. ADDENDUM, OCTOBER 2018

NOVEMBER 2026

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Addendum to the Fall/Winter 2016 Offshore Environmental Sampling Report for the Northeast Supply Enhancement Project New Jersey, New York

October 2018

Prepared for:

Transcontinental Gas Pipe Line Company, LLC

Prepared by:

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List of Abbreviations and Acronyms

COC	Chemicals of Concern
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethylene
DDT	dichlorodiphenyltrichloroethane
E & E	Ecology and Environment, Inc.
EPA	U.S. Environmental Protection Agency
ESC	environmental screening criteria
FERC	Federal Energy Regulatory Commission
µg/kg	micrograms per kilogram
mg/L	milligrams per liter
mm	millimeters
NJDEP	New Jersey Department of Environmental Protection
NYSDEC	New York State Department of Environmental Conservation
PAHs	polycyclic aromatic hydrocarbons
PCBs	polychlorinated biphenyls
pg/g	picograms per gram
Project	Northeast Supply Enhancement Project
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan
TOGS	Technical and Operational Guidance Series
Transco	Transcontinental Gas Pipe Line Company, LLC
USACE	U.S. Army Corps of Engineers

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1 Introduction

1.1 Project Description

Transcontinental Gas Pipe Line Company, LLC (Transco), has filed an application with the Federal Energy Regulatory Commission (FERC) for a Certificate of Public Convenience and Necessity (Certificate) for the Northeast Supply Enhancement Project (Project) (Docket No. CP17-101-000). The Offshore Environmental Sampling Report was prepared to facilitate FERC's review and development of an environmental impact statement for the Project and to support the corresponding state-level environmental evaluation of the Project. On June 27, 2017, Transco submitted its Offshore Environmental Sampling Report as Appendix J of Transco's application for a U.S. Army Corps of Engineers (USACE) permit. On June 30, 2017, Transco submitted its Offshore Environmental Sampling Report to FERC. See the Offshore Environmental Sampling Report for a Project Description.

On September 7, 2017, Transco submitted an initial addendum to the Offshore Environmental Sampling Report as Attachment 6 to Volume 1 of Supplemental Information Filing 3 to Transco's application for a FERC Certificate. On October 6, 2017, Transco submitted a second addendum to the Offshore Environmental Sampling Report as Attachment 5 to Volume 1 of Supplemental Information Filing 4 to Transco's application for a FERC Certificate.

On September 15, 2017, Transco submitted an initial addendum to the Offshore Environmental Sampling Report to USACE as Appendix J of Supplemental Information Filing 1 to Transco's Joint Permit application. On December 22, 2017, Transco submitted a second addendum to the Offshore Environmental Sampling Report to USACE as Appendix J of Supplemental Information Filing 2 to Transco's Joint Permit application.

On May 16, 2018, Transco resubmitted its application for a New York State Department of Environmental Conservation (NYSDEC) joint permit, which included Transco's Offshore Environmental Sampling Report and all addenda to date as Appendix C.

1.2 Scope of Work

Transco conducted a supplemental field survey and laboratory analysis beginning in January 2018 to address the geotechnical and chemical characteristics of sediment within the Raritan Bay Channel and Chapel Hill Channel at depths deeper

than what were sampled previously. The survey was conducted to support evaluation of the route for both engineering design and environmental review purposes. Laboratory analysis was completed for a focused list of contaminants, specifically metals, based on historical contamination documented in the area. Transco contracted Ecology and Environment, Inc. (E & E) to support the environmental permitting effort for the Project. The Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP) Version 2.0 developed by E & E and reviewed by regulatory agencies for the previous offshore survey was used for this supplemental field survey. The SAP/QAPP Version 2.0 prepared for the early 2018 offshore survey is provided in Appendix B of this addendum to the Offshore Environmental Sampling Report (note Appendices D, E, and F of this version of the SAP/QAPP are not relevant to the offshore sampling discussed in this report, and are not attached). Agency comments on initial versions of the SAP/QAPP are provided in Appendix D of the original submittal of the Offshore Environmental Sampling Report (June 2017). Transco contracted Alpine Ocean (a Gardline company) to conduct the survey and arrange for sediment testing at onshore laboratories.

Figures A-1 and A-2 show the locations of the sites sampled along the proposed Raritan Bay Loop route during the early 2018 offshore study.

As part of the early 2018 survey campaign, additional sediment samples were collected at numerous sites along the proposed pipeline route for physical and chemical analysis in support of an evaluation of sediment suitability for placement at the Historic Area Remediation Site (HARS). The HARS is an ocean dredged material disposal site located in federal waters approximately 4 miles seaward of Monmouth County, New Jersey. A description of the sampling procedures and the analytical results for the HARS-related study will be presented separately in the Report on the Sampling and Testing of Material from the Northeast Supply Enhancement Project for Dredging and HARS Placement, which is scheduled for submittal to the USACE by November 2018.

1.3 Previous Investigations

See the Offshore Environmental Sampling Report for a discussion of previous investigations.

1.4 Winter 2018 Survey Results

This report presents the results of sample collection and analyses performed to support the sediment chemistry evaluation for the Project. Field data collected as part of the early 2018 survey are included in the appendices. Appendix A contains figures referred to in the body of this report; Appendix B contains the SAP/QAPP Version 2.0; Appendix C contains the tabularized laboratory results for all chemical parameters analyzed for; Appendix D contains the sediment chemistry laboratory reports; and Appendix E contains the Data Usability Summary Reports.

2 Sediment Sampling

The SAP/QAPP Version 2.0 developed to evaluate the site-specific sediment conditions along the proposed pipeline route was designed specifically to address chemicals of concern (COCs) identified by NYSDEC and the NJDEP in regard to offshore dredge and fill activities. These chemicals would be of concern if found above threshold levels. The threshold values utilized reflect levels above which there could be harmful effects on aquatic life.

2.1 Thresholds for Chemicals of Concern

As introduced in the Offshore Environmental Sampling Report (June 2017), thresholds for COCs in sediment samples collected in New York State waters were taken from the Technical and Operational Guidance Series (TOGS) 5.1.9 document, *In-Water and Riparian Management of Sediment and Dredged Material* (NYSDEC 2004) and the *Screening and Assessment of Contaminated Sediment* (NYSDEC 2014). Thresholds for COCs in sediment samples collected in New Jersey waters were taken from *The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters* (NJDEP 1997). Additional guidance on COCs in samples collected within the offshore Project area was taken from the USACE/U.S. Environmental Protection Agency's (EPA) *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal* (2016). These agency documents were developed to provide guidance on the statutory and regulatory requirements for dredging activities and to promote uniformity in the certification and/or permitting of dredging projects.

For further discussion of thresholds used, see the Offshore Environmental Sampling Report.

2.2 Sampling Methods

Sediment was sampled by core boring and vibracoring at a total of 19 sites, all in New York waters. The sampling depths at each site were designed to account for sediment within the Raritan Bay Channel and Chapel Hill Channel at depths deeper than what were sampled previously. This was done in response to discussions with USACE regarding the potential deeper burial requirement at these channels.

Deep core samples (core boring) extending up to 125 feet below the seafloor were collected from 11 sampling sites. Deep core samples were analyzed for geotechnical properties only; the results will be used in the engineering design of the

channel crossings and are not further discussed in this report. Deep core sample sites are identified as "B" in Table 2-1. All deep core sampling sites were reviewed and approved by the New York State Historic Preservation Office, as appropriate, prior to the sampling.

Table 2-1 Sediment Sampling Sites in New York State Waters

Sample Site [a,b]	Latitude	Longitude	Coring Depth Range (feet)	Sample Site [a,b]	Latitude	Longitude	Coring Depth Range (feet)
B-20	40.503400	-74.176250	0 - 87.0	VC15	40.503286	-74.176237	15.0 - 25.4
B-21	40.504109	-74.174607	0 - 87.0	VC16	40.504057	-74.174471	15.0 - 27.9
B-22	40.504819	-74.172964	0 - 72.0	VC17	40.505196	-74.171867	15.0 - 24.1
B-23	40.505528	-74.171320	0 - 87.0	VC18	40.505968	-74.170079	15.0 - 30.2
B-24	40.506237	-74.169679	0 - 87.0	VC38	40.526834	-74.043371	15.0 - 26.1
B-25	40.527385	-74.044643	0 - 82.0	VC39	40.526449	-74.041158	12.0 - 21.2
B-26	40.527015	-74.042666	0 - 72.0	VC40	40.526125	-74.039373	15.0 - 22.8
B-27	40.526643	-74.040677	0 - 77.0	VC41-ALT	40.525453	-74.036149	12.0 - 19.4
B-28	40.526271	-74.038690	0 - 77.0				
B-29	40.525899	-74.036699	0 - 82.0				
B-30	40.525503	-74.034715	0 - 87.0				
B-32	40.503335	-74.175054	0 - 112.0				
B-33	40.505172	-74.172038	0 - 82.0				

Reference datum: NAD 1983.

Notes:
(a) Sampling sites labeled "B" indicate where boring sampling occurred. Sampling sites labeled "VC" indicate where vibracore sampling occurred.
(b) Sampling sites labeled "-ALT" indicate shifts that occurred based on a Phase I archaeological survey.

Shallow core samples (vibracores) were collected at eight sites. The shallow core sampling sites are identified as "VC" in Table 2-1 and were reviewed and approved by the New York State Historic Preservation Office, as appropriate, prior to the sampling. Sites identified with "-ALT" indicate sampling sites that were shifted from the original designated site to avoid potential cultural resources. Shallow core samples were collected for testing and analysis of bulk sediment chemistry properties.

Once retrieved, the sediment core soil types were classified according to the United Soil Classification System, and sediment samples were collected from the core and shipped to a laboratory for chemical analysis. Each shallow core was separated into 3-foot-long sections. The sections were examined to identify any distinct layers of contamination more than 1 foot thick based on the field geologist's evaluation of appearance and/or odor, but no such layers were identified. The minimum 1-foot thickness provided for sufficient sample volume for all sediment chemistry analyses. A total of 29 samples were collected, as identified in Appendix B. A complete list of chemical analyses performed and the laboratory

standard operating procedures are provided in the SAP/QAPP Version 2.0 (included as Appendix B to this application). The list of chemical analyses performed is based upon the NYSDEC TOGS 5.1.9 and the NJDEP Site Remediation Program Effects Range Low and Effects Range Median ecological screening criteria. A summary of the results are discussed below, and the complete analytical results are provided in Appendices C and D.

Quality assurance/quality control (QA/QC) samples were collected in accordance with the SAP/QAPP (Appendix B). Analytical data have been validated for samples collected as part of this sampling effort. Field QC samples included one sediment duplicate per matrix per 20 samples for each analysis. Field equipment blanks were used at a rate of one per equipment set per day. Equipment blanks were only used on equipment sets that were subject to decontamination. Dedicated or disposable equipment did not require the use of equipment blanks.

The results of the bulk chemistry analysis of vibracore samples are presented below.

2.3 Sediment Chemistry Results

In the following discussion, sampling sites within the offshore study area are organized into two geographic groups: the Raritan Bay Channel and the Chapel Hill Channel (see Table 2-2). Since all samples collected were entirely within New York State waters, the following discussion compares analytical results to the NYSDEC thresholds only.

Table 2-2 Geographic Sediment Sample Groups

Group ID	Included Samples	State
Raritan Bay Channel	VC15 to VC18	NY
Chapel Hill Channel	VC38 to VC42-ALT	NY

The methods used to calculate the concentrations of the following analytes discussed below are presented in the SAP/QAPP (see Appendix B): sum of DDD (dichlorodiphenyldichloroethane) + DDE (dichlorodiphenyldichloroethylene) + DDT (dichlorodiphenyltrichloroethane); total PCB (polycholrinated biphenyl) Aroclors; total PAHs (polycyclic aromatic hydrocarbons); and the total toxicity equivalency factor for dioxins and furans.

2.3.1 Raritan Bay Channel

Metals

Each sediment sample was analyzed for 27 metals, including mercury (see Appendix C for a complete list of these metals). Each of the 27 metals was detected in at least one sample collected in early 2018 (see Table 2-3 for the sampling sites where metals were detected and Table 2-4 for concentration ranges). Class A

and/or Class C thresholds for nine of these metals are available, and threshold exceedances occurred for all nine metals. The concentrations of seven of the detected metals exceeded Class A thresholds, and the concentrations of two of the metals exceeded Class C thresholds. The metals detected within this segment that exceeded Class A and/or Class C thresholds are identified in Table 2-5.

Table 2-3 Number of Detections at Sample Sites

Sampling Site[a]	PAHs	Dioxins/ Furans	Pesticides	PCB Aroclors	Metals
VC15	13	--	0	0	24
VC16	17	9	8	0	27
VC17	10	--	0	0	27
VC18	14	--	0	0	24
VC38	0	--	0	0	23
VC39	0	--	0	0	23
VC40	2	--	0	0	24
VC41-ALT	0	--	0	0	19

Note:
[a] Dioxins and furans were analyzed only for sampling site VC16.

Table 2-4 Metals Detected in Raritan Bay Channel Sampling Sites

Metals	Range (mg/kg)	Metals	Range (mg/kg)
Aluminum	13,600-16,900	Mercury	0.004-1.93
Antimony	0.267-1.46	Molybdenum	1.12-1.87
Arsenic	10.1-23.0	Nickel	25.3-35.2
Barium	26.2-64.8	Potassium	3,570-4,340
Beryllium	0.804-1.03	Selenium	2.39-3.99
Cadmium	0.099-1.67	Silver	0.096-3.69
Calcium	5040-9240	Sodium	3,640-10,300
Chromium	31.9-111	Strontium	35.2-59.1
Cobalt	9.47-11.6	Thallium	0.124-0.688
Copper	11.9-115	Tin	1.06-14.9
Iron	32,700-39,800	Titanium	224-396
Lead	12.2-117	Vanadium	35.3-52.7
Magnesium	8,460-10,100	Zinc	71.4-230
Manganese	376-561		

Table 2-5 Sampling Sites in Raritan Bay Channel and Samples Exceeding Class A and/or Class C Thresholds for Metals

Analyte	Concentrations that Exceeded Thresholds		Sampling Site	Sample Depth(s)[a] (feet)
	Exceeded Class A (mg/kg)	Exceeded Class C (mg/kg)		
Arsenic	10.1-23	N/A	VC15	15-18, 18-21, 21-24, 24-25.1
			VC16	15-18, 18-21, 21-24, 24-27.9
			VC17	15-18, 18-21, 21-24.1
			VC18	15-18, 18-21, 21-24, 24-27, 27-29.3
Cadmium	1.67	N/A	VC16	15-18
Chromium	111	N/A	VC16	15-18
Copper	115	N/A	VC16	15-18
Lead	117	N/A	VC16	15-18
Mercury	0.219 - 0.275	1.93	VC16	***15-18,*** 18-21
Nickel	25.3-35.2	N/A	VC15	15-18, 18-21, 21-24, 24-25.1
			VC16	15-18, 18-21, 21-24, 24-27.9
			VC17	15-18, 18-21, 21-24.1
			VC18	15-18, 18-21, 21-24, 24-27, 27-29.3
Silver	N/A	3.69	VC16	***15-18***
Zinc	230	N/A	VC16	15-18

Note:
[a] Sample depths where Class C thresholds were exceeded are in bold and italics.

Key:
N/A = Detected concentrations did not exceed the threshold.

Pesticides

Each sediment sample was analyzed for 27 pesticides (see Appendix C for a complete list of these pesticides). The sum of DDT + DDE + DDD was also calculated for each sediment sample.

Table 2-6 Pesticides Detected in Raritan Bay Channel Sampling Sites

Pesticide	Range (µg/kg)
O,P'-DDD	7.98
O,P'-DDE	4.99
P,P'-DDD	0.499-2.07
P,P'-DDE	6.47
P,P'-DDT	0.742
cis-Chlordane	0.424
Dieldrin	1.03
Endrin aldehyde	2.54

Key:
µg/kg = micrograms per kilogram

Eight of the 27 pesticides analyzed for were detected in at least one sample collected along the proposed route (see Table 2-6). Of these eight pesticides, only dieldrin has an available Class A and Class C threshold, and there were no exceedances of the Class A threshold for dieldrin. The sum of DDT + DDE + DDD also has a Class A and Class C threshold, and there were no exceedances of the Class A threshold for the sum of DDT + DDE + DDE.

Polychlorinated Biphenyls (PCBs)

Each sediment sample was analyzed for PCBs expressed as seven Aroclors. The sum of PCB Aroclors was also calculated. A complete list of the Aroclors included in the analysis is provided in Appendix C.

None of the seven Aroclors analyzed for were detected in samples in the proposed route. Therefore, the Class A threshold for the sum of Aroclor compounds was not exceeded.

Polycyclic Aromatic Hydrocarbons (PAHs)

Each sediment sample was analyzed for 18 PAHs. All 18 PAHs were detected in at least one sample collected along the proposed route (see Table 2-3 for the sampling sites where PAHs were detected and Table 2-7 for concentration ranges). The sum of PAHs, the sum of low-molecular weight PAHs, and the sum of high-molecular weight PAHs were also calculated for each sediment sample. Class A and Class C thresholds are available only for total PAHs. Total PAHs did not exceed the Class A threshold in any of the samples.

Table 2-7 PAHs Detected in Raritan Bay Channel Sampling Sites

PAH	Range (µg/kg)	PAH	Range (µg/kg)
2-Chloronaphthalene	0.946-0.955	Benzo(k)fluoranthene	0.729-137
2-Methylnaphthalene	0.873-44.0	Chrysene	0.851-195
Acenaphthene	0.803-13.8	Dibenz(a,h)anthracene	2.54-37
Acenaphthylene	0.627-34.9	Fluoranthene	1.92-287
Anthracene	1.03-66.5	Fluorene	1.48-31.1
Benz(a)anthracene	21.1-192	Indeno(1,2,3-cd)pyrene	2.05-134
Benzo(a)pyrene	24.6-233	Naphthalene	7.05-69.4
Benzo(b)fluoranthene	1.23-187	Phenanthrene	2.34-130
Benzo(g,h,i)perylene	1.03-149	Pyrene	1.06-275

Key:
µg/kg = micrograms per kilogram

Dioxins/Furans

Based on the analytical results for samples collected in late 2016 and feedback from NYSDEC staff, only sediment samples from sampling site VC16 were analyzed for dioxins and furans. Each sediment sample from VC16 was analyzed for 17 dioxins and furans, and the total toxicity equivalency factor was calculated. Of

the dioxins and furans analyzed for, only one dioxin (2,3,7,8-tetrachlorodibenzo-p-dioxin) and the total toxicity equivalence factor of all dioxins and furans have Class A and/or Class C thresholds.

Nine of the 17 dioxins and furans analyzed for were detected in at least one sample (see Table 2-3 for the sampling sites where dioxins and furans were detected and Table 2-8 for concentration ranges).

2,3,7,8-Tetrachlorodibenzo-p-dioxin exceeded the Class A threshold (there is no Class C threshold available) in the 15- to 18-foot sample at VC16 (see Table 2-9). The total toxicity equivalency factor exceeded the Class A threshold in the 15- to 18-foot sample at VC16.

Table 2-8 Dioxins/Furans Detected in Raritan Bay Channel Sampling Sites

Dioxin/Furan	Range[a] (pg/g)	Dioxin/Furan	Range[a] (pg/g)
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	75.8	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	20.9
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin	113-1,670	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	11.9
1,2,3,4,6,7,8-Heptachlorodibenzofuran	44.6	2,3,7,8-Tetrachlorodibenzofuran	6.79
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	17.8-132	2,3,7,8-Tetrachlorodibenzo-p-dioxin	5.58
1,2,3,4,7,8-Hexachlorodibenzofuran	6.46		

Note:
[a] Individual concentrations of dioxins and furans are provided if an analyte was only detected in one sample.

Key:
pg/g = picograms per gram

Table 2-9 Sampling Sites and Samples Exceeding Class A and/or Class C Thresholds for Dioxins and Furans in Raritan Bay Channel Sampling Sites

Analyte	Concentrations that Exceeded Thresholds		Sampling Site	Sample Depth(s)[a] (feet)
	Exceeded Class A (pg/g)	Exceeded Class C (pg/g)		
2,3,7,8-Tetrachlorodibenzo-p-dioxin	5.58	--	VC16	15-18
Total Toxicity Equivalency Factor	16.1	N/A	VC16	15-18

Key:
-- = Threshold was not available.
N/A = Detected concentrations did not exceed the threshold.
pg/g = picograms per gram.

2.3.2 Chapel Hill Channel

Metals

Each sediment sample was analyzed for 27 metals, including mercury (see Appendix C for a complete list of these metals). Each of the 27 metals was detected in at least one sample collected in early 2018 (see Table 2-3 for the sample sites where metals were detected and Table 2-10 for concentration ranges). Class A and/or Class C thresholds for nine of these metals are available. Class A thresholds for two of the metals were exceeded, but no Class C thresholds for metals were exceeded. The metals detected within this segment that exceeded Class A thresholds are presented in Table 2-11.

Table 2-10 Metals Detected in Chapel Hill Channel Sampling Sites

Metals	Range (mg/kg)	Metals	Range (mg/kg)
Aluminum	1,900-6,050	Mercury	0.003
Antimony	0.425	Molybdenum	0.132-0.689
Arsenic	1.9-8.35	Nickel	4.96-22.0
Barium	3.45-45.0	Potassium	276-1,110
Beryllium	0.124-0.375	Selenium	1.06-1.42
Cadmium	0.035-0.042	Silver	0.057
Calcium	154-1620	Sodium	795-3,250
Chromium	5.26-16.3	Strontium	2.74-9.78
Cobalt	1.85-13.5	Thallium	0.103
Copper	1.88-8.44	Tin	0.273-0.864
Iron	4,860-20,300	Titanium	69.9-124
Lead	1.32-6.6	Vanadium	6.98-22.8
Magnesium	721-2,020	Zinc	11.8-27.6
Manganese	44.9-160		

Key:
mg/kg = milligrams per kilogram

Table 2-11 Sampling Sites and Samples Exceeding Class A and/or Class C Thresholds for Metals in Chapel Hill Channel

Analyte	Concentrations that Exceeded Thresholds		Sampling Site	Sampling Depth(s)[a] (feet)
	Exceeded Class A (mg/kg)	Exceeded Class C (mg/kg)		
Arsenic	8.35	N/A	VC38	18-21
Nickel	22	N/A	VC38	21-24

Key:
mg/kg = milligrams per kilogram
N/A = Detected concentrations did not exceed the threshold.

Pesticides

Each sediment sample was analyzed for 27 pesticides (see Appendix C for a complete list of these pesticides). The sum of DDT + DDE + DDD was also calculated for each sediment sample.

None of the 27 pesticides analyzed were detected in any of the samples collected in early 2018. Therefore, the Class A threshold for the sum of DDT + DDE + DDD was not exceeded.

Polychlorinated Biphenyls (PCBs)

Each sediment sample was analyzed for PCBs expressed as seven Aroclors. The sum of PCB Aroclors was also calculated. A complete list of the Aroclor compounds included in the analysis is provided in Appendix C.

None of the seven Aroclor compounds analyzed were detected in samples collected in the proposed route. Therefore, the Class A threshold for the sum of Aroclors was not exceeded.

Polycyclic Aromatic Hydrocarbons (PAHs)

Each sediment sample was analyzed for 18 PAH compounds. Two PAHs were detected in at least one sample collected along the proposed route (see Table 2-12 for ranges and Appendix C for the sampling sites where PAHs were detected). The sum of PAHs, the sum of low-molecular weight PAHs, and the sum of high-molecular weight PAHs were also calculated for each sediment sample. Class A and Class C thresholds are available only for total PAHs. Total PAHs did not exceed the Class A threshold in any of the samples.

Table 2-12 PAHs Detected in Chapel Hill Channel Sampling Sites

PAH	Range (µg/kg)
Benzo(g,h,i)perylene	0.724-0.833
Dibenz(a,h)anthracene	1.24-1.61

Key:
µg/kg = micrograms per kilogram

Dioxins/Furans

Based on the analytical results for samples collected in late 2016 and feedback from NYSDEC staff, none of the sediment samples from Chapel Hill Channel were analyzed for dioxins and furans.

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3 References

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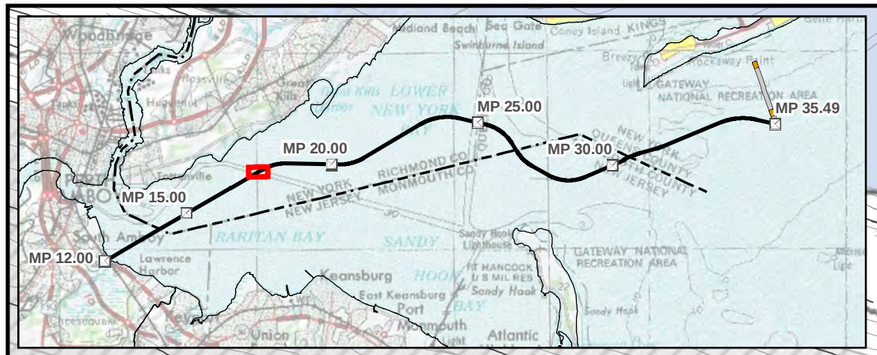
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A Figures

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16

17

22

26

28

13

17

G

C "25"

RARITAN BAY WEST REACH

MP 17.90

MP 17.80

MP 17.70

MP 17.60

MP 17.50

MP 17.40

VC-18.5T

BHA-24

VC18

BHA-23

VC17

BHA-22

VC-16.5T

BHA-21

VC16

BHA-20

VC15

-20

-25

-30

-40

-45

-35

Latitude/Longitude Coordinates - GCS North American Datum 1983

Existing Sample Site
New Sample Site (Vibracore)
New Sample Site (Borehole)

100-ft Buffer
75-ft Buffer
Maintained Navigation Channel

Milepost for Proposed Pipeline
Proposed Raritan Bay Loop
Bathymetric Contour (5-ft interval)
Bathymetric Contour (1-ft interval)

0 100 200 300 Feet

0 50 100 Meters

Bathymetry from Rogers Surveying, PLLC. 2016

NOTES

NOTE: Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigation charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

SITE LOCATION

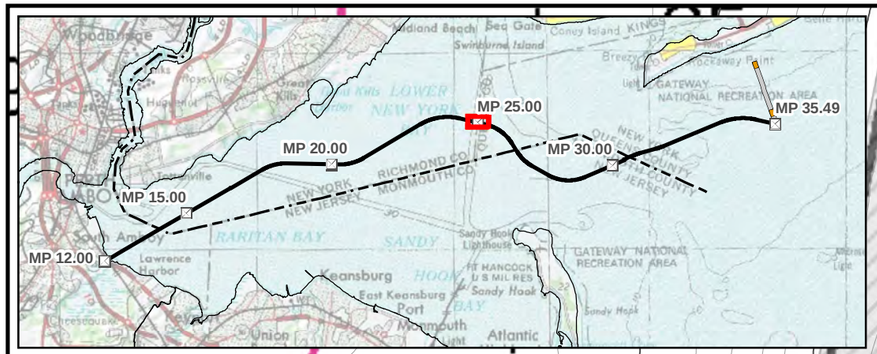


FIGURE A-1
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
SUPPLEMENTAL SAMPLING SITES AT RARITAN CHANNEL
RARITAN BAY LOOP
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

Revision Date: 10/1/2018

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MP 24.70
BHA-25
VC-37.5T
MP 24.80
VC38
BHA-26
MP 24.90
BHA-27
VC39
MP 25.00
VC40-ALT
BHA-28
MP 25.10
BHA-29
MP 25.20
VC41-ALT
BHA-30
VC-41.5T
MP 25.30
28 Obstns
28 Obstns
28
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24 Rks Obstn

Latitude/Longitude Coordinates - GCS North American Datum 1983

- Existing Sample Site
- New Sample Site (Vibracore)
- New Sample Site (Borehole)
- 100-ft Buffer
- 75-ft Buffer
- Maintained Navigation Channel
- Milepost for Proposed Pipeline
- Proposed Raritan Bay Loop
- Bathymetric Contour (5-ft interval)
- Bathymetric Contour (1-ft interval)

0 100 200 300 Feet

0 50 100 Meters

Bathymetry from Rogers Surveying, PLLC. 2016

NOTES

NOTE: Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigation charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

SITE LOCATION



FIGURE A-2
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
SUPPLEMENTAL SAMPLING SITES AT CHAPEL HILL CHANNEL
RARITAN BAY LOOP
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Revision Date: 10/1/2018

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B SAP/QAPP Version 2.0

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TRANSCONTINENTAL GAS PIPE LINE COMPANY, LLC

OFFSHORE SAMPLING AND ANALYSIS PLAN / QUALITY ASSURANCE PROJECT PLAN

VERSION 2.0

NORTHEAST SUPPLY ENHANCEMENT PROJECT

JANUARY 2018

PUBLIC

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Cross Reference Table

NESE SAP/QAPP – (Version 1.2) June 2017		NESE SAP/QAPP – (Version 2.0) January 2018	
Section	**Sub Section**	**Action**	**Notes**
1.0 Project Management	--	Updated	Sampling for evaluation for HARS disposal added
1.1 Project Organization	--	Updated	Sampling for evaluation for HARS disposal added, benthic sampling removed
1.2 Project Description/Background (Problem Definition)	--	Updated	Sampling for evaluation for HARS disposal added, benthic sampling removed
	1.2.1 Environmental Setting	No change	--
	1.2.2 Sediment Characteristics and Potential Contaminants of Concern	Updated	Results from Project-related 2016/2017 sediment sampling added
	1.2.3 Benthic Community	Not included	No benthic community sampling planned
1.3 Sampling and Analysis Task Description	--	Updated	Sampling for evaluation for HARS disposal added, benthic sampling removed
1.4 Quality Goals and Objectives	--	Updated	Sampling for evaluation for HARS disposal added, benthic sampling removed
	1.4.1 Precision	No change	--
	1.4.2 Accuracy	No change	--
	1.4.3 Representativeness	No change	--
	1.4.4 Completeness	No change	--
	1.4.5 Comparability	No change	--
	1.4.6 Sensitivity	No change	--
1.5 Special Training/Certification	--	Updated	NJ requirements for deep core boring removed
1.6 Documentation and Records	--	Updated	Sampling for evaluation for HARS disposal added, benthic sampling removed
	1.6.1 Field Documentation and Reporting	Updated	Sampling for evaluation for HARS disposal added
	1.6.2 Laboratory Data Reporting	Updated	Sampling for evaluation for HARS disposal added, benthic sampling removed
	1.6.3 Summary Reports	Updated	Sampling for evaluation for HARS disposal added, benthic sampling removed
	1.6.4 Record Retention	No change	--

Cross Reference Table

NESE SAP/QAPP – (Version 1.2) June 2017		NESE SAP/QAPP – (Version 2.0) January 2018	
Section	**Sub Section**	**Action**	**Notes**
2.1 Sampling Process Design	--	Updated	Sampling for evaluation for HARS disposal added, benthic sampling removed
2.2 Sampling Methods	--	No change	--
	2.2.1 Core Boring	No change	--
	2.2.2 Vibracoring	No change	--
	2.2.3 Sediment Grabs	Updated	Benthic sampling removed, reference site sediment grab samples for evaluation for HARS disposal added
	2.2.4 Water Sampling	New	Water sampling for evaluation for HARS disposal added
	2.2.5 Contingency Resampling Procedures	Updated	Sampling for evaluation for HARS disposal added
	2.2.6 Equipment Decontamination	Updated	Reference site sediment grab and water sampling added for evaluation for HARS disposal
	2.2.7 Investigation-Derived Waste	No change	--
2.3 Sample Handling and Custody	--	No change	--
	2.3.1 Sample Containers, Preservation, and Holding Times	Updated	Benthic sampling removed, sampling for evaluation for HARS disposal
	2.3.2 Sample Handling	Updated	Benthic sampling removed, sampling for evaluation for HARS disposal added
	2.3.3 Sample Identification	Updated	Benthic sampling removed, sampling for evaluation for HARS disposal added
	2.3.4 Sample Custody	No change	--
2.4 Analytical Method Requirements	--	Updated	Benthic sampling removed, sampling for evaluation for HARS disposal added
2.5 Quality Control	--	Updated	Section sub-headings added
	2.5.1 Geotechnical	New	Separated into sub-heading, no field QC

Cross Reference Table

NESE SAP/QAPP – (Version 1.2) June 2017		NESE SAP/QAPP – (Version 2.0) January 2018	
Section	**Sub Section**	**Action**	**Notes**
	2.5.2 Bulk Sediment Chemistry	New	Separated into sub-heading, no trip blanks
	2.5.3 Evaluation for HARS Disposal	New	QC for HARS disposal added
2.6 Instrument/Equipment Testing, Inspection, and Maintenance	--	No change	--
	2.6.1 Field Equipment Testing and Maintenance	No change	--
	2.6.2 Analysis Instrument Maintenance	No change	--
2.7 Instrument/Equipment Calibration and Frequency	--	No change	--
2.8 Inspection/Acceptance of Supplies and Consumables	--	No change	--
2.9 Non-Direct Measurements	--	No change	--
2.10 Data Management	--	Updated	Benthic sampling removed
3.1 Assessment and Response Actions	--	Updated	Benthic sampling removed
	3.1.1 Peer Review	No change	--
	3.1.2 Technical Systems Assessments	Updated	Benthic sampling removed
	3.1.3 Corrective Action	No change	--
3.2 Reports to Management	--	No change	--
4.1 Data Review, Validation, and Verification Requirements	--	No change	--
4.2 Validation and Verification Methods	--	Updated	Benthic sampling removed
4.3 Reconciliation with User Requirements	--	Updated	Benthic sampling removed
5.0 References	--	Updated	--
Appendix A	--	Updated	New figures added for new sample site locations and for sampling for evaluation for HARS disposal. Previous figures removed.

Cross Reference Table

NESE SAP/QAPP – (Version 1.2) June 2017		NESE SAP/QAPP – (Version 2.0) January 2018	
Section	**Sub Section**	**Action**	**Notes**
Appendix B	--	Updated	Previous sample coordinates removed, table of coordinates added for sampling for samples proposed for 2018
Appendix C	--	Updated	Test table for New Jersey sample sites removed, test table for New York sample sites updated, test table for evaluation for evaluation for HARS disposal added
Appendix D	--	Updated	SOPs for laboratories conducting evaluation for HARS disposal added
Appendix E	--	New	Checklists of USACE NYD DMMS reporting requirements for biological and chemical data for evaluation for HARS disposal
Appendix F	--	New	USACE NYD DMMS and EPA Region 2 Sampling and Testing Requirements Plan

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LIST OF ACRONYMS

°C	degrees Celsius
ASTM	American Society for Testing and Materials
Ag	Silver
As	Arsenic
Cd	Cadmium
CFR	Code of Federal Regulations
COC	chain-of-custody
CPT	cone penetrometer test
Cr	Chromium
Cu	Copper
DI	deionized
DMMS	Dredge Material Management Section
E & E	Ecology and Environment, Inc.
ELAP	Environmental Laboratory Accreditation Program
EPA	U.S. Environmental Protection Agency
HARS	Historic Area Remediation Site
HDD	horizontal directional drill
Hg	Mercury
LCS	laboratory control sample
MP	milepost
MS/MSD	matrix spike/matrix spike duplicate
Ni	Nickel
NJDEP	New Jersey Department of Environmental Protection
NYD	New York District
NYSDEC	New York State Department of Environmental Conservation
PAH	polycyclic aromatic hydrocarbon
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
Pb	Lead
PCB	polychlorinated biphenyl
Project	Northeast Supply Enhancement Project
QA/QC	quality assurance/quality control
RPD	relative percent difference
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan

SOP	standard operating procedure
SPP	suspended particulate phase
TOC	total organic carbon
Transco	Transcontinental Gas Pipe Line Company, LLC
USACE	U.S. Army Corps of Engineers
USGS	U.S. Geological Survey
Zn	Zinc

1 PROJECT MANAGEMENT

This combined Offshore Sampling and Analysis Plan and Quality Assurance Project Plan (SAP/QAPP) has been prepared by Ecology and Environment, Inc. (E & E) for Transcontinental Gas Pipe Line Company, LLC (Transco) in support of the Northeast Supply Enhancement Project (Project). This SAP/QAPP is applicable to any offshore sediment or water sampling activities and associated field or laboratory analyses that generate data that may be submitted to state or federal agencies to support the regulatory evaluation of the Project. This SAP/QAPP does not apply to offshore hydrographic (remote-sensing) survey activities. This SAP/QAPP is also applicable to any offshore sediment or surface water sampling activities and associated field or laboratory analyses that generate data that may be submitted to the U.S. Army Corps of Engineers (USACE) New York District (NYD) Dredge Material Management Section (DMMS) and the U.S. Environmental Protection Agency (EPA) Region 2 for evaluation of dredged sediments for potential disposal at the Historic Area Remediation Site (HARS). The Cross Reference Table (starting on page iii) identifies sections of this SAP/QAPP that are new or have been updated from the previous version. Version 1.2 of the SAP/QAPP was submitted as part of Transco's application for a USACE NYD permit (USACE Pre-application File No. NAN-2016-00515-EHA, June 27, 2017). Methods that differ between samples collected for geotechnical analysis, core samples collected for bulk sediment chemistry analysis, and core samples collected for the evaluation for HARS disposal are identified with subheadings in Sections 1.3, 2.1, 2.3, 2.4, and 2.5.

This SAP/QAPP presents the policies, organization, objectives, functional activities, and specific quality assurance/quality control (QA/QC) procedures that will be used by Transco to help ensure that all offshore technical sampling data generated are accurate, representative, and ultimately capable of withstanding judicial scrutiny. The SAP/QAPP has been prepared in accordance with the EPA's *Requirements for Quality Assurance Project Plans*, EPA QA/R-5 (March 2001) and *Guidance for Quality Assurance Project Plans*, EPA QA/G-5 (December 2002) (EPA 2001, 2002a). This SAP/QAPP is formatted to address the four major sections listed in the 2001 EPA guidance document: Project Management, Data Generation and Acquisition, Assessment and Oversight, and Data Validation and Usability.

1.1 Project Organization

In general, the sampling and field analyses will be conducted by the sampling contractor, Alpine Ocean Seismic Survey, Inc. (Alpine) and overseen by Transco representatives, likely including staff from Transco's consultants – E & E and/or INTECSEA (a division of the Worley

Parsons Group). Geotechnical samples will be collected in the field and transported to the NGI laboratory by the sampling contractor (Alpine) or Transco's representatives. Sediment samples to be analyzed for bulk chemistry will be processed on the sampling vessel and transferred to an Alpha Analytical laboratory representative by the sampling contractor (Alpine). Sediment samples for HARS evaluation will be processed on the sampling vessel and transferred to an EnviroSystems, Inc. (ESI) laboratory representative by the sampling contractor (Alpine). All field collected information will be delivered to Transco and may simultaneously be shared with INTECSEA, Lake Superior Consulting, and/or E & E. It is anticipated that geotechnical data and information from the sampling contractor and geotechnical laboratory will be delivered simultaneously to Transco and INTECSEA (and/or Lake Superior Consulting). All laboratory data for bulk sediment chemistry and data generated for HARS evaluation will be delivered to E & E for review prior to transmittal to Transco. It is expected that INTECSEA (and/or Lake Superior Consulting) will review and process the geotechnical data and laboratory report(s), then provide a Geotechnical Site Investigation Report to Transco for review and approval prior to final application for Project design and construction purposes. It is also expected that E & E will review and process the chemical laboratory reports and then prepare an Offshore Environmental Sampling Report Addendum that will be provided to Transco for review and approval prior to submittal to regulatory agencies. The testing results collected for evaluation of HARS disposal will be compiled by ESI and provided to E & E and Transco for review prior to submittal to the USACE NYD DMMS. Any relevant data obtained during the HARS evaluation may also be included in the Offshore Environmental Sampling Report Addendum. Table 1.1-1 identifies points-of-contact and their responsibilities with respect to the offshore sampling and analysis activities.

Table 1.1-1
Key Offshore Sampling and Analysis Personnel

Name	Role	Responsibilities
Transcontinental Gas Pipe Line Company, LLC		
Chris Martinez	Offshore Project Engineering Lead	Overall authority for application of geotechnical data and reports to offshore pipeline design and construction.
Webb Winston, P.E.	Offshore Engineer	Final QA review and approval of geotechnical data and reports prior to application for offshore pipeline design for the Project.
Scott Horner	Offshore Environmental Manager	Final QA review and approval of environmental reports (including chemical analyses) prior to submittal to regulatory agencies.

Table 1.1-1
Key Offshore Sampling and Analysis Personnel

Name	Role	Responsibilities
Ecology and Environment, Inc.		
Sara Mochrie	Consulting Environmental Project Manager	Overall responsibility for chemical/HARS sampling and analysis activities.
Dave Albers, P.E.	Lead Environmental Engineer	Provides QA review of environmental reports.
Marcia Galloway, CQA, CQM	Quality Manager	Overall QA oversight of chemical and HARS sampling and analysis.
Steven MacLeod	Offshore Technical Lead	Responsible for developing the environmental sampling and analysis report.
Nischint Sundar	System Controls Lead	Responsible for managing chemical sediment survey data and report files.
Maxwell Martin, P.E.	HARS Disposal Task Engineer	Responsible for coordination with USACE NYD Dredge Material Management Section and HARS Manager and oversight of HARS-related sampling.
Lynne Parker	Lead Project Chemist	Responsible for QA review of chemical analysis data.
INTECSEA / Worley Parsons Group		
Vance Nixon	Consulting Engineering Project Manager	Overall responsibility for geotechnical sediment sampling and design activities.
Mike Paulin, P.E.	Principal Geotechnical Engineer	Jointly responsible for QA oversight on geotechnical investigations and providing technical information on geotechnical considerations to the Transco offshore pipeline design and construction teams.
Bernard Remmes, P.E.	Principal Geotechnical Engineer	
Lake Superior Consulting		
Paul Beardon	Consulting Project Director	Responsible for QA review of geotechnical sediment sampling and design activities with respect to HDD activities.
Riley Shuoo	Engineering Supervisor	Responsible for review of geotechnical data and providing technical information with respect to HDD activities.
Sampling Contractor / Alpine Ocean Seismic Survey, Inc.		
Mark Kosakowski	Operations Manager	Responsible for development of the survey vessel plan and client/agency communications.
Jeff Motti	Party Chief and Senior Geologist	Responsible for overseeing mobilization and demobilization, testing, and equipment trials. Supervises survey operations, including QA and sample transfer to laboratories. Completes daily reports and other QA documents.
Laboratory Contractor (Geological – Primary[1]) / NGI		
Steve Garmon	Geotechnical Laboratory Project Manager	Overall responsibility for geotechnical data QA review and development of primary geotechnical laboratory report.
Laboratory Contractor (Chemical – Primary[1]) / Alpha Analytical		
Liz Porta	Chemical Laboratory Project Manager	Overall responsibility for development of chemical laboratory report for sediment chemistry samples.
James Todaro	QA Officer	Responsible for overall laboratory QA protocols and QA review of sediment chemistry data.

Table 1.1-1
Key Offshore Sampling and Analysis Personnel

Name	Role	Responsibilities
Laboratory Contractor (HARS Testing – Primary[1]) / EnviroSystems, Inc.		
Kirk Cram	Toxicity Laboratory Manager	Overall responsibility for development of interim Laboratory Reports (including chemical testing, physical testing, toxicity testing, and bioaccumulation testing), and the Final Laboratory Report, which includes all results and statistics required for HARS consideration.
Catie Sasso	QA Officer	Responsible for overall laboratory QA protocols and QA review of all HARS-related data.

[1] References in this document to a primary laboratory also include any entity this is sub-contracted to perform Project-related work for the given laboratory.

Key:
HARS = Historic Area Remediation Site
HDD = horizontal directional drill
NYD = New York District
QA = quality assurance
USACE = U.S. Army Corps of Engineers

1.2 Project Description/Background (Problem Definition)

Transco, a subsidiary of Williams Partners, L.P., is developing the Project to support National Grid's long-term growth, reliability, and flexibility beginning in the 2019/2020 heating season. The Project would provide up to 400,000 dekatherms per day of additional firm transportation capacity by adding to existing onshore Transco facilities in Pennsylvania and New Jersey, as well as expanding existing offshore facilities in New Jersey and New York waters. Transco anticipates that construction of the Project will begin early-2019 to meet an in-service date in late 2019.

The offshore portion of the proposed Project includes a new 26-inch outer-diameter pipeline that would extend from the Borough of Sayreville shoreline in Middlesex County, New Jersey, to the Rockaway Transfer Point, which is the interconnection point between Transco's existing Rockaway Delivery Lateral and the Lower New York Bay Lateral in New York waters. The new offshore pipeline, referred to as the "Raritan Bay Loop," would extend approximately 23.33 miles east-northeast across Raritan Bay and Lower New York Bay to the Rockaway Transfer Point, located in the Atlantic Ocean approximately 3 statute miles seaward of Rockaway, New York. From the Rockaway Transfer Point, the Rockaway Delivery Lateral supplies gas to an existing onshore meter and regulating station located in Kings County, New York. Approximately 6.11 miles of the Raritan Bay Loop route crosses New Jersey waters, while the remaining 17.38

miles crosses New York waters. The route passes offshore of Monmouth County and Middlesex County, New Jersey, and Richmond County and Queens County, New York.

Figures showing the location of the offshore study area are provided in Appendix A. This offshore study area is generally a 5,000-foot-wide corridor centered along the proposed route that is expected to serve as the offshore workspace, i.e., the area in which construction vessels will operate and anchor. Thus, the total offshore study area along the 23.33-mile route is approximately 68.6 million square yards. In addition to the 341,559 square yards that may be disturbed during the dredging/trenching for the pipeline, three temporary pits will be dredged at the offshore pipeline horizontal directional drill (HDD) entry/exit points. The sizes of these pits may total roughly 18,392 square yards, and Transco has collected two vibracore samples for chemical analysis at the sample sites for these potential pit locations in anticipation of the greater disturbance volume (versus one vibracore sample for chemical analysis at all other sites). Additional excavation would occur at the Rockaway Transfer Point and two Neptune cable crossings totaling roughly 6,873 square yards.

To support the design and evaluation of the Project, Transco collected sediment located along the proposed offshore route and analyzed the geotechnical and chemical characteristics in late 2016/early 2017 (as described in the Version 1.2 of the SAP/QAPP). Additional sampling and analysis is planned for 2018 to address the geotechnical and chemical characteristics of sediment within the Raritan Bay Channel and Chapel Hill Channel at depths deeper than what were sampled previously. To ensure that sediment sampling and analysis is sufficient for their review of an in-water dredge/fill project, New York State Department of Environmental Conservation (NYSDEC) and the New Jersey State Department of Environmental Protection (NJDEP) recommend that applicants submit a sampling and analysis plan for review before initiating sampling activities. The sampling design and analytical methods were developed based on a review of the environmental conditions and previous sampling data described in Sections 1.2.1 and 1.2.2.

The offshore portion of the Raritan Bay Loop includes installation of the pipeline and associated subsea facilities using several different methods, including a clamshell dredge, a jet trencher, and HDD techniques. For areas to be excavated using a clamshell dredge west of the New York/New Jersey state boundary near the Ambrose Channel crossing, Transco submitted a request to the USACE NYD DMMS for approval to dispose of suitable dredged material in the HARS under Section 103 of the Marine Protection, Research and Sanctuaries Act of 1972 (Section 103 request), consistent with the *Site Management and Monitoring Plan for the Historic*

Area Remediation Site, request dated September 15, 2017. As of August 2017, the total volume of clamshell dredged material was estimated to be approximately 590,000 cubic yards. Transco understands that the proposed use of the HARS must be evaluated according to the criteria published by the EPA in Title 40 of the Code of Federal Regulations (CFR) Parts 220–228. Figures 4 through and 8 in Appendix A show the HARS-related sampling locations as determined by the USACE NYD DMMS.

1.2.1 Environmental Setting

The proposed study area will cross a continuous expanse of open marine and estuarine waters, which consists of three major waterbodies—Raritan Bay, Lower New York Bay, and the Atlantic Ocean. The Project area is located within the Monmouth watershed management area and the Atlantic Ocean/Long Island Sound watershed, which drains most of the surrounding metropolitan areas and encompasses all marine waters in New York Harbor, Long Island Sound, Block Island Sound, Lower New York Bay/Raritan Bay, Sandy Hook Bay, and the waters that drain into them. The study area would cross three federally authorized navigation channels, including Ambrose Reach B (Ambrose Channel), Chapel Hill North (Chapel Hill Channel), and Raritan Bay West Reach (Raritan Bay Channel) (NOAA 2015). Ambrose Channel was dredged within the last 10 years as part of the USACE NYD's overall New York/New Jersey Harbor deepening project. Based on a Freedom of Information Act (FOIA) request submitted to the USACE NYD, Transco determined the Chapel Hill Channel was last dredged in March 2012. Transco has not yet determined when the Raritan Bay Channel was last dredged.

Measuring from mean lower low water, water depths along the route range from 0 to 77 feet. The minimum water depth is 0 feet at the shore. A proposed HDD segment exits in approximately 8 feet of water approximately 1,800 feet from the shoreline. From the shoreline to the Raritan Bay Channel the water depth varies from approximately 0 to 20 feet. The authorized depth within the Raritan Bay Channel at the proposed crossing is 35 feet, although actual depths extend to 46 feet along the Project route. From the Raritan Bay Channel to the Chapel Hill Channel the water depth ranges between approximately 14 feet and 25 feet. The authorized depth within the Chapel Hill Channel at the proposed crossing is 30 feet, though actual depths extend to 35 feet. The water depth for the route from Chapel Hill Channel to Ambrose Channel varies from approximately 10 to 41 feet. The maximum water depth (77 feet) is located within the Ambrose Channel. From Ambrose Channel to the Rockaway Transfer Point the water depth ranges from approximately 15 to 40 feet.

1.2.2 Sediment Characteristics and Potential Contaminants of Concern

Several previous investigations have been completed within Raritan Bay and Lower New York Bay and extending into the Atlantic Ocean near the Rockaway Peninsula to identify the physical characteristics of the sediments as well as the degree to which contaminants may exist. Based on data collected by the U.S. Geological Survey (USGS) and available in USGS Open File Report OFR 03-241 and on recent investigations conducted by E & E as part of the Rockaway Delivery Lateral Project (E & E 2009, 2011), the predominant sediment type in this area has been identified as mostly fine-to-coarse sands. Greater silt and clay content was generally observed near the mouth of the Raritan River, the entrance to Upper New York Bay, and Sandy Hook Bay. However, a review of sediment sample data obtained in 2002 as part of the Neptune Regional Transmission System project (E & E 2003) indicates that material primarily classified as silt may also extend throughout Raritan Bay.

Additionally, the 2002 sediment sample data obtained during the Neptune Regional Transmission System project indicated that a number of potential chemical contaminants had been identified in the vicinity of the proposed route for the Project (E & E 2003). Inorganic contaminants (i.e., metals) such as mercury, zinc, and arsenic were present, while organic contaminants such as polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) were detected to a much lesser degree. These sampling results are generally consistent with earlier sediment sampling in the vicinity of the Project (Adams and Benyi 2003; Adams et al. 1998), but there was a wide variation in results, depending upon sample location and depth. More recent investigations, conducted by E & E as part of the Rockaway Delivery Lateral Project, indicated that substrate immediately seaward of the Rockaway Peninsula was predominantly sandy with minimal contaminant levels (E & E 2009, 2011).

Transco conducted Project-specific sediment sampling in late 2016 under Version 1.2 of this SAP/QAPP. This survey was conducted, in part, to identify any potentially contaminated sediments along the offshore portion of the Raritan Bay Loop route. The laboratory results from the 2016 sediment sampling effort were generally consistent with previous surveys, and included concentrations of metals, pesticides, PCBs, semi-volatile organic compounds (including PAHs), and dioxins/furans that exceeded respective state screening thresholds along the offshore portion of the Raritan Bay Loop.

Transco's 2016 sediment sampling results identified elevated levels of contaminants in samples that were typically comprised of higher amounts of silt and clay, although contaminant concentrations do not appear to clearly correlate with the levels of silt and clay for all sample

locations. The contaminant levels were generally greater in the upper 3 feet of the seabed, except for a few notable locations where several contaminants were observed to be comparatively higher below the 3-foot depth, including at the Raritan Bay Channel, near the Chapel Hill Channel, and near the Ambrose Channel. The results of the 2016 survey also identified sediments in New York waters with contaminant levels that may be considered "acutely toxic" to aquatic life (i.e., exceeding NYSDEC Class C thresholds) are present along the Project route in or near the Raritan Bay Channel and Chapel Hill Channel crossings, the Morgan Shore Approach HDD entry site near Milepost (MP) 12.50, as well as in Raritan Bay between MP14.00 and MP15.00. A summary of the sediment chemistry analyses, complete tables of the sediment chemistry testing results, and the Project-specific SAP/QAPP for offshore sediment sampling are presented in the Offshore Environmental Sampling Report (Appendix J to Transco's application for a USACE NYD permit, dated June 27, 2017) and subsequent addenda and errata. Laboratory reports and data usability summary reports were also provided in that report.

Considering the variability in the 2016 physical and chemical sediment sample analysis results, Transco sought to define clamshell dredging prisms for the HARS suitability evaluation based on the likelihood that a given prism would pass all HARS testing criteria. Therefore, Transco identified recent federal maintenance dredging projects in the New York/New Jersey Harbor complex for which material had been evaluated for disposal at the HARS. Transco then submitted a request to the USACE NYD under FOIA for the results of physical and chemical sediment testing associated with the maintenance dredging projects, along with sample locations. The USACE NYD provided the requested information on April 24, 2017 (FOIA File No. FA-17-0091). The chemical and physical data were compared with the requested USACE NYD data and Transco's 2016 sampling data. The resulting clamshell dredging prisms were delineated as presented in Table 1.2-1.

Table 1.2-1
Evaluation for HARS Disposal
Dredge Prism Designations

Prism	MP Start	MP End
A1	15.77	16.60
A2	25.01	25.22
A2a[1]	25.01	25.30
A3	30.33	30.40
B1[2]	12.38	12.75
B2[2]	14.23	14.99
B3[2]	17.46	17.73

Table 1.2-1
Evaluation for HARS Disposal
Dredge Prism Designations

Prism	MP Start	MP End
B4[2]	24.46	24.91
C	12.75	14.23
D	14.99	15.77
E1	17.31	17.46
E1a[1]	17.23	17.46
E2	17.73	17.88
E2a[1]	17.73	17.97
F	24.00	25.01
Fa[1]	24.00	25.01
G	29.52	29.60

Notes:

(1) This prism is an alternative prism proposed for a deeper dredging scenario in Transco's additional Section 103 request, dated December 20, 2017

(2) Prism B is NOT proposed for ocean disposal. It is a clamshell-dredged area that will be disposed at an upland site and is provided for information purposes only.

Key:

HARS = Historic Area Remediation Site

MP = Milepost

1.3 Sampling and Analysis Task Description

1.3.1 Geotechnical and Bulk Sediment Chemistry Cores

The goal of the sampling and analysis program is to support the design and evaluation of the Project. To achieve this goal at the pre-construction stage, the following major tasks will be performed:

1. Collect offshore sediment samples for geotechnical and chemical analyses;
2. Evaluate physical/geotechnical properties of the sediment along the proposed Project route that could be disturbed during Project construction; and
3. Identify the concentrations of chemical contaminants that are present in sediment that could be disturbed during Project construction.

Figure 1 in Appendix A provides an overview of the offshore study area, while Figures 2 and 3 show the supplemental offshore sample sites. Sampling for chemical analysis will occur at sites previously sampled in late 2016 and 2017, and these sites are also included in Figures 2

and 3. Transco plans to further characterize sediment at two channel crossings (Raritan Bay West Reach and Chapel Hill North Channel) in 2018.

1.3.2 Evaluation for HARS Disposal

An additional goal of the 2018 sampling and analysis program is to determine the suitability of identified dredge material within the proposed prisms for HARS disposal. To achieve this goal, the following tasks will be performed, as appropriate, in accordance with the USACE NYD and EPA's *Region 2 Final Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal* (2016 Guidance) (April 2016) and the USACE NYD DMMS / EPA Region 2 Sampling and Testing Requirements for NAN-2016-00908-EHA (Sampling and Testing Requirements) provided to Transco on December 7, 2017 and January 18, 2018 (included as Appendix F):

1. Collect offshore sediment and surface water samples for toxicity and/or bioaccumulation testing (i.e., bioassays);
2. Collect offshore sediment and surface water samples for chemical and elutriate analysis; and
3. Collect offshore sediment for physical testing.

Figures 4 through 8 in Appendix A show planned offshore core locations. Sampling and analysis activities that occurred in 2016 and 2017 are also included in Figures 4 through 8 for reference.

1.4 Quality Goals and Objectives

Data quality objectives are qualitative and quantitative statements that clearly define the objectives of a project, define the most appropriate type of data, determine the appropriate procedures for data collection, and specify acceptable decision-error limits that establish the quantity and quality of data needed for decision-making. The Project's major sampling and analysis tasks are identified in Section 1.3. The general data quality objectives for each of these tasks are summarized in Table 1.4-1. Acceptance and performance criteria for field and analytical QC samples are outlined in Section 2.

Acceptance and performance criteria are often specified in terms of precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) parameters. Numerical acceptance criteria cannot be assigned to all PARCCS parameters, which are briefly described below, but general performance goals are established for most data-collection activities. Data assessment procedures throughout this SAP/QAPP outline the steps to be taken,

responsible individuals, and implications if QA objectives are not met. Measurement performance criteria, if applicable, were established following selection of the analytical laboratories (see Table 1.4-2).

Table 1.4-1
Data Quality Objects for Offshore Sampling and Analysis Tasks

Task	Data Quality Objective	Standards/Guidance[1]	Acceptance and Performance Criteria
Geotechnical Sampling and Analysis	Acquire geotechnical data sufficient to characterize offshore substrate for the purposes of engineering design, including the determination of suitable construction methods. Also determine sediment grain-size distribution as a basis for hydrodynamic modeling of potential Project-related sediment suspension and transport.	ASTM methods appropriate for sediment classification and each geotechnical test.	Criteria are based on the ASTM methods appropriate for each geotechnical test identified in Section 2 of this SAP/QAPP.
Sediment Chemistry Sample Collection and Analysis	Acquire sediment chemistry data sufficient to characterize the potential level of contaminants within the sediment that may be disturbed during construction to assist in review of potential environmental effects of the Project.	NYSDEC TOGS 5.1.9 In-Water and Riparian Management of Sediment and Dredged Material (2004); NJDEP's The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters (1997); and USACE and EPA Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal (2016)	Data must meet the acceptance and performance criteria documented in Section 2 of this SAP/QAPP.
Sediment and Surface Water Collection and Analysis for HARS disposal	Acquire sediment and surface water to support the chemical, physical, biotoxicity, and bioaccumulation testing required for consideration of the sediment for HARS disposal.	USACE and EPA Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal (2016), USACE NYD DMMS / EPA Region 2 Sampling and Testing Requirements for NAN-2016-00908-EHA	Data must meet the acceptance and performance criteria documented in Section 2 of this SAP/QAPP.

Table 1.4-1
Data Quality Objects for Offshore Sampling and Analysis Tasks

Task	Data Quality Objective	Standards/Guidance[1]	Acceptance and Performance Criteria

Note:
(1) Actual procedures and criteria subject to Project-specific modification, as described in this SAP/QAPP.

Key:
ASTM = American Society for Testing and Materials
DMMS = Dredge Material Management Section
EPA = U.S. Environmental Protection Agency
HARS = Historic Area Remediation Site
NJDEP = New Jersey Department of Environmental Protection
NYD = New York District
NYSDEC = New York State Department of Environmental Conservation
SAP/QAPP = Sampling and Analysis Plan / Quality Assurance Project Plan
TOGS = Technical and Operational Guidance Series
USACE = U.S. Army Corps of Engineers

Bulk Sediment Chemistry

Performance and acceptance criteria specific bulk sediment chemistry are provided in Table 1.4-2 and are based upon criteria provided in Alpha Analytical standard operating procedures (SOPs).

Table 1.4-2
Summary of PARCCS Parameter Criteria

Sampling Parameter	Analytical Method	PARCCS Parameters	Measurement Performance Criteria
Inorganics/Metals	SW-846 6020A	Precision	MS/MSD or DUP RPD: ≤20%
		Accuracy	LCS: 75–125% MS/MSD: 75–125%
Mercury	SW-846 7474	Precision	MS/MSD or DUP RPD: ≤20%
		Accuracy	LCS: 78–128% MS/MSD: 80–120%
		Accuracy	LCS: 70–130% MS/MSD: 70–130%
Polycyclic Aromatic Hydrocarbons	SW-846 8270D-SIM	Precision	MS/MSD RPD: 30%
		Accuracy	LCS: 40–140% MS/MSD: 40–140% Surrogates: 30–130%
Pesticides	SW-846 8081B	Precision	MS/MSD RPD: ≤50%
		Accuracy	LCS: 40–140% MS/MSD: 40–140%

Table 1.4-2
Summary of PARCCS Parameter Criteria

Sampling Parameter	Analytical Method	PARCCS Parameters	Measurement Performance Criteria
			Surrogates: 30–150%
Polychlorinated Biphenyl Aroclors	SW-846 8082A	Precision	MS/MSD RPD: ≤50%
		Accuracy	LCS: 40–140% MS/MSD: 40–140% Surrogates: 30–150%
Dioxins	SW-846 1613B	Precision	MS/MSD RPD: ≤20%
		Accuracy	LCS: In-house limits MS/MSD: In-house limits See laboratory SOP in Appendix D.
Total Organic Carbon	EPA 9060A	Precision	MS/MSD RPD: ≤25%
		Accuracy	LCS: 75–125% MS/MSD: 75–125%

Key:
LCS = laboratory control samples
MS/MSD = matrix spike/matrix spike duplicate
PARCCS = precision, accuracy, representativeness, completeness, comparability, and sensitivity (as described in Section 1.4)
RPD = relative percent difference
SOP = standard operating procedure

Evaluation for HARS Disposal

Performance and acceptance criteria specific to HARS disposal-related analyses are provided in Tables 1.4-3 through 1.4-10 and are based upon criteria provided in the 2016 Guidance. In case of conflict between the tables below and the 2016 Guidance, the 2016 Guidance shall prevail unless the deviations have been approved in writing by the USACE NYD DMMS and/or EPA Region 2. The analyses outlined in Tables 1.4-7, 1.4-9, and 1.4-10 each require reference toxicant testing on each batch of organisms received from an outside supplier or monthly for all in-house cultures as outlined in the 2016 Guidance.

Table 1.4-3
Evaluation for HARS Disposal
Metals (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn) Performance/Acceptance Criteria

QC Measurement	Frequency	Calculation	Control/Acceptance Criteria
Laboratory method blank	1 per 20 samples	NA	No analyte should be detected at > RL
Matrix spikes (triplicate)	1 set per 20 samples	% recovery and % RSD	Recovery: 70–130% RSD: 30%
Standard Reference Material (SRM) (water only)	1 per 20 samples	% recovery *evaluated for analytes > 3x RL	Recovery: 70–130%

Table 1.4-3
Evaluation for HARS Disposal
Metals (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn) Performance/Acceptance Criteria

QC Measurement	Frequency	Calculation	Control/Acceptance Criteria
LCS/LFB	1 per 20 samples	% recovery	Recovery: 70–130%
Initial calibration check standards	Immediately following calibration curve	% recovery	Recovery: 90–110%
Continuing calibration checks using standards	Minimum – check calibration at middle and end of each batch or 1 per 10 analyses, whichever is greater	% recovery	Recovery: 90–110% from initial calibration for each analyte

Source: USACE and EPA 2016.

Key:
Ag = Silver
As = Arsenic
Cd = Cadmium
Cr = Chromium
Cu = Copper
HARS = Historic Area Remediation Site
Hg = Lead
LCS = laboratory control sample
LFB = laboratory fortified blank
NA = not applicable
Ni = Nickel
Pb = Lead
QC = quality control
RL = reporting limit
RSD = relative standard deviation
Zn = Zinc

Table 1.4-4
Evaluation for HARS Disposal
Organics (Pesticides, PCBs, PAHs) Performance/Acceptance Criteria

QC Measurement	Frequency	Calculation	Control/Acceptance Criteria
Laboratory method blank	1 per 20 samples	NA	No analyte should be detected at > RL
Matrix spikes (triplicate)	1 set per 20 samples	% recovery and % RSD	Recovery: 50–150% RSD: 50%
Standard reference material (SRM) (water only)	1 per 20 samples	% recovery *evaluated for analytes >3x the RL	Recovery: 50–150%
LCS/LFB	1 per 20 samples	% recovery	Recovery: 50–150%
Surrogate Standards	Each sample	% recovery	Recovery: 30–150% *all applicable surrogate standards must be within the acceptance range to be considered acceptable
Initial calibration check standards	Immediately following calibration curve	% recovery	Recovery: 80–120%

Table 1.4-4
Evaluation for HARS Disposal
Organics (Pesticides, PCBs, PAHs) Performance/Acceptance Criteria

QC Measurement	Frequency	Calculation	Control/Acceptance Criteria
Continuing calibration checks using calibration standards	Minimum – middle and end of each batch or 1 per 10 analyses, whichever is greater	% recovery	Recovery: 80–120% from initial calibration for each analytes

Source: USACE and EPA 2016.

Key:
HARS = Historic Area Remediation Site
LCS = laboratory control sample
LFB = laboratory fortified blank
NA = not applicable
PAHs = polycyclic aromatic hydrocarbon
PCBs = polychlorinated biphenyls
QC = quality control
RL = reporting limit
RSD = relative standard deviation

Table 1.4-5
Evaluation for HARS Disposal
Dioxins/Furans Performance/Acceptance Criteria

QC Measurement	Frequency	Calculation	Control/Acceptance Criteria
Laboratory method blank	1 per 20 samples	NA	No analyte should be detected at > RL
Matrix spikes (triplicate)	1 set per 20 samples	% recovery and % RSD	Recovery: 50–150% RPD: 50%
Standard reference material (SRM) (sediment only)	1 per 20 samples	% recovery *evaluated for analytes >3x the RL	Recovery: 50–150%
LCS/LFB	1 per 20 samples	% recovery	Recovery: 50–150%
Internal standards (labeled compounds)	Each sample	% recovery	Recovery: 25–150%
Initial calibration check standards	Immediately following calibration curve	% recovery	Recovery: 80–120% for both unlabeled and labeled compounds
Continuing Calibration Checks using calibration standards	Minimum – middle and end of each batch or 1 per 10 analyses, whichever is greater	% recovery	Recovery: 80–120% from initial calibration for unlabeled compounds 50–150% from initial calibration for labeled compounds

Table 1.4-5
Evaluation for HARS Disposal
Dioxins/Furans Performance/Acceptance Criteria

QC Measurement	Frequency	Calculation	Control/Acceptance Criteria
Source: USACE and EPA 2016.			
Key: HARS = Historic Area Remediation Site LCS = laboratory control sample LFB = laboratory fortified blank NA = not applicable QC = quality control RL = reporting limit RPD = relative percent difference RSD = relative standard deviation			

Table 1.4-6
Evaluation for HARS Disposal
TOC and Physical Parameters for Sediment Performance/Acceptance Criteria

QC Measurement	QC Measurement	Frequency	Calculation	Control/Acceptance Criteria
TOC, Grain Size, Specific gravity, Bulk density	Triplicate	1 triplicate per 20 samples (for each parameter)	% RSD	RSD: 20%
Source: USACE and EPA 2016.				
Key: HARS = Historic Area Remediation Site QC = quality control RSD = relative standard deviation TOC = total organic carbon				

Table 1.4-7
Evaluation for HARS Disposal
Summary of Test Conditions and Test Acceptability Criteria for Solid Phase Toxicity Tests

Test Organism	Mysid, *Americamysis bahia*	Amphipod, *Ampelisca abdita* [1]
Test Type	Static non-renewal (initiate static renewal methods if un-ionized ammonia in overlying water exceeds levels in Table 1.4-8)	Static non-renewal (static renewal if porewater ammonia concentrations exceed 20 ppm during test)
Test Duration	10 days	
Temperature	20 ± 2°C	*A. abdita* 20 ± 2°C; *R. abronius* 15 ± 2°C; *E. estuarius* 15 ± 2°C; *L. plumulosus* 25 ± 2°C
Light Quality	Ambient laboratory illumination	

Table 1.4-7
Evaluation for HARS Disposal
Summary of Test Conditions and Test Acceptability Criteria for Solid Phase Toxicity Tests

Test Organism	**Mysid, *Americamysis bahia***	**Amphipod, *Ampelisca abdita*** [1]
Photoperiod	16-hour light, 8-hour dark	Constant light (reference toxicant test can be conducted using a 16-hour light, 8-hour dark cycle)
Test Chamber Size	1 L	1 L or one quart Mason Jars
Sediment-water Ratio	Add ~625 mL water to 200 cc (~175 mL) homogenized whole-sediment	
Age of Test Organisms	1–5 days; 24-hour range in age	Using nested sieves: *A. abdita* - subadults, 3–5 mm, retained on 0.7 mm sieve after passing through a 1.0 mm sieve *R. abronius* - 3–5 mm, retained on 1.0 mm sieve with large individuals (≥ 5mm) excluded *E. estuarius* - 3–5 mm, retained on 1.0 mm sieve with large individuals (≥ 5mm) excluded *L. plumulosus* - subadults, 2–4 mm, retained on 0.5 mm sieve after passing through a 0.7 mm sieve
No. of Organisms per Test Chamber	20	
Treatment Levels	Control, reference, test sediment	
No. of Replicates per Treatment / Concentration	5	
No. of Organisms per Treatment / Concentration	100	
Feeding Regime	Add up to 0.2 mL *Artemia* nauplii concentrate at least once a day during biological to prevent cannibalism	Must not be fed during 10-day biological test
Test Chamber / Solution Aeration	Gently aerate all chambers to achieve approximately 100 bubbles/minute	
Dilution Water	Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water, or clean seawater	
Salinity	30 ± 2 ‰	*A. abdita* 28 ± 2‰ *R. abronius* 28 ± 2‰ *E. estuarius* 20 ± 2‰ *L. plumulosus* 20 ± 2‰
pH	7.8 ± 0.5 (target)	
Dissolved Oxygen	DO concentrations must not fall below 40% saturation in any chamber	DO concentrations in each chamber should be maintained at ≥ 90% saturation
Endpoint	Mortality – Record total number of live organisms at the end of the test	

Table 1.4-7
Evaluation for HARS Disposal
Summary of Test Conditions and Test Acceptability Criteria for Solid Phase Toxicity Tests

Test Organism	Mysid, *Americamysis bahia*	Amphipod, *Ampelisca abdita* [1]
Test Acceptability Criterion	≥ 90% mean survival in control organisms. If mean reference and mean test organism survivals are <90%, test may need to be repeated.	≥ 90% mean survival in control organisms. If mean reference and mean test organism survivals are <80%, test may need to be repeated.
Test Method References	ASTM (2011); Ferretti et al. (2000); Miller et al. (1990); EPA (1994, 2002b)	ASTM (2011); Ferretti et al. (2000); EPA (1994, 2002b)

Source: USACE and EPA 2016.

Notes:

(1) Preferred species is *Ampelisca abdita. Leptocheirus plumulosus* is the alternate species when test sediment is composed of more than 5% mud (silt + clay) and no greater than 85% clay. *Eohaustorius estuarius* and *Leptocheirus plumulosus* are possible alternates when the mud content (silt + clay) of the test sediment is less than 5%.

Key:

°C = degrees Celsius
cc = cubic centimeter
DI = deionized
HARS = Historic Area Remediation Site
L = liter
mL = milliliters
mm = millimeters
ppm = parts per million

Table 1.4-8
Evaluation for HARS Disposal
Not-to-exceed Overlying Water Un-ionized Ammonia Threshold and pH Values for Solid Phase Testing Using *A. bahia*

Average 10-Day pH	Not to Exceed Threshold (ppm)
≥ 8.1	0.5
7.9 – 8.0	0.4
7.7 – 7.8	0.3
≤ 7.6	0.2

Source: USACE and EPA 2016.

Key:

HARS = Historic Area Remediation Site
ppm = parts per million

Table 1.4-9
Evaluation for HARS Disposal
Summary of Test Conditions and Test Acceptability Criteria for Water Column (SPP) Toxicity Tests

Test Organism	**Mysid, *Americamysis bahia***	**Fish, *Menidia berylina* [1]**	**Bivalve Larvae, *Mytilus galloprovincialis* [2]**
Test Type	Static non-renewal		
Test Duration	96 hours		48–72 hours development into straight hinge prodissoconch larvae (D-shape stage)
Temperature	20 ± 2°C		*M. edulis* 16 ± 2°C; *M. galloprovincialis*, 16 ± 2°C; *M. mercenaria* 25 ± 2°C; *C. virginica* 25 ± 2°C; *M. lateralis* 25 ± 2°C
Light Quality	Ambient laboratory illumination		
Photoperiod	16-hour light, 8-hour dark		
Test Chamber Size	600 mL minimum	250 mL minimum	
Test Solution Volume	400 mL minimum	200 mL minimum	
Age of Test Organisms	1–5 days; 24-hour range in age	9–14 days; 24-hour range in age	Use embryos within 4 hours of fertilization
No. of Organisms per Test Chamber	20		20–30 embryos/mL
No. of Replicates per Concentration	5		
No. of Organisms per Concentration	100	100 minimum	Per test chamber N = S (Vs / Vt), where: N = embryo density in test chamber S = mean embryo density in stock suspension Vs = volume of stock added to test chamber Vt = total volume of test solution Embryos per test chamber divided by embryos per mL equals mL of stock per chamber
Feeding Regime	*Artemia* nauplii are made available while holding prior to the test; add 0.2 mL *Artemia* nauplii concentrate daily to each test chamber.	*Artemia* nauplii are made available while holding prior to the test; 0.2 mL <24 hour old concentrated *Artemia* sp. to each testing chamber after 48 hours of testing.	None during test as uneaten food might decrease DO and biological activity of some test materials, and the embryos/larvae can survive without feeding for 72+ hours

Table 1.4-9
Evaluation for HARS Disposal
Summary of Test Conditions and Test Acceptability Criteria for Water Column (SPP) Toxicity Tests

Test Organism	Mysid, *Americamysis bahia*	Fish, *Menidia berylina* [1]	Bivalve Larvae, *Mytilus galloprovincialis* [2]
Test Solution Aeration	None, unless DO concentration falls below 4.0 mg/L; rate should not exceed 100 bubbles/min; if aeration is necessary, all chambers must receive the same treatment.		None, since bubbles can collect within larval mantle cavity. Aerate only if DO falls 60% saturation
SPP	Use unfiltered near (sub)surface dredging site water. Dredged sediment and water are combined in a sediment-to-water ratio of 1:4 (volume/volume).		
SPP Holding Time	Use within 24 hours of preparation		
Dilution Water	Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water, or clean natural seawater.		
Salinity	30 ± 2 ‰	30 ± 2‰	30 ± 3 ‰
pH	7.8 ± 0.5 (target)		
DO	DO concentrations in each chamber must not fall below 40% saturation.		DO concentrations in each test chamber must be between 60 and 100% saturation at all times
Test Concentrations	Minimum of four suspended particulate treatment levels (%): 0 (control, diluent), 10, 50, 100 (1% if high toxicity expected).		
Endpoint	Mortality (LC_{50}) - Record survivorship per replicate at 0, 24, 48, 72 and 96 hours.		Mortality (LC_{50}) - mortality / abnormality (EC_{50})
Test Acceptability Criterion	≥ 90% survival in control (i.e., 0% SPP) organisms	≥ 90% survival in control (i.e., 0% SPP) organisms	The number of embryos that result in live larvae with completely developed shells at the end of the control (i.e., 0% SPP) test must be at least 70% of the initial number for oysters or 60% for mussels and clams, and at least 90% of all introduced embryos should be alive at the end of test.
Test Method References	ASTM (2002a, 2003); EPA (2002b)	ASTM (2002a); ASTM (2003); EPA (2002b)	ASTM (2004)

Table 1.4-9
Evaluation for HARS Disposal
Summary of Test Conditions and Test Acceptability Criteria for Water Column (SPP) Toxicity Tests

Test Organism	Mysid, *Americamysis bahia*	Fish, *Menidia berylina* [1]	Bivalve Larvae, *Mytilus galloprovincialis* [2]

Source: USACE and EPA 2016.

Notes:

(1) *Menidia berylina* or *M. menidia* are the preferred species. *M. peninsulae* is an alternate species. It is anticipated that *M. berylina* will be used.

(2) *Mytilus edulis* or *Mytilus galloprovincialis* are the preferred species. *Mercenaria mercenaria, Crassostrea virginica,* or *Mulinia lateralis* are alternate species. It is anticipated that *M. galloprovinicallis* will be used.

Key:

°C = degrees Celsius
DI = deionized
DO = dissolved oxygen
EC_{50} = median effective concentration
HARS = Historic Area Remediation Site
LC_{50} = median lethal concentration
mg/L = milligrams per liter
mL = milliliters
SPP = suspended particulate phase

Table 1.4-10
Evaluation for HARS Disposal
Summary of Test Conditions and Test Acceptability Criteria for 28-day Bioaccumulation Tests

Test Organism	Sand Worms, *Neanthes virens*	Bivalves, *Macoma nasuta*
Test Type	Flow-through, six volume exchanges per day or static renewal, 80% water volume change every two days	
Test Duration	28 days	
Temperature	20 ± 2°C	12–14°C
Light Quality	Ambient laboratory illumination	
Photoperiod	16-hour light, 8-hour dark	
Test Chamber Size	37 L (minimum of 20 L of water per tank)	
Sediment Depth / Overlying Water	Minimum of 5 cm of sediment/tank; 7–11 cm recommended. 16 L overlying water minimum volume.	Minimum of 5 cm of sediment/tank. 7–11 cm recommended, especially when sediment has high silt content, may need up to 11 cm of sediment/tank (minimum 16 L overlying water)
No. of Organisms per Test Chamber	20	
Pretest Tissue	Tissues of randomly selected organisms, held in clean seawater (i.e. sediment-free water) for 24 hours to be tested prior to initiation of bioaccumulation testing to determine pretest concentrations.	
Treatment Levels	Control, reference, test sediment	

Table 1.4-10
Evaluation for HARS Disposal
Summary of Test Conditions and Test Acceptability Criteria for 28-day Bioaccumulation Tests

Test Organism	**Sand Worms, *Neanthes virens***	**Bivalves, *Macoma nasuta***
No. of Replicates per Treatment	3 control, 5 reference, 5 test	
No. of Organisms per Treatment	60 control, 100 reference, 100 test (contingent on tissue mass required to perform chemical analyses)	60 control, 100 reference, 100 test
Feeding Regime	None	
Test Aeration	Flow through: aerate only if DO falls below 4.0 mg/L. If aeration necessary, all tanks must be aerated. Static renewal: aerate all aquaria	Flow through: aerate only if DO falls below 4.0 mg/L. Static renewal: aerate all aquaria
Dilution Water	Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water, or clean seawater.	
Salinity	30 ± 2 ‰	
pH	7.8 ± 0.5 (target)	
DO	DO concentrations in each chamber must not fall below 60% saturation	
Depuration	24 hours in clean seawater at end of test	
Endpoint	Measured tissue concentrations (no. of live organisms at end of test should be recorded)	
Test Acceptability Criterion	If < 90% survival in control organisms, then determine whether: a) there are adequate replicates to obtain sufficient statistical power; b) there is adequate tissue for chemical analyses; c) organisms stressed (e.g., reference toxicant results outside 2 standard deviations, failure to burrow, reduced sediment processing rate, evidence of disease); d) there is contamination of the system; e) the control sediment is contaminated; f) pretest tissue concentrations were high; and g) there are other quality control problems. If the answer is “no” to a or b, or “yes” or “not sure” to c-f, bioaccumulation tests may have to be rerun. Consult with NYD immediately.	
Test Method References	ASTM (2002b); Boese and Lee (1992); Lee et al. (1993)	ASTM (2002b); Boese and Lee (1992); Ferraro et al. (1990)

Source: USACE and EPA 2016.

Key:
°C = degrees Celsius
cm = centimeter
DO = dissolved oxygen
HARS = Historic Area Remediation Site
L = liter
mg/L = milligrams per liter
NYD = New York District

1.4.1 Precision

Precision measures the reproducibility of measurements under a given set of conditions. Specifically, it is a quantitative measure of the variability of a group of measurements compared with their average value, usually stated in terms of standard deviation or coefficient of variation. It may also be measured as the relative percent difference (RPD) between two values. Precision includes the interrelated concepts of instrument- or method-detection limits and multiple field sample variance. Sources of this variance are sample heterogeneity, sampling error, differences among observers, and analytical error. Communication between individuals in charge of sampling, via the field logbook or otherwise, should reduce any measurement-precision errors.

1.4.2 Accuracy

Accuracy measures the bias of the measurement system. Sources of this error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analysis. Data interpretation and reporting may also be significant sources of error. Typically, analytical accuracy is assessed through the analysis of spiked samples and may be stated in terms of percent recovery or the average (arithmetic mean) of the percent recovery. Blank samples may also be analyzed to assess sampling and analytical bias (i.e., sample contamination). Background measurements similarly assess measurement bias. The number of samples collected will affect the confidence of the statistical data evaluation.

1.4.3 Representativeness

Representativeness expresses the degree to which data represent a characteristic of a population, a parameter variation at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that is most concerned with proper design of the measurement program. Sample/measurement locations may be biased (judgmental) or unbiased (random or systematic). Representativeness of the sampling scheme will be determined with evaluation of the historical data and statistical evaluation of the results compared with the reference site or existing data.

1.4.4 Completeness

Completeness is defined as the percentage of measurements that are judged to be valid. Although a quantitative goal must be specified, the completeness goal is the same for all data uses—that a sufficient amount of valid data is generated. A completeness goal of 95% will be established for this Project.

1.4.5 Comparability

Comparability is a qualitative parameter that expresses the confidence with which one dataset may be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved through the use of standard techniques to collect and analyze samples. Historical data will be evaluated to ensure the methods and reporting limits are comparable to the proposed sampling. Data will only be evaluated if it is determined to be comparable.

1.4.6 Sensitivity

Sensitivity refers to the capability of a method or instrument to detect a given analyte concentration and reliably quantitate the analyte at that concentration. Analytical results for samples that are non-detect for a particular analyte that have reporting limits greater than the applicable standards and/or screening levels cannot be used to demonstrate compliance with the applicable standards and/or screening levels.

1.5 Special Training/Certification

The laboratory conducting geotechnical analyses, NGI, is expected to be certified by the American Society for Testing and Materials (ASTM) for testing soil and rock. The laboratory conducting bulk sediment chemistry analyses, Alpha Analytical, will be certified by the New York State Department of Health Environmental Laboratory Accreditation Program (NYSDOH ELAP) for Solid and Hazardous Waste and Contract Laboratory Program. The laboratory conducting sediment and surface water analyses for evaluation of sediment for HARS disposal, ESI, will be certified by the U.S. Department of Defense ELAP (DoD ELAP).

1.6 Documentation and Records

Geotechnical Boring and Bulk Sediment Chemistry Cores

Data generated during the sampling and analysis activities will be documented through the formats listed in Table 1.6-1. Field records will be generated in hard copy field logs or on Project-specific datasheets (i.e., paper media), then scanned or transferred into electronic format. Electronic Project records will be maintained on internal, secure Project drives that are accessible only to the Project team.

The Project documents identified in this SAP/QAPP that will be submitted to regulatory agencies include the sediment chemistry laboratory reports, which will be provided as appendices to an Offshore Environmental Sampling and Analysis Summary Report Addendum (see Sections 1.6.2 and 1.6.3 for further discussion). In addition, the chemical and biological reports generated

from the HARS testing may be included in the addendum. The core logs and/or other portions of the Geotechnical Site Investigation Report may also be provided to regulatory agencies, as warranted. As applicable, Project documents will be submitted to regulatory agencies as pdf files via e-mail, electronic media (i.e., CD or DVD), and/or posted on a password-protected file transfer protocol site. Abridged hard copies (i.e., certain appendices may not be printed) will also be provided to agencies.

Table 1.6-1
Geotechnical and Bulk Sediment Chemistry
Offshore Sampling and Analysis Data Records

Document Type	Format	Originator	Repository	Submittal Frequency
Field Logbooks	Hard copy and Electronic (pdf and/or Excel spreadsheet)	Sampling Contractor	Sampling Contractor	As needed to consultant (E & E or INTECSEA) or Transco
Progress Reports and Sediment Core Log Sheets	Hard copy and Electronic (pdf and/or Excel spreadsheet)	Sampling Contractor	Sampling Contractor and Consultant (E & E or INTECSEA)	Electronic form only: Daily to Consultant (E & E and/or INTECSEA) and Transco Hard copy: Included as appendix with field report.
Laboratory Reports	Hard copy (with exceptions)[1] and electronic (pdf and Excel spreadsheet or CSV)	Laboratory	Laboratories and Consultant (E & E or INTECSEA)	Preliminary and/or final sediment chemistry reports to consultant and Transco within 30 days of sample delivery. Final sediment chemistry lab reports to regulatory agencies as part of survey summary report.[2]
Summary Reports	Hard copy and Electronic (pdf)	Sampling Contractor, Consultant (E & E)	Sampling Contractor and/or Consultant (E & E or INTECSEA)	Draft and final survey summary reports to Transco. Final environmental (chemistry) survey summary report(s) to regulatory agencies.[2]

Notes:

(1) Very large appendices may be submitted only in electronic format, at Transco's discretion.

(2) Reports submitted to regulatory agencies as part of license/permit applications will be retained by those agencies according to their policies and state/federal laws.

Key:

CSV = comma separated value

E & E = Ecology and Environment, Inc.

Evaluation for HARS Disposal

Data generated during the sampling and analysis activities will be documented through the formats listed in Table 1.6-2. Field records will be generated in hard copy field logs or on Project-specific datasheets (i.e., paper media), then scanned or transferred into electronic format.

Electronic Project records will be maintained on internal, secure Project drives that are accessible only to the Project team.

The Project documents identified in this SAP/QAPP that will be submitted to USACE NYD DMMS include all chemical and biological laboratory reports, which will be provided as part of a Final Laboratory Report (see Section 1.6.3 for further discussion). Checklists for the format and content for biological and chemical data packages to be submitted to the USACE NYD DMMS will be completed by ESI and are provided in Appendix E. The core logs may also be provided to regulatory agencies, if requested. These reports may also be submitted to other regulatory agencies as part of an Offshore Environmental Sampling Report Addendum.

Table 1.6-2
Evaluation for HARS Disposal
Offshore Sampling and Analysis Data Records

Document Type	Format	Originator	Repository	Submittal Frequency
Field Logbooks	Hard copy and Electronic (pdf and/or Excel spreadsheet)	Sampling Contractor	Sampling Contractor	As needed to consultant (E & E) or Transco
Progress Reports and Sediment Core Log Sheets	Hard copy and Electronic (pdf and/or Excel spreadsheet)	Sampling Contractor	Sampling Contractor and Consultant (E & E)	Electronic form only: Daily to Consultant (E & E) and Transco Hard copy: Included as appendix with field report.
Laboratory Reports	Electronic (pdf and Excel spreadsheet or CSV)	Laboratory	Laboratories and Consultant (E & E)	Interim Chemical and Biological Reports will be generated throughout the program as the analyses are completed (per matrix).
Final Laboratory Report with Executive Summary	Hard copy and Electronic (pdf)	Laboratory	Sampling Contractor and/or Consultant (E & E)	Final Laboratory Report with Executive Summary containing all data and statistics as required for HARS evaluation will be provided at the conclusion of the testing.[1]

Notes:

(1) Reports submitted to regulatory agencies as part of license/permit applications will be retained by those agencies according to their policies and state/federal laws.

Key:

CSV = comma separated value
E & E = Ecology and Environment, Inc.
HARS = Historic Area Remediation Site

1.6.1 Field Documentation and Reporting

The sampling contractor will maintain a daily operations log that records the time and description of all significant events during the investigation, which will be made available to Transco on request. Based on the daily operations logs, the sampling contractor will prepare a daily progress report that includes the following:

1) Brief summary of work performed and narrative of events
2) Changes to scope
3) Disputed time
4) Delays, malfunctions, deviations, and / or special problems encountered
5) Weather
6) Safety incidents
7) Tally of events including working time, depth of corings, downhole tests performed, standby time, downtime and cause (i.e., weather, equipment or vessel), consumables
8) Percentage of work scope completed
9) Vessel location
10) Brief outline of findings
11) Work planned for next 24-hour period and three-day look-ahead
12) Company and contractor comments

The sampling contractor will provide the daily progress report to Transco by 08:00 a.m. (Central Time) the following day.

The sampling contractor and/or Transco's designated field geologist or geotechnical engineer will visually inspect all vibracore samples for bulk sediment chemistry and for evaluation for HARS disposal, documenting measurements for individual horizons or strata in a field logbook.

Individual field logs will be made for each geotechnical sample collected in the field. Logs will include the following information: full information on position, coordinate system, and water depth; and length in feet of final penetration and sample recovery. All geotechnical boring logs (hard copies and electronic files) will be presented at the same vertical scale. All other geotechnical information will be recorded when processed at NGI for inclusion in the Geotechnical Site Investigation Report.

For sediment chemistry core samples, basic information collected will be recorded on a sediment core log and will include the following:

1) Project and core sample identification numbers
2) Latitude/longitude coordinates of the sample location
3) Digital photographs of each sample
4) Type of equipment used to obtain the sample
5) Visual classification of each major sediment horizon or strata encountered in accordance with the United Soil Classification System
6) Water depth and sea state core penetration length
7) Core recovery length
8) Presence of debris, contaminants, or living organisms

Site information for surface sediment grab samples will also be recorded in a field logbook, including the actual sampling coordinates, sample number, water depth, stage of the tide, time, and the sea/weather conditions.

The sampling contractor will compile a Field Investigation Report of the field sampling and analysis activities. The sampling contractor will submit the draft field report to Transco following completion of all their field activities, at the time of vessel demobilization. Transco will distribute the draft report to other contractors such as INTECSEA and E & E for review as appropriate. The field report will include, at a minimum, the following:

- Brief assessment of sampling activities performed;
- Set of preliminary core logs as produced in the field, including full-size photocopies for any interpretation logs that are hand-drawn;
- Summary log of core information (samples, descriptions, and results of onboard tests);
- Sampling activity summary that concisely highlights all significant features; and
- Copy of the daily operations log (hard and digital).

The sampling contractor will issue a final version of the Field Investigation Report following review and approval by Transco.

1.6.2 Laboratory Data Reporting

The sampling contractor and/or INTECSEA will be responsible for obtaining results of geotechnical analyses from NGI in a format that can be incorporated into the Geotechnical Site Investigation Report (see Section 1.6.3).

After completing the bulk sediment chemistry laboratory analysis, Alpha Analytical will submit a Chemistry Analytical Data Report to E & E, and E & E will provide the report to Transco for review. The laboratory will provide a full laboratory report (Level IV or Contract Laboratory Program-like deliverable) including QA/QC documentation in pdf format, electronic data deliverables, and excel summary tables of all results. The laboratory will submit revised reports as necessary to address QA/QC deficiencies or errors.

As data is completed for each matrix for the HARS sediment evaluation, ESI will submit interim chemical and biological reports to E & E for review. Each interim laboratory report will be consistent with Level IV reporting or Contract Laboratory Program-like deliverable including QA/QC documentation in pdf format, electronic data deliverables, and excel summary tables of all results. The laboratory will submit revised reports as necessary to address QA/QC deficiencies or errors. The interim laboratory reports will also be consistent with the requirements outlined (per the 2016 Guidance) in Appendix E.

1.6.3 Summary Reports

Geotechnical Boring

It is anticipated that the sampling contractor or INTECSEA (and/or Lake Superior Consulting) will develop a Geotechnical Site Investigation Report(s). This report will include a summary of all information collected during the geotechnical investigation(s), the Field Investigation Report(s), and the results of the standard and any advanced geotechnical testing performed by NGI. A draft version of the Geotechnical Site Investigation Report, and any supplements, will be submitted to Transco following completion of all geotechnical laboratory analyses. The report will include the following information:

- An overview of the geotechnical (boring) results, with highlights of all hazards, obstructions, constraints and significant anomalies encountered;
- Purpose and defining limits of geotechnical investigation task, dates of acquisition, brief description of utilized equipment and vessel;
- Discussion of local sediment stratigraphy and an interpretation of the local sediment properties;
- Laboratory test results, including ‘grouping’ summaries for similar facies;
- Description of positioning systems, calibrations, data reduction, results, and performance (may be provided in the field report);

- Description of geotechnical acquisition systems, calibrations, data reduction, results, and performance (may be provided in the field report); and
- Supporting data, including final core profiles, vessel specifications, coring systems, positioning and water-depth measurements, field-sampling procedures, field-sediment testing, drill logs, and daily progress reports, if not previously submitted in the field report. All geotechnical boring logs will be presented at the same vertical scale.

The sampling contractor or INTECSEA (and/or Lake Superior Consulting) will issue a final version of the Geotechnical Site Investigation Report, and any supplements, following Transco's review and approval of final edits.

In addition to reporting on hard copy media, the sampling contractor will submit the final Geotechnical Site Investigation Report in electronic format, including charts, drawings, figures, and plots. The media and precise format will be confirmed during reporting, but it is anticipated to be a browseable CD or DVD with the report presented in pdf format and all data provided by NGI laboratory will be in Microsoft Excel tables. All charts and maps will be made available in hard copy and in electronic format such as AutoCAD 2000-compatible digital format.

Bulk Sediment Chemistry

An Offshore Environmental Sampling and Analysis Summary Report addendum that synthesizes the results of Alpha Analytical's sediment chemistry reports will also be prepared by E & E for inclusion as part of Project license and permit application packages. E & E will provide a draft version of this summary report addendum in electronic format to Transco for review and approval. Following Transco's approval, a final version of the Offshore Environmental Sampling and Analysis Summary Report addendum will be submitted to agencies to provide a more concise description of chemical characteristics of the sediment for ease of agency review. This summary report addendum will include the original laboratory reports as appendices.

Evaluation for HARS Disposal

After HARS-related testing is complete, a Final Laboratory Report will be developed by ESI, reviewed by Transco and E & E, and submitted to the USACE NYD DMMS electronically. This report will include data summaries for all matrices, biological tests, and analyses conducted, and will detail all sample and test results; quality control sample results; pertinent sampling, extraction, and analysis locations and dates; statistical analyses; supporting raw data (i.e., "… any laboratory worksheets, records, memoranda notes, or exact copies thereof, that are the result

of original observations and activities or a study and are necessary for the reconstruction and evaluation of the report of that study" [40 CFR 792]); and any other calculated performance criteria. Checklists outlining the minimum requirements for biological testing and chemical analytical data reporting will be completed as (per the 2016 Guidance) and are provided as Appendix E.

In addition, ESI will develop an executive summary that will be submitted to Transco and E & E for review and approval. The executive summary will include names of laboratories and staff members that performed the tests; figures and tables with sample locations; summary table of the physical, chemical, and biological testing results, including all individual replicates and all control and reference replicates; relevant statistical comparisons and calculations; a discussion of any problems encountered and corrective actions taken; and any departures from QA or protocols specified in this SAP/QAPP. Transco will submit the Final Laboratory Report and executive summary to the USACE NYD DMMS and to the USACE NYD regulatory office. Reports generated from the HARS testing may also be transmitted to other agencies as part of the Offshore Environmental Sampling Report Addendum.

1.6.4 Record Retention

Transco's sampling contractor will keep accurate logs and records of all sampling activities and will provide Transco with complete, legible copies of logs and records upon completion of activities at each sample site. The sampling contractor will preserve all records in good condition until they are delivered and deemed acceptable by Transco. Transco will have the right to examine such records at any time prior to delivery.

The geotechnical laboratory (NGI) will submit all data and laboratory reports to the sampling contractor, who will forward them to Transco and the appropriate consultant (E & E, INTECSEA, and/or Lake Superior Consulting). The chemical and biological laboratories (Alpha Analytical and ESI) will submit all data and laboratory reports to E & E, who will forward them to Transco. The laboratories will retain data and reports for a period of time dictated by their individual protocols and certifications. Laboratory data and reports will also be retained by the consultants for the minimum period identified in Table 1.6-1 and Table 1.6-2.

2 DATA GENERATION AND ACQUISITION

This section of the SAP/QAPP describes the design and implementation of field, laboratory, and data-handling procedures for the Project's offshore sampling activities. The

SAP/QAPP provides the basis for ensuring that appropriate methods are used and thoroughly documented.

2.1 Sampling Process Design

2.1.1 Geotechnical Cores

Supplemental sediment core samples extending up to 125 feet below the seafloor will be collected from 13 new sample sites within New York waters. These samples will be analyzed for only the geotechnical properties presented in Table 1.4-1. The geotechnical core sample sites are identified as "B" in Table 1A in Appendix B, which presents the planned coordinates and core depths for each site. All planned geotechnical core sample sites have been reviewed and approved by the New York State Historic Preservation Office, as appropriate. These geotechnical core sites are located within and adjacent to the Raritan Bay Channel – West Reach and the Chapel Hill Channel. The geotechnical data will be used to evaluate the feasibility of using trenching or HDD techniques at deeper depths at the two channel crossings.

2.1.2 Bulk Sediment Chemistry

Supplemental sediment core samples will be collected from eight previously sampled sites in New York waters. These samples will be analyzed for the bulk chemistry parameters presented in Table 1.4-1. The bulk chemistry core sample sites are identified as "VC" in Table 1A in Appendix B, all of which have been reviewed and approved by the New York State Historic Preservation Office, as appropriate. The cores will extend up to 32 feet below the seafloor at areas near navigation channels and designated anchorage areas. The bulk chemistry core samples will be collected in areas where the pipeline would be installed by trenching methods and/or where HDD pits would be excavated, potentially using a clamshell dredge. Dredged material may be side-cast adjacent to the trench and, following pipeline installation, would be used to backfill the trench to the extent practicable.

Transco will collect samples to depths that reflect the maximum anticipated depth of disturbance during Project construction (see Table 2.1-1), accounting for appropriate pipeline burial depths. The USACE NYD provided the following anticipated pipeline burial requirements for the Project based on information presented as of June 2016 (Handell 2016):

- Navigable waters located outside of designated channels and anchorage areas:
  - Minimum 4 feet of cover below present bottom in soft sediment
  - Minimum 2 feet of cover below present bottom in consolidated rock

- Designated navigation channels:
  - Minimum burial 8 feet below authorized depth (including side-slopes), and
  - Minimum 4 feet of cover in soft sediment
- Designated (charted) anchorage grounds:
  - Minimum 7 feet of cover below present bottom in soft sediment for un-maintained anchorage
  - Minimum burial 11 feet below authorized (or actively maintained) depth for maintained (dredged) anchorage and minimum 7 feet of cover in soft sediment.

Sediment core samples will be divided into sections at the time of collection. The size of the planned core sections will provide sufficient volume to allow for QA/QC analyses.

2.1.3 Evaluation for HARS Disposal

In a letter dated September 15, 2017, Transco submitted an initial Section 103 request to USACE NYD DMMS to transport dredged materials for the purposes of disposal at the HARS. The request identified nine proposed dredge prisms (see Table 1.2-1). Based upon the resultant Sampling and Testing Requirements received from USACE NYD DMMS and EPA Region 2 on December 7, 2017, sediment core samples will be collected at 31 locations, 9 of which are located within New Jersey waters and 22 of which are in New York waters. On December 20, 2017, Transco submitted an additional Section 103 request was submitted to USACE NYD DMMS for approval of an alternative scenario for disposal of sediments for deeper dredging depths at two channel crossings (see Table 1.2-1). Based upon the approved Sampling and Testing Requirements received from USACE NYD DMMS and EPA Region 2 on January 18, 2018, additional sediment core samples will be collected at 14 locations, all of which are in New York waters. As this additional request submitted is an alternative scenario for proposed channel crossings, the additional prisms, and multiple sample locations are co-located with the original request. See Table 1.2-1 for all prism locations.

The coordinates and corresponding dredge prisms for the HARS-related core locations are identified in Table 1B in Appendix B, which have been reviewed and approved by the New Jersey Historic Preservation Office and New York State Historic Preservation Office, as appropriate. Sites identified with "-ALT" indicate those that have been shifted to avoid potential cultural resources or existing cables. The cores will extend up to 31 feet below the seafloor. The core samples will be collected in areas to be excavated using a clamshell dredge west of the New

York/New Jersey state boundary near the Ambrose Channel crossing, and proposed for disposal at the HARS.

Sediment core samples will be collected for HARS-related testing and analysis of physical, chemical (elutriate), and biological (toxicity/bioaccumulation) properties (see Table 1.4-1). In the Sampling and Testing Requirements provided by USACE NYD DMMS and EPA Region 2, the chemical analysis of sediment was waived in lieu of previous sediment testing from 2016 and 2017. Due to sample volume and analysis requirements, separate core samples will be collected at each core sample site, as outlined in Table 1B in Appendix B. Thus, a total of approximately 79 core samples will be collected for HARS-related analyses. Vibracore samples will be examined and if discernable stratification greater than 2 feet thick is observed, the stratification will be divided into separate containers at the time of collection. As recommended by the 2016 Guidance, Transco's representative will contact the USACE NYD DMMS on how to proceed if such stratification is encountered. If USACE NYD DMMS determines that the stratified material must be tested separately from the rest of the core, then additional cores may have to be collected at each location to achieve enough sample volume for analysis of each strata. The final composite for each prism must be comprised of the same number of cores from each location. The size of the planned number of cores collected will provide sufficient volume to allow for QA/QC analyses. In the event that difficulties are encountered in the field obtaining samples from the locations specified in Table 1B of Appendix B, Transco's representative will consult the USACE NYD DMMS on how to proceed.

2.2 Sampling Methods

Summaries of offshore sampling methods are provided below. Unless otherwise noted in this SAP/QAPP, sampling procedures will substantially adhere to the applicable testing methods identified in Section 2.4. Sample sites will be located in the field using a real-time kinetic differential global positioning system equipment with an accuracy of approximately ± 3 feet. Water depths will be determined using a survey-grade echo sounder with an accuracy and precision of 3 feet or better. Sediment cores and water samples for HARS-related analyses will be collected within the radii outlined in Tables 1B and 1C of Appendix B respectively.

2.2.1 Geotechnical Boring

A rotary drill system will be used to collect the sediment core samples for geotechnical analysis. Offshore boring will use a self-propelled lift boat with a land-based tracked drill rig, working on a 24-hour basis. The jack-up platform is lifted out of the water by jacking against the legs mounted on the platform. The drilling method will use a combination of split spoon, Shelby

tubes, piston sampling, and liner sampling. The proposed sampling approach, as presented in Tables 2.2-1, 2.2-2, and 2.2-3, is based upon anticipated soil conditions. The sampling method will be adjusted in the field based upon actual soil formations.

Table 2.2-1
Proposed Raritan Bay Channel Geotechnical Boring and Sample Depths

Sample Interval (ft)		Sample Type by Site				
Start	**End**	**B-20**	**B-21**	**B-22**	**B-23**	**B-24**
0	2	Tube	Tube	Tube	Tube	Tube
3	5	Tube	Tube	Tube	Tube	Tube
6	8	SS	SS	SS	SS	SS
9	11	Tube	Tube	Tube	Tube	Tube
12	14	SS	SS	SS	SS	SS
15	17	Tube	Tube	Tube	Tube	Tube
18	20	Tube	Tube	Tube	Tube	Tube
21	23	SS	Tube	SS	SS	SS
25	27	Tube	SS	SS	Tube	Tube
30	32	SS	Tube	Tube	Tube	SS
35	37	SS	SS	SS	SS	SS
40	42	Tube	Tube	Tube	Tube	Tube
45	47	SS	SS	SS	SS	SS
50	52	Tube	Tube	Tube	Tube	Tube
55	57	SS	SS	SS	SS	SS
60	62	Tube	Tube	Tube	Tube	Tube
65	67	SS	SS	SS	SS	SS
70	72	SS	SS	SS	SS	SS
75	77	SS	SS	--	SS	SS
80	82	SS	SS	--	SS	SS
85	87	SS	SS	--	SS	SS

Key:
ft = feet
Tube = Shelby Tube or Liner
SS = Split Spoon
-- = depth not sampled

Table 2.2-2
Proposed Chapel Hill Channel Geotechnical Boring and Sample Depths

Sample Interval (ft)		Sample Type by Site					
Start	**End**	**B-25**	**B-26**	**B-27**	**B-28**	**B-29**	**B-30**
0	2	SS	SS	SS	SS	SS	SS
3	5	SS	SS	SS	SS	SS	SS
6	8	SS	SS	SS	SS	SS	SS
9	11	SS	SS	SS	SS	SS	SS
12	14	SS	SS	SS	SS	SS	SS
15	17	SS	SS	SS	SS	SS	SS
18	20	SS	SS	SS	SS	SS	SS
21	23	Tube	Tube	Tube	Tube	Tube	Tube
25	27	SS	SS	SS	SS	SS	SS
30	32	SS	SS	SS	SS	SS	SS
35	37	SS	SS	SS	SS	SS	SS
40	42	Tube	Tube	Tube	Tube	Tube	Tube
45	47	SS	SS	SS	SS	SS	SS
50	52	SS	SS	SS	SS	SS	SS
55	57	SS	SS	SS	SS	SS	SS
60	62	Tube	Tube	Tube	Tube	Tube	Tube
65	67	SS	SS	SS	SS	SS	SS
70	72	SS	SS	SS	SS	SS	SS
75	77	SS	--	SS	SS	SS	SS
80	82	SS	--	--	--	SS	SS
85	87	--	--	--	--	--	SS

Key:
ft = feet
Tube = Shelby Tube or Liner
SS = Split Spoon
-- = depth not sampled

Table 2.2-3
Proposed Chapel Hill Channel Geotechnical Boring and Sample Depths

Sample Interval (ft)		Sample Type by Site		Sample Interval (ft)		Sample Type by Site
Start	**End**	**B-32**		**Start**	**End**	**B-33**
0	2	Tube		0	2	Tube
40	42	SS		30	32	SS
45	47	SS		35	37	SS
50	52	SS		40	42	SS
55	57	SS		45	47	SS
60	62	SS		50	52	SS
65	67	SS		60	62	SS
70	72	SS		70	72	SS
75	77	SS		80	82	SS
80	82	SS		--	--	--
85	87	SS		--	--	--
90	92	SS		--	--	--
95	97	SS		--	--	--
100	102	SS		--	--	--
110	112	SS		--	--	--

Key:
ft = feet
Tube = Shelby Tube or Liner
SS = Split Spoon
-- = depth not sampled

A “casingless” first push will be attempted in order to obtain samples in surface sediments, provided that geophysical studies have been performed prior to boring selection. However, due to the currents in the channel areas, a casing will most likely be needed. If a casing is required, a temporary 6-inch-diameter steel casing extending through the water column from the deck of the barge will be used to stabilize the sediments 5 to 10 feet below the seafloor (or the depth required to provide a stable setup), and samples will be collected based on Tables 2.2-1 and 2.2-2, or as directed by the onboard Transco representative(s). Drilling fluid additives will be used to stabilize the remaining depth of the borehole. The drilling fluid additives will consist primarily of attapulgite clay powder (e.g., ZeoGel) and/or barite or guar gum, as necessary, to provide additional weight/density.

Some sediment samples will be obtained with a standard 2-inch outer-diameter split-spoon sampler, which will be driven through four 6-inch intervals with a 140-pound hammer that freefalls for 30 inches. A standard penetration test will be used to determine resistance at each sample interval, and the number of blows required to drive the sampler through each of the four 6-inch increments will be recorded, up to 100 blows (i.e., point of refusal). The procedures will generally adhere to ASTM D1586, which includes a description of using a liner in the sampler. The selection of a Liner sampler is based upon the expectation that the sample will be taken in soft, cohesive sample.

In some cohesive soils, high quality (minimal sample disturbance) samples will be obtained utilizing 3-inch-diameter liner or thin-walled Shelby tubes. A liner sampler will be used until the shear strength is 300 pounds per square foot or greater; Shelby tubes will be used for the remaining interval. The Shelby tubes, when used, will be sealed using Shelby Tube Expansion Packers if the soil samples are not extruded in the field. Transco's designated field geologist or geotechnical engineer will make a field determination as to when it is appropriate to attempt a liner or Shelby tube sample. The associated procedures generally adhere to ASTM D1587.

A cone penetrometer test (CPT) will be conducted in addition to boring. No sediment samples will be collected during the CPT. Rather, the CPT unit will be lowered to the seafloor and push rods will force the cone assembly through the sediment to the desired depth. It may take several hours to conduct a CPT to a depth of up to 125 feet, depending on the substrate encountered. The CPT procedures generally adhere to ASTM D5778 and International Society for Soil Mechanics and Geotechnical Engineering (1999, corrected 2001) International Reference Test Procedure for the Cone Penetration Test.

After drilling, the casing will be raised to an elevation of approximately 1 to 3 feet below the mudline (i.e., seafloor) and seawater will be pumped in through the bottom of the drill casing until fluid collected on deck runs clear. Approximately one and a half sites will be collected per 24-hour period depending on site conditions, such as weather and type of substrate encountered.

2.2.2 Vibracoring

Bulk Sediment Chemistry

Vibracoring will be used to collect sediment core samples with a Model P Pneumatic Vibratory Corer, or equivalent. All vibracores samples will be taken from a vessel held on position under power; no seabed disturbance will occur at these sites other than taking the samples

themselves. At each sample site, vibracore samples that are collected concurrently will be spaced approximately 20 feet from each other to avoid sediment disturbance. A 20-foot-long steel core barrel with a 4-inch-diameter or a 30-foot-long steel core sampling barrel will be employed. Prior to use, this barrel will be cleaned with a washdown pump or pressure washer using site water. Following the cleaning of the core barrel, a new core liner will be inserted into the core barrel. A dedicated (single-use) rigid plastic liner will be used for collection of sediment chemistry samples. A stainless steel core catcher will then be inserted into the end of the barrel and riveted or threaded in place. The vibracorer will then be deployed into the water column. When the core nose reaches the bottom, the vibracoring unit will be turned on. The vibracorer will continue vibrating and coring until reaching the desired depth or a point of refusal (i.e., penetration of less than 6 inches over a one-minute period). The unit will be laid horizontally on the deck, the core catcher will be removed, and the liner will be pulled from the core. For sediment core samples sampled at previously established sample sites, the upper depth intervals sampled and tested in 2016 will be discarded. The remainder of the core intended for bulk sediment chemistry analyses will be cut into 3-foot (maximum) sections, which will be treated as sub-samples according to the depth intervals identified in Section 2.3. The physical features of the sediment sub-sample, such as color, texture, and odor will be noted in a field logbook. Distinct layers more than 1-foot thick will be separated as unique sub-samples subject to sediment chemistry analyses if they appear contaminated, based on the field geologist's evaluation of appearance and/or odor. The minimum 1-foot thickness will provide sufficient sample volume for all sediment chemistry analyses. Depending on site conditions such as weather and type of substrate encountered, approximately 5 to 10 vibracore samples will be collected per day.

Evaluation for HARS Disposal

Sediment for evaluation for HARS disposal will be collected similarly to the vibracoring methods described above, with the following differences:

- HARS-related sediment core samples will not be sub-sampled as described above unless discernable stratification of more than 2 feet is observed. See Section 2.3.2 for further information on sample handling.
- HARS-related sediment core samples may be collected using longer liners to ensure full recovery to the required sample depth.
- All core samples collected from each individual sample location will be processed in order to document the sediment lithology. Each core will be cut into 3 feet

sections, capped, and moved to a processing table. The core will then be split open, and the sediment lithology described, including observations of color, texture, odor, apparent stratification, and presence of contaminants, which will be recorded in the logbook. Photos of each core will be taken from directly above, without shadows. Depending on the layering observed (as described above), the sediment will be removed and placed into a labeled 5-gallon bucket. If discernable stratification is observed, additional cores may be collected pending confirmation by USACE NYD DMMS.

2.2.3 Sediment Grabs

Per the 2016 Guidance, a reference sediment sample will be collected for comparison to sediments proposed for HARS disposal. A modified Day sediment grab sampler will be used to collect samples of reference sediment from the Mud Dump Site Reference Site (40° 20' 13" N, 73° 52' 11" W). Samples collected must contain predominantly sand (>95%), with minor silt and clay (<5%), low moisture content (~23%), and negligible total organic carbon (TOC) (<0.2%) in order to be representative of the Mud Dump Reference Site. Sediments collected containing black mud, silt, coarse-grained sand, or sand with silt lenses will not be included in these analyses.

The modified Day sediment grab sampler shall be thoroughly cleaned prior to use i.e. acid and solvent rinsed or steam cleaned. The sediment grab sampler is not Teflon®-coated; therefore, reference sediments will be removed from the center of each grab to eliminate sediment that has come into contact with the sampling device.

The laboratory subcontractor will be responsible for providing the control sediments. Control sediments will be free of contaminants and compatible with the biological needs of the test organisms. Sediments collected from sites where field-collected test organisms were obtained or sediments included in with the organisms during shipping or culturing are acceptable as a control sediment, provided the sediments are sieved prior to biological testing.

2.2.4 Water Sampling

Water samples for use in HARS-related chemistry analysis, chemical elutriate analysis, and suspended particulate phase (SPP) toxicity tests will be collected using an oil-less pump such as a peristaltic. Any components within several meters of the sample intake should be non-contaminating (i.e., Teflon®, or sheathed in polypropylene or epoxy coated). Samples will be collected within 1 meter of the surface and during high slack tide. One water sample will be collected from within the boundaries of each prism and within the specific buffers of the locations

provided in Table 1C. The oil-less pump will be dropped over the side of the vessel with a rope tether. Once the pump reaches the desired depth, the system will be flushed with at least 10 times (approximately three gallons for a 50-foot length of tubing) the volume of the tubing prior to the collection of the 100-liter sample. The site water will be collected into certified chemical-free Cubitainers®. The water samples will be refrigerated in an onboard cooler to 4 degrees Celsius (°C). Upon collection of the site water, the samples will be transferred to shore and picked up by laboratory courier service.

2.2.5 Contingency Resampling Procedures

Bulk Sediment Chemistry

In the event of poor recovery, breakage of the liner, or buckling of the core barrel during vibracoring, a second (and potentially third) attempt will be made in the same target area unless the presence of a hard layer is expected to be the cause of poor recovery or core barrel buckling. If poor recovery or buckling of the core barrel is judged to be the result of encountering a hard layer, the Transco representative onboard the vessel will provide instructions on whether a second attempt will be made at the same location, an offset location, or at all.

In the event that vibracoring is aborted for whatever reason, and the Transco representative onboard the vessel confirms that an additional attempt will be made at an offset location, the sampling contractor will relocate to not more than 20 feet from the primary location, and actual coordinates will be logged.

Evaluation for HARS Disposal

In the event of poor recovery, discernable stratification, or any of the other events identified above occur, the sampling contractor will notify Transco. Transco's representative will consult the USACE NYD DMMS on how to proceed.

2.2.6 Equipment Decontamination

Bulk Sediment Chemistry

The vibracorer nose catcher assembly must be decontaminated prior to each use. Because of the direct contact with sample material, the vibracorer nose catcher assembly will be brushed clean of gross contamination, washed with non-phosphate detergent (Alconox or equivalent), rinsed with 10% nitric acid solution, and rinsed with potable water or seawater. Seawater from the site may be used to rinse the nose catcher assembly, given that it will be submerged in seawater immediately prior to sample collection.

The sample preparation implements must be decontaminated prior to each use. Implements used to process samples for bulk sediment chemistry analyses (e.g., spoons, bowls, mixing blades) will be decontaminated by the following procedure: brushed clean of gross contamination, washed with non-phosphate detergent (Alconox or equivalent), rinsed with potable water, rinsed with 10% nitric acid solution, thoroughly rinsed with deionized water, and dried.

Contaminated protective clothing (e.g., coveralls and disposable gloves) will be kept in plastic garbage bags to be isolated for separate treatment (e.g., laundering) and/or disposal at an appropriate onshore facility.

Evaluation for HARS Disposal

The vibracorer nose catcher assembly must be decontaminated prior to collecting each set of cores from each sampling location. Because of the direct contact with sample material, the vibracorer nose catcher assembly will be brushed clean of gross contamination, washed with non-phosphate detergent (Alconox or equivalent), and rinsed with potable water or seawater. Seawater from the site may be used to rinse the nose catcher assembly, given that it will be submerged in seawater immediately prior to sample collection. Between collection of cores at a given sampling location, the vibracorer nose catcher assembly will be brushed cleaned of gross contamination and rinsed with potable water or seawater.

The sample preparation implements must be decontaminated prior to each use. Implements used to process the HARS sediment samples (e.g., putty knife, saw blade) will be decontaminated by the following procedure: brushed clean of gross contamination, washed with non-phosphate detergent (Alconox or equivalent), rinsed with potable water, thoroughly rinsed with deionized water, and dried.

For water samples collected with the peristaltic pump, the system will be flushed 10 times the volume of the collection tubing prior to each sample collection. For a 50-foot length of tubing, the total volume required to be flushed is approximately three gallons of seawater.

Contaminated protective clothing (e.g., coveralls and disposable gloves) will be kept in plastic garbage bags to be isolated for separate treatment (e.g., laundering) and/or disposal at an appropriate onshore facility.

2.2.7 Investigation-Derived Waste

Cuttings, drilling fluids, and hazardous decontamination fluids (e.g., acid rinse) will be disposed by the sampling contractor in compliance with state and federal requirements.

Excess sediment retained after subsampling during vibracoring will be returned to the seafloor at the point of collection. No excess sediment will be retained on board the vessel.

2.3 Sample Handling and Custody

This section describes the general procedures that will be used from the time samples are initially logged onboard the sampling vessel to the time they are transferred to the onshore laboratories.

2.3.1 Sample Containers, Preservation, and Holding Times

Geotechnical Samples

Geotechnical boring samples extruded for geotechnical analyses will be wrapped or bagged in plastic and/or aluminum foil. Boring samples collected in Shelby tubes will be capped and retained in their tubes. All tested geotechnical samples will be retained by the sampling contractor (or designated laboratory) up to 30 days after laboratory testing is complete. All untested geotechnical samples will be retained by the sampling contractor (or designated laboratory) up to two years from the date of acquisition.

Bulk Sediment Chemistry

The volume of material, containers, and holding times required for each core sub-sample intended for sediment chemistry analyses are indicated in Table 2.3-1. Table 2.3-2 presents the corresponding volume of water, containers, and holding times for equipment blanks. The laboratory will provide the necessary containers, labels, and chain-of-custody (COC) forms sufficient to accommodate any quality control replicates and an additional 10% contingency to cover breakage, etc. The laboratory will maintain a record of the sample bottle-lot numbers that were shipped in the event of a contamination problem.

Table 2.3-1
Sediment Container and Holding Times for Non-HARS Chemical Analyses

Test Description	Type and Size of Container for each Group of Analytes	Number of Containers and Volume (per sample)	Preservative	Holding Time
Inorganics/Metals	16-oz glass jar with Teflon®-lined cap	1 - fill completely	Cool to 4±2°C	6 months
Mercury				28 days
Polycyclic Aromatic Hydrocarbons				Extract within 14 days, analyze within 40 days

Table 2.3-1
Sediment Container and Holding Times for Non-HARS Chemical Analyses

Test Description	Type and Size of Container for each Group of Analytes	Number of Containers and Volume (per sample)	Preservative	Holding Time
Pesticides				Extract within 14 days, analyze within 40 days
Polychlorinated Biphenyl Aroclors				Extract within 14 days, analyze within 40 days
Total Organic Carbon				28 days
Total Solids				7 days
Dioxin/Furans	4-oz Amber glass jar with Teflon®-lined cap	1 - fill completely	Cool to 4±2°C	Extract within 365 days, analyze within 365 days.

Key:
°C = degrees Celsius
HARS = Historic Area Remediation Site
oz = ounces

Table 2.3-2
Equipment Blank Container and Holding Times for Non-HARS Chemical Analyses

Test Description	Type and Size of Container for each Group of Analytes	Number of Containers and Volume (per sample)	Preservative	Holding Time
Inorganics/Metals	250-mL HDPE preserved to pH ≤2 with HNO_3	1	Cool to 4±2°C	6 months
Mercury				28 days
Polycyclic Aromatic Hydrocarbons	1-L Amber glass bottle with Teflon®-lined cap	6 (3 minimum, plus 3 contingency)	Cool to 4±2°C	Extract within 7 days, analyze within 40 days
Pesticides				Extract within 7 days, analyze within 40 days
Polychlorinated biphenyl Aroclors				Extract within 7 days, analyze within 40 days
Dioxin/Furans	1-L Amber glass bottle with Teflon®-lined cap	4 (2 minimum, plus 2 contingency)	Cool to 4±2°C	Extract within 365 days, analyze within 365 days.

Table 2.3-2
Equipment Blank Container and Holding Times for Non-HARS Chemical Analyses

Test Description	Type and Size of Container for each Group of Analytes	Number of Containers and Volume (per sample)	Preservative	Holding Time
Key: °C = degrees Celsius HARS = Historic Area Remediation Site HDPE = high-density polyethylene HNO_3 = nitric acid L = liter mL = milliliters				

Evaluation for HARS Disposal

A total of 30 gallons of sediment will be collected from each dredge prism for physical, chemical (elutriate), and biological (toxicity/bioaccumulation) testing. In the field, the HARS-related sediment core samples will be stored in high-density polyethylene 5-gallon buckets lined with food grade or other chemically clean liners (see Table 2.3-3). After homogenization and/or compositing of the prism cores by the laboratory, the sediment may be transferred to the containers noted in Table 2.3-4 for storage prior to analysis or for shipment to subcontracted laboratories. Table 2.3-4 provides the sediment holding time for each analysis.

A total of 25 gallons of sediment will be collected from the Mud Dump Reference Site for use as HARS reference sediment for physical and biological (toxicity/bioaccumulation) testing. In the field, the sediment grab samples will be stored in high-density polyethylene 5-gallon buckets lined with food grade or other chemically clean liners (see Table 2.3-3). After homogenization and/or compositing of the grab samples by the laboratory, the sediment may be transferred to the containers noted in Table 2.3-4 for storage prior to analysis or for shipment to subcontracted laboratories. Table 2.3-4 provides the sediment holding time for each analysis.

A total of 25 gallons of surface water will be collected from within the boundaries of each dredge prism for chemical analysis and elutriate preparation. If required, 25 gallons of surface water will also be collected from the Mud Dump Reference Site. In the field, the surface water will be collected and stored in 20-L Cubitainers® (see Table 2.3-3). The laboratory will subdivide and preserve the samples (as required) upon receipt at the laboratory. The recommended containers and holding times for surface water and elutriate samples are noted in Table 2.3-5.

Since bulk sediment chemistry analyses are not required and due to the volume of sediment to be collected, equipment blanks will not be collected from the drilling apparatus. The drilling equipment must be thoroughly cleaned per the procedures outlined in Section 2.2.6 after the collection of cores from each site. One equipment blank will be collected from the peristaltic pump after being flushed with DI water at least 10 times (approximately three gallons for a 50-foot length of tubing) the volume of the tubing. The equipment blank shall be analyzed for metals, PCBs, and pesticides by ESI, and collected in bottles as outlined in Table 2.3-3.

Table 2.3-6 presents the required tissue volume, recommended containers, and holding times for tissue samples generated from the bioaccumulation analysis. If the tissue will be shipped to an offsite laboratory, the laboratory will maintain a record of the sample bottle-lot numbers that were used in the event of a contamination problem.

Table 2.3-3
Evaluation for HARS Disposal
Field Sample Collection Containers

Material	Type of Container for each Group of Analytes		Sample Preservation
Sediment	30 gallons - HDPE 5-gallon buckets lined with food grade or other chemically clean liners		Cool, 4°C
Water	25 gallons - Bioassay-grade 20-L Cubitainers®		Cool, 4°C
Equipment Blank	Metals	2 x 250-mL HDPE bottle	Cool, 4°C, Preserved to pH < 2 with HNO_3 upon receipt at laboratory
	Mercury	2 x 125-mL glass bottle	Cool, 4°C, Preserved to pH < 2 with HCl upon receipt at laboratory
	PCBs	2 x 1-lL clear glass bottle	Cool, 4°C
	Pesticides	2 x 1-L clear glass bottle	Cool, 4°C

Key:
°C = degrees Celsius
HARS = Historic Area Remediation Site
HDPE = high-density polyethylene
L = liter
mL = milliliter
NO_3 = nitric acid

Table 2.3-4
Evaluation for HARS Disposal
Sediment Sample Aliquot Container and Holding Times for Physical and Chemical Analysis and Use in Biological Testing

Test Description[1]	Type of Container for each Group of Analytes	Sample Preservation	Holding Time
Biological Testing	170-L; HDPE bucket	Cool, 4°C[2]	8 weeks[3]
Physical Parameters	2-L; No specific container requirement	Cool, 4°C[2]	1 year
Preparation of SPP / chemical elutriate	4-L, plastic	Cool, 4°C[2]	8 weeks[4,5]
Total Organic Carbon	4 oz wide mouth amber glass (fill 75%)	Cool, 4°C[2]	28 days
		≤-20°C; Keep in dark	1 year

Notes:

[1] Sediment samples which require each listed analyses are provide in Table 2.4-4.

[2] Sediments may not be frozen prior to biological or physical testing.

[3] Sediments should be used as soon as possible to obviate the need for resampling should a test need to be repeated. Once SPP elutriate is prepared, the biological tests must be started within 24 hours.

[4] SPP must also be prepared within 14 days of site water collection

[5] If an elutriate test must be repeated at a later date due to QA issues, sediment that has been kept frozen in the dark at ≤-20°C for less than one year can be used to prepare it. In all cases, chemical elutriate must be prepared using water collected within 7 days.

Key:

°C = degrees Celsius
HARS = Historic Area Remediation Site
HDPE = high-density polyethylene
L = liter
oz = ounces
SPP = suspended particulate phase

Table 2.3-5
Evaluation for HARS Disposal
Site Surface Water and Elutriate Container and Holding Times for Chemical Analyses and Suspended Particulate Phase Preparation

Test Description	Sample Matrix	Type and Size of Container for each Group of Analytes	Sample Preservation	Holding Time
Organic Compounds (Pesticides, PCBs)	Site water, equipment blank, & elutriate[1]	1 L amber glass	Cool, 4°C	Extract within 7 days, analyze within 40 days
Total metals		250 mL rigid plastic or glass[3]	HNO_3 to pH <2	6 months
Mercury		250 mL rigid plastic or glass[3]	HNO_3 to pH <2	28 days
or				
(Method 1631e)		125 mL Teflon®	HCl to pH <2	90 days

Table 2.3-5
Evaluation for HARS Disposal
Site Surface Water and Elutriate Container and Holding Times for Chemical Analyses and Suspended Particulate Phase Preparation

Test Description	Sample Matrix	Type and Size of Container for each Group of Analytes	Sample Preservation	Holding Time
Suspended Particulate Phase	Site Water[2]	10 L, HDPE (fish) 10 L, HDPE (invertebrate) 2 L, HDPE (zooplankton)	Cool, 4°C	14 days[3]

Notes:
[1] Elutriate must be prepared within seven days of water sample collection for organic compounds and metals.
[2] Site water used to prepare 100% SPP for all tests and diluted with laboratory control / dilution water.
[3] Prepared SPP must be used in tests within 24 hours of preparation.

Key:
°C = degrees Celsius
HARS = Historic Area Remediation Site
HCl = hydrochloric acid
HDPE = high-density polyethylene
HNO3 = nitric acid
L = liter
mL = milliliters
PCB = polychlorinated biphenyl
SPP = suspended particulate phase

Table 2.3-6
Evaluation for HARS Disposal
Tissue Sample Container and Holding Times

Test Description	Type and Size of Container for each Group of Analytes	Sample Preservation	Holding Time
Organic Compounds (Pesticides, PCBs, PAHs, Dioxins, Furans)	10–50g ≈ 4 oz wide mouth amber glass with Teflon® -lined lid	≤-20°C; keep in dark	1 year
Total Metals	10–50g ≈ 4 oz wide mouth glass (amber optional) or plastic (HDPE)	≤-20°C; keep in dark optional	

Key:
°C = degrees Celsius
HARS = Historic Area Remediation Site
HDPE = high-density polyethylene
g = grams
oz = ounces
PAH = polycyclic aromatic hydrocarbon
PCB = polychlorinated biphenyl

2.3.2 Sample Handling

All geotechnical and sediment samples, and core sections will be handled in a manner that minimizes disturbance, maintains integrity of the samples, and prevents detrimental effects due to the nature of the samples (e.g., contaminants). Any material potentially considered hazardous will be packaged, marked, labeled, and shipped according to the U.S. Department of Transportation regulations in 49 CFR 171 through 177.

Geotechnical Borings

Boring samples for geotechnical analyses obtained with a Shelby tube will not be extruded on board the vessel. The unextruded samples will be sealed and capped in a manner that preserves moisture content, with a clear indication of top and bottom. The geotechnical boring samples will be labeled with company/project name, date, boring (location reference) number, sample number, sub-sample designation, and/or sample depth interval. The unextruded samples will be stored vertically according to their original orientation in the seabed and in a manner that ensures the samples cannot freeze. The sampling contractor will transport the geotechnical boring samples to shore every 24 hours, weather permitting. Samples will be stored on shore at the sampling contractor's office until the completion of the geotechnical sampling campaign, at which time samples will be transported to the laboratory.

Bulk Sediment Chemistry

Following inspection and logging of the sediment core samples intended for chemistry analyses, sediment sub-samples will be prepared for storage and transportation to the onshore laboratory. If these samples cannot be processed immediately, they will be stored in capped/sealed liners at 4°C ± 2°C until processing. Sediment from each bulk chemistry core interval will first be sampled for dioxin analysis at sites where dioxin analysis is warranted. The area of the interval that is most likely to contain dioxins (e.g., high clay/silt content) will be collected for analysis, as estimated by Transco's designated field geologist or geotechnical engineer. Each core interval will then be composited. All sediment intended for chemical analyses (other than dioxins) will be collected from this composite mixture and stored in suitable containers as noted in Table 2.3-1. Clean, dedicated implements (e.g., bowls, spoons, mixing blades) will be used to process each sub-sample for sediment chemistry analyses; stainless steel may be re-used following decontamination (see Section 2.2.6) while other materials will be considered disposable. The sediment chemistry samples will be properly labelled (including sample name, date and time of collection, project name, and preservation), recorded on the COC forms, and placed in a dedicated storage area immediately following processing and maintained at a temperature of 4°C

± 2°C. The sampling contractor will transport sediment chemistry samples to the shore every 48 hours, weather permitting.

Evaluation for HARS Disposal

Following inspection and logging of the sediment core samples intended for evaluation for HARS disposal, the sediment will be prepared for storage and transportation to the onshore laboratory. After inspection, all sediment samples will be extruded from the core liner into high-density polyethylene buckets lined with food grade or other chemically clean liners for transportation to the laboratory. At the laboratory, individual core samples (or stratum) will be homogenized and samples will be collected for grain size, TOC, and percent moisture (see Table 2.3-3 and 2.3-4 for recommended storage containers). Afterwards, all of the cores that comprise a prism will be composited into a final sediment mixture for physical, chemical, toxicity, and bioaccumulation analysis. Any native organisms, debris, or rocks greater than 1.0 millimeter will be documented in the logbook and removed from the sample using a 2.0 millimeters (or smaller) sieve (samples taken for physical analyses will not be press sieved). The sediment samples will be sieved as required for each analysis as outlined in the 2016 Guidance.

Reference sediment samples collected by grab sampler from the Mud Dump Reference Site will be visually inspected to determine whether they meet the criteria specified in Section 2.2.3. Grab samples that are acceptable for inclusion in laboratory analyses will be stored as outlined in Table 2.3-3.

All surface water samples will be collected into bioassay-grade Cubitainers®. The laboratory will sub-sample the surface water into storage containers as outlined in Table 2.3-4.

The sampling contractor will transport all HARS-related samples to the shore every 48 hours, weather permitting.

2.3.3 Sample Identification

Samples (or sub-samples) will be assigned a unique, alphanumeric sample identification number by the sampling contractor. All pertinent information associated with this sub-sample will be entered into the sample log system, e.g., client name (Transco), date/time, sample description, and initials of the person entering the data. This number will be recorded on the core tube or sample container using a permanent (indelible) marker.

Geotechnical Boring

The sample identification format for geotechnical samples will include the sampling site number and the corresponding depth interval, e.g., "B-5, 20'-22'" for the interval of the boring between 20 feet and 22 feet below the seafloor at sample site B5.

Bulk Sediment Chemistry

The sample identification format for bulk sediment chemistry analyses will consist of the site number followed by a dash (-) and the subsequent depth (D) intervals, in feet. It is anticipated that the samples will be sub-sampled in approximately 3 feet intervals. The sub-sample intervals will not exceed 3 feet. Transco will notify and consult with NYSDEC regarding any changes to the sample intervals. The letter "E" (for environmental) will follow this number. Table 2.3-7 contains the proposed list of sample identifications for the environmental cores. The final identification numbers will reflect the actual depths represented in the sub-sample, e.g., if a sub-sample is taken at a depth of 9 and 11 feet at location VC1-ALT, the final sample identification will be VC1-ALT-D9-11E. Sub-samples that are field-duplicated for QC purposes will be identified in a manner that allows for "blind" submittal to the laboratory.

Table 2.3-7
Examples of Identification Numbers for Sediment Chemistry Sub-Samples

Location	Depth Sampled to 2016, ft	Proposed Sample Depths 2018, ft	Example Name for First Sub-Sample	Example Name for Second Sub-Sample	Estimated Number of Sub-Samples
VC15	16.5	15.0 - 25.4	VC15-D15-18E	VC15-D18-21E	4
VC16	15.0	15.0 - 27.9	VC16-D15-18E	VC16-D18-21E	5
VC17	17.5	15.0 - 24.1	VC17-D15-18E	VC17-D18-21E	4
VC18	16.5	15.0 - 30.2	VC18-D15-18E	VC18-D18-21E	6
VC38	17.6	15.0 - 26.1	VC38-D15-18E	VC38-D18-21E	4
VC39	12.0	12.0 - 21.2	VC39-D12-18E	VC39-D15-18E	4
VC40	15.0	15.0 - 22.8	VC40-D15-18E	VC40-D18-21E	3
VC41-ALT	14.6	12.0 - 19.4	VC41-D12-15E	VC41-D15-18E	3
Key: ft = feet					

Evaluation for HARS Disposal

The sample identification format for sediment collected for HARS evaluation will consist of the location identification number outlined in the Sampling and Testing Requirements (see Table 1B of Appendix B for location identification numbers). The sample numbering system will consist of the site number (e.g., HC-01), followed by the core identifier (e.g., A, B, or C to

differentiate individual cores taken from the same target location), followed by a dash (-) and the subsequent depth (D) intervals, in feet, in the event that an entire core cannot fit in a single 5-gallon bucket. The final identification numbers will reflect the actual depths represented by the entire core, e.g., if the first core collected at site HC-01 extends to 6.5 feet below the seafloor, the final sample identification will be HC-01-D0-6.5. In the event that stratification greater than 2 feet is observed in a shallow core sample, the core will be sub-sampled, and the depth intervals will be denoted with an "S" instead of a "D" (i.e., HC-01A-S0-2, HC-01A-S2-5, and HC-01A-S5-6.5). Table 2.3-8 contains the proposed list of sample identification cores sub-sampled for stratification.

Table 2.3-8
Evaluation for HARS Disposal
Examples of Identification Numbers for Sub-Samples

Sample Site	Descriptor	First Sub-Sample	Second Sub-Sample	Third Sub-Sample
HE1-14	Full Core	HE1-14-D0-19.2	--	--
HE1-14	Stratification	HE1-14-S0-4	HE1-14-S4-6	HE1-14-S6-19.2
Etc.	Etc.	Etc.	Etc.	Etc.

Key:
ft = feet
HARS = Historic Area Remediation Site

For surface water samples collected for use in HARS-related chemical testing, elutriate and SPP toxicity tests, the sample numbering system will consist of the letter "H" to denote the association with the evaluation for HARS disposal, followed by the associated dredge prism, followed by a dash, then "SW", a second dash, and then the date (MMDDYY) (i.e., HC-SW-012918). The sample name will be recorded on the Cubitainers® using a permanent (indelible) marker.

2.3.4 Sample Custody

A COC form will be prepared for all samples. The COC form will contain the following information: sample identification number or borehole designation, date/time of sample collection, type of sample, sample matrix, preservative (if applicable), sample collection location (state), sample volume or core diameter and length, the analyses requested, and the number and type of sample containers.

The primary objective of COC procedures is to provide an accurate written or computerized record that can be used to trace the possession and handling of a sample from time of sampling through completion of all required analyses. A sample is in custody if it is:

- In the physical possession or view of an individual signing the COC form (or representative designated by such an individual);
- Kept in a limited-access storage area on the sampling vessel that is reserved for samples only, in a container that is locked or taped shut with a custody seal at the end of each work day;
- Placed in a locked room or taped carrying container with a custody seal; or
- Stored at the laboratory in a manner consistent with the laboratory's standard operating procedure (SOP).

The COC form will initially be signed by an individual who assisted in the collection of the samples. A copy will be retained by this person while the remaining carbon copies of the form accompany the samples to the onshore laboratory.

Evaluation for HARS Disposal

In addition to the items outlined above, the COC for sediment samples collected for evaluation for HARS disposal will also include instructions for the laboratories. For instance, if a sediment core is sub-sampled due to stratification (as outlined in Section 2.3.3), then the COC will indicate that the sub-sample containing the stratification cannot be homogenized with the remainder of the HARS-related sediment core.

2.4 Analytical Method Requirements

2.4.1 Geotechnical Boring

A standard testing program for geotechnical boring samples (i.e., boring samples) will be used to identify some or all of the geotechnical properties listed in Table 2.4-1. The assigned geotechnical analyses will be determined in the field by Transco's designated geotechnical engineer based on the field characterization of the soil.

Table 2.4-1
Potential Standard Geotechnical Analyses for Boring Sediment Samples

Test Property	ASTM Standard[3]
Soil Classification	ASTM D2487 ASTM D2488
Moisture Content	ASTM D2216
Density (unit weight)	ASTM D7263
Atterberg Limits[1]	ASTM D4318
Particle Size Distribution - sieve	ASTM D422

Table 2.4-1
Potential Standard Geotechnical Analyses for Boring Sediment Samples

Test Property	ASTM Standard[3]
Particle Size Distribution – hydrometer	ASTM D422
Hand Vane (Torvane and/or torque watch) and/or Pocket Penetrometer Tests[2]	ASTM WK27337 for pocket penetrometer
Miniature Vane Test	ASTM D4648
Remolded Miniature Vane Test	ASTM D4648
Triaxial Compression	ASTM D4767 ASTM D7181
Specific Gravity	ASTM D854
Carbonate Content (calcite equivalent)	ASTM D4373
X-ray Tubes	ASTM D4452

Notes:
(1) Test for Atterberg limits to be conducted at same elevation level as moisture contents.
(2) The ASTM standard for pocket penetrometer is under development. There is no ASTM standard for Torvane or pocket vane. However, processes and procedures to use these tools exist.
(3) Any deviations from the ASTM standard are noted in the laboratory report.

Key:
ASTM = American Society of Testing and Materials

An advanced testing program may be conducted on selected boring sub-samples to provide additional geotechnical data in support of the engineering design. The advanced geotechnical program may include one or more tests for the properties listed in Table 2.4-2.

Table 2.4-2
Potential Advanced Geotechnical Analyses for Boring Sediment Samples

Test Property	ASTM Standard
Consolidation (one-dimensional using incremental loading)	ASTM D2435
Consolidation (one-dimensional using controlled strain loading)	ASTM D4186
Static Shear Tests (consolidated-drained)	ASTM D3080
Static Shear Tests (consolidated-undrained) (for cohesive soils)	ASTM D6528[1]
Static Triaxial Compression Tests (Ko- and anisotropically consolidated-undrained and drained) with pore measurements	ASTM D4767[2] ASTM D7181[2]

Note:
(1) This still exists but was withdrawn by ASTM in 2016, as too much time had elapsed since the last update.
(2) Ko represents a subset of this ASTM standard.
(3) Any deviations from the ASTM standard are noted in the laboratory report.

Key:
ASTM = American Society of Testing and Materials

2.4.2 Bulk Sediment Chemistry

Chemical analyses to be conducted on bulk chemistry core samples are summarized in Table A in Appendix C (assuming sufficient sub-sample volume is obtained) and include metals, PAHs, pesticides, PCB Aroclors, total organic carbon, and dioxins/furans. The samples will be analyzed in accordance with the laboratory's NYSDOH ELAP certification and as outlined in the laboratory's SOPs included in Appendix D. All analyses in Appendix C will be conducted on all sediment sub-samples, with the exception of dioxin/furan analysis, which will be collected for analysis only at site VC-16 (all intervals). The analytes noted in Appendix C were selected based on chemicals of concern listed in the NYSDEC Technical and Operational Guidance Series 5.1.9 document, In-Water and Riparian Management of Sediment and Dredged Material (2004), as well as feedback from NYSDEC agency staff.

2.4.3 Evaluation for HARS Disposal

Physical analyses to be conducted on HARS-related sediment samples as required in the 2016 Guidance and the Sampling and Testing Requirements are provided in Table 2.4-3. The geotechnical testing laboratory is ISO/IEC 17025:2005 accredited and certified to perform ASTM testing for the U.S. Army Corps of Engineers.

Chemical analyses to be conducted on HARS-related surface water, elutriate, sediment, and bioassay (tissue) samples as required in the 2016 Guidance. The Sampling and Testing Requirements are provided in Table 2.4-4. The reporting limits required are provided in Appendix C. ESI may provide lower reporting limits than are required by the 2016 Guidance; however, all spiking concentration (LCS, MS/MSD/MST, SRM) shall be based on the reporting limits provided in the 2016 Guidance. The following deviations from the Guidance are noted and have been approved by USACE and EPA: Triplicate analysis of the surface water and elutriate samples is not required as long as triplicate matrix spikes (MS/MSD/MST) are performed for each analytical batch. The resulting supernatant from the elutriate preparation may be centrifuged prior to sampling in lieu of filtering with a 0.45 µm filter. The metals analysis of surface water and elutriate, the calibration solutions, laboratory control samples, and procedural blank will be prepared in a 2.0% sodium chloride solution to match the sample matrix.

The surface water and elutriate samples will be analyzed in accordance with the laboratory's DoD ELAP or National and Environmental Laboratory Accreditation Conference (NELAC) certification. Table 2.4-4 specifies which sediment sample types are included in each physical and chemical analyses listed in Tables 2.4-3 and Appendix C. Table 2.4-4 also identifies

which sediment sample types are included in each bioassay. Laboratory specific SOPs for these analyses are provided in Appendix D.

Table 2.4-3
Evaluation for HARS Disposal
Potential Standard Physical Analyses Sediment Samples

Test Property	Methods	Frequency
Soil Classification	ASTM D2487	As required
Description and Identification of Soils	ASTM D2488	As required
Total Solids / Percent Moisture	ASTM D2216	Per homogenized sediment core, reference (composited grabs), control sample, and bioassay sediment composite [1]
Atterberg Limits	ASTM D4318	Per bioassay sediment composite
Grain Size	ASTM D7928	Per homogenized sediment core, reference (composited grabs), control sample, and bioassay sediment composite [1]
Specific Gravity	ASTM D854	Per bioassay sediment composite
Bulk Density	ASTM D4531	Per bioassay sediment composite

Note:
[1] If stratification is observed, each stratum within a core must be analysed separately

Key:
ASTM = American Society of Testing and Materials
HARS = Historic Area Remediation Site

Table 2.4-4
Evaluation for HARS Disposal
Analyses Required for Sediment, Water/Elutriate, and Tissue Samples

Analysis	Core (or stratum)	Composite Dredged Material	HARS Reference Sediment	Control Sediment(s)	Water / Elutriate	Bioaccumulation Tissue
Physical Analyses[1]						
Grain Size	Yes	Yes	Yes	Yes	No	No
% Solids / Moisture	Yes	Yes	Yes	Yes	No	No
Specific Gravity	No	Yes	No	No	No	No
Bulk Density	No	Yes	No	No	No	No
Atterberg Limits	No	Yes	No	No	No	No
Biological Testing						
SPP Toxicity	No	No[2]	No	No	Yes	No
Solid Phase Toxicity	No	Yes	Yes	Yes	No	No
Bioaccumulation	No	Yes	Yes	Yes	No	No

Table 2.4-4
Evaluation for HARS Disposal
Analyses Required for Sediment, Water/Elutriate, and Tissue Samples

Analysis	Core (or stratum)	Composite Dredged Material	HARS Reference Sediment	Control Sediment(s)	Water / Elutriate	Bioaccumulation Tissue
Chemical Analyses						
Total Organic Carbons	Yes	Yes	Yes	Yes	No	No
Metals	No	No	No	No	Yes	Yes
PCBs	No	No	No	No	Yes	Yes
Pesticides	No	No	No	No	Yes	Yes
Semi-volatile Organic Compounds (including PAHs)	No	No	No	No	No	Yes
Dioxins/Furans	No	No	No	No	No	Yes

Source: USACE and EPA 2016.

Note:
(1) Physical analyses are to be conducted prior to press sieving the sample.
(2) The composite dredge material is used to create the elutriate for the SPP Toxicity test.

Key:
HARS = Historic Area Remediation Site
PAH = polycyclic aromatic hydrocarbon
PCB = polychlorinated biphenyl
SPP = suspended phase particulate

Core (or strata) samples will be homogenized and/or composited by the subcontracted laboratory for physical, chemical (elutriate), and biological (toxicity/bioaccumulation) testing. In the event that cores are sub-sampled for stratification, the contributions from each core to the composite will be proportional to the core length until a sufficient composite volume is prepared. If other organisms are still visible in sub-samples to be used for solid-phase toxicity testing, additional sieving with a 0.5 millimeter sieve will be required. A sample of each core (or stratum) will be archived by the laboratory subcontractor.

Biotoxicity and bioaccumulation tests will be conducted on HARS-related sediment samples as required in the 2016 Guidance. The analyses will be performed on the composited and homogenized dredge prism sediments along with appropriate reference and control treatments as outlined in the 2016 Guidance. These biological tests include 10-day benthic (solid phase) toxicity testing, water column SPP toxicity testing, and 28-day bioaccumulation testing.

Surface water collected from within each prism boundary will be mixed with associated sediment to prepare the SPP for biological testing and to prepare an elutriate sample for chemical analysis of dissolved contaminants of concern present in the liquid phase. Control water will be provided by the subcontracted laboratory for use as the overlying and dilution water for biological test and control treatments, and may be collected from the field or created from artificial sea salt mixtures. Control water will be the same water in which test organisms are acclimated to prior to testing. The subcontracted laboratory will be responsible for analyzing the control water on an annual basis for parameters specified in ASTM 2002a, and will be responsible for constructing and maintaining control data files from results of control treatments.

The target compound list for site water and elutriate chemical analysis are provided in Table 2.4-4. All analyte concentrations will be reported to three significant figures. Inorganic results will be reported as micrograms per liter (µg/L), and pesticides and PCB congener results will be reported as nanograms per liter (ng/L).

Tissues generated from the 28-day bioaccumulation test for each prism, reference sediment, and pre-test tissue will be analyzed for the analytes outlined in Table 2.4-4 and as outlined in the 2016 Guidance. Pre-test tissue analyses will be used to confirm that the bioaccumulation test organisms sources contain concentrations below guidance values for analytes of concern and will be performed prior to the start of the bioaccumulation test. Tissue from control organisms, and any remaining tissue from test, reference, and pre-test organisms will be archived (frozen) until six months after the announcement of test results in a USACE NYD Public Notice. Results will be reported on both a dry-weight and wet-weight basis and reported to three significant figures. Inorganic results will be reported to as micrograms per gram (µg/g), organic results (except dioxins/furans) will be reported as nanograms per gram (ng/g), and dioxins/furan results will be reported as pictograms per gram (pg/g).

2.5 Quality Control

2.5.1 Geotechnical

Field QC will not be collected on samples for geotechnical analysis. Laboratory QC are described in the appropriate ASTM testing methods.

2.5.2 Bulk Sediment Chemistry

QC data are necessary to determine precision and accuracy and to demonstrate the absence of interferences such as contamination of sample containers/glassware and reagents.

Field and laboratory QC sample frequencies for bulk chemistry sample analyses are summarized on Tables 2.5-1 and 2.5-2, respectively.

Table 2.5-1
Field Quality Control Samples for Non-HARS Chemical Analyses

QC Sample	Description	Acceptance Criteria	Corrective Action
Field Duplicate	One per matrix per 20 samples for each analysis.[1]	RPDs of 70% for sediments	Qualify related samples and assess data usability.
Field Equipment Blank	One per equipment set per day. Only equipment sets that are subject to decontamination require equipment blanks. Dedicated or disposable equipment do not require equipment blanks. The equipment blank should be collected by rinsing the decontaminated equipment (bowls and spoons) with deionized water and collecting the water into sample containers.	No positive detections >one-half the reporting limit in the equipment blank samples. Equipment blanks are not required for single-use (disposal) sample processing implements (e.g., spoons and bowls).	Qualify related samples and assess data usability. If time permits, modify equipment decontamination procedures.

Note:
(1) Field duplicates are not required for sediment archived for dioxin analysis.

Key:
RPD = relative percent difference

Table 2.5-2
Laboratory Quality Control Checks for Non-HARS Chemical Analyses

Methods	Quality Check	Frequency	Acceptance Criteria	Corrective Action
All methods	MB	One per matrix per preparation batch.	The goal is for method blanks to be free of contamination. Low-level contamination may be present but must be less than the reporting limit.	If contamination is more than the reporting limit, samples will be reanalyzed. If contaminants are present in the method blank but not in Project samples, no further action is required.

Table 2.5-2
Laboratory Quality Control Checks for Non-HARS Chemical Analyses

Methods	Quality Check	Frequency	Acceptance Criteria	Corrective Action
All methods	LCS	One per matrix per preparation batch for each analysis. The LCS must contain all target analytes of concern at the site.	The LCS recovery must be within method control limits to demonstrate acceptable method performance. Sporadic marginal failures of a few target analytes are allowed when more than five target analytes are reported.	If LCS recoveries are outside QC criteria for more than a few target analytes, recoveries are significantly low, or the compounds were detected in the samples, then corrective action should be taken according to the laboratory's standard operating procedures. Corrective action is normally to extract and reanalyze the batch if within holding times.
Organic Methods	Surrogate Spikes	All samples and QC checks.	Surrogate recoveries must be within QC criteria for method blanks and LCSs to demonstrate acceptable method performance. Surrogate recoveries that are outside QC criteria for a sample indicate a potential matrix effect.	If sample surrogates recoveries are outside of QC criteria, the laboratory will re-analyze the sample to confirm matrix effects. If confirmed, the results will reviewed along with corresponding laboratory QC results.
All methods	MS/MSD	One per matrix for each analysis batch. The spike solution must contain a broad range of the analytes of concern at the site. The overall frequency of MS/MSD on project samples must be at least one set per 20 samples. [1]	Method QC criteria or LCS criteria if not specified. MS recoveries outside the control limits applied to the LCS indicate matrix effects. QC criteria for MSD RPDs are noted for each method in Table 1.4-2.	Sample clean-up procedures may be warranted for samples with severe matrix effects.

Note:
(1) MS/MSD is not required for sediment archived for dioxin analysis.

Key:
LCS = laboratory control sample
MB = method blank
MS/MSD = matrix spike/matrix spike duplicate
QC = quality control

2.5.3 Evaluation for HARS Disposal

The acceptance criteria for chemical analysis related to HARS evaluation are described above in Tables 1.4-3 to 1.4-6. The acceptance criteria for biotoxicity and bioaccumulation testing related to HARS evaluation are described in Tables 1.4-7 to 1.4-10. Further details are provided. in the 2016 Guidance. The acceptance criteria for the equipment blank are provided in Table 2.5-3 below.

Table 2.5-3
Field Quality Control Samples for HARS Chemical Analyses

QC Sample	Description	Acceptance Criteria	Corrective Action
Field Equipment Blank	Once during the sampling event (not the first sample). One equipment blank will be collected from the peristaltic pump after being flushed with DI water at least 10 times (approximately three gallons for a 50-foot length of tubing) the volume of the tubing.	No positive detections >one-half the reporting limit in the equipment blank samples. Equipment blanks are not required for single-use (disposal) sample processing implements (e.g., new tubing at each prism).	Qualify related samples and assess data usability. If time permits, modify equipment decontamination procedures.
Key: RPD = relative percent difference			

2.6 Instrument/Equipment Testing, Inspection, and Maintenance

Offshore sampling activities will not be authorized until Transco has accepted the sampling vessel(s) as operational and ready for work.

2.6.1 Field Equipment Testing and Maintenance

All of the onboard equipment will be supplied with adequate spare parts and back-up units for ordinary maintenance and repair at sea. The sampling contractor will provide Transco with a complete inventory of equipment, back-up systems, and spare parts available on the vessel(s). The sampling contractor will identify all equipment that is critical to maintaining full sampling vessel operability and define the contingency action in the event of failure.

Deck trials will be conducted at the time of vessel mobilization to demonstrate that all sampling and processing systems are operational. The trials will include testing all vibracoring and grab sampling equipment and validating vessel positioning systems unless the sampling

contractor can suitably document functionality or equipment redundancy to Transco through other means.

During offshore activities, all lifting equipment such as cranes, booms, hoists, spreader bars, slings, etc. will be inspected at least once per shift during the sampling activities.

All above activities will be documented as appropriate and reported to Transco.

2.6.2 Analysis Instrument Maintenance

Laboratory and field instruments and equipment used for sample analyses will be serviced and maintained only by qualified personnel. Laboratory instrument maintenance procedures will be evaluated according to the laboratory's SOP to verify that there will be no impacts on analysis of Project samples due to instrument malfunction. For example, the laboratory must have duplicate instrumentation and/or maintained major equipment under service agreements with the manufacturer that require rapid response by manufacturer-approved service agents.

Field instruments owned by the sampling contractor or rented through approved suppliers will have manufacturer-approved maintenance programs. Field equipment will be checked upon receipt to verify that instruments are in good working condition and are accompanied by appropriate calibration records or certifications. On-site operation will be performed in accordance with manufacturer manuals. If any problems occur, the instrument will be recalibrated and verified. If calibration cannot be verified, the instrument will be replaced.

2.7 Instrument/Equipment Calibration and Frequency

Before initiating sampling, the sampling contractor will calibrate any vessel instrumentation according to manufacturer's instructions and record the findings in the field logbook or records. The sampling contractor will provide the following information for Transco's review and approval:

- Evidence of recent (validated) calibration of field sampling equipment and onboard testing instruments;
- Calibration methods for vessel navigation and positioning systems;
- Detailed field calibration and operating procedures for sampling equipment and onboard testing instruments (including probes); and
- Calibration verification procedures for vessel positioning systems, field sampling equipment, and onboard testing instruments.

2.8 Inspection/Acceptance of Supplies and Consumables

The sampling contractor will be responsible for inventorying and ensuring that adequate supplies are available for the offshore sampling activities before beginning sampling, including spare equipment parts and personal protection equipment. At the time of delivery, the sampling contractor or Transco's designated representative will verify that coolers/crates, sample containers, custody seals, and COCs are in good condition and sufficient in number to maintain the offshore sampling activities until the scheduled date/time for the next supply pick-up or delivery.

2.9 Non-Direct Measurements

Sampling activities will rely on some data collected by outside sources such as tidal stage and elevation information from National Oceanographic and Atmospheric Administration tidal stations. If available, the precision and accuracy of such sources will be identified and reported as appropriate.

2.10 Data Management

The sampling contractor will create electronic logs or spreadsheets for all data collected in the field and transmit that information to Transco and the appropriate consultant(s). The laboratory performing geotechnical analyses will submit the results to the sampling contractor in a format that was determined following selection of the laboratory, and such data will be incorporated into the Geotechnical Site Investigation Report by the sampling contractor or INTECSEA (and/or Lake Superior Consulting).

The subcontracted laboratory performing sediment chemistry analyses will provide an electronic data deliverable in the most current NYSDEC EQuIS format for all Project data. Data will be retained on a secure server by the consultant(s) that use this data to produce summary reports for or on behalf of Transco. Electronic data for sediment chemistry analyses will be submitted to regulatory agencies in a format to be determined in consultation with NYSDEC and the NJDEP (e.g., NYSDEC EQuIS™ format).

The subcontracted laboratory performing sediment testing for evaluation for HARS disposal will provide an electronic data deliverable in the most current NYSDEC EQuIS format for all chemical data. Non-chemical related data will be provided as spreadsheets. Data will be retained on a secure server by the consultant(s) that uses this data to produce summary reports for or on behalf of Transco.

3 ASSESSMENT AND OVERSIGHT

Individual consultants and contractors will operate under their own internal quality programs that apply to their routine technical oversight programs. Project-specific assessment and oversight procedures are discussed below.

3.1 Assessment and Response Actions

The sampling contractor will provide checklists in the Geotechnical, Bulk Sediment Chemistry, and HARS Investigation for the offshore trials and calibrations. During the offshore sampling, the sampling contractor will complete procedural checklists for sampling activities and equipment calibrations, to be observed by a Transco representative.

3.1.1 Peer Review

All contractors and consultants are required to perform an independent technical review on the sampling and analysis reports before submitting the reports to Transco and regulatory agencies. The sample contractor's lead field personnel will review summary geotechnical and/or environmental reports to ensure accurate descriptions of field sampling activities, as warranted (e.g., statements not taken directly from the sampling contractor's field report).

3.1.2 Technical Systems Assessments

The entire Project's offshore sampling and analysis team is responsible for ongoing assessment of the technical work performed by the team; identification of nonconformance with the Project objectives; and initiation, implementation, and documentation of corrective action.

During offshore activities, a Transco representative will be in the field observing sampling activities to help ensure compliance with the technical specifications of the SAP/QAPP and associated sampling and field testing methods. The Transco representative will have authority to stop work if the field operations do not comply with the specifications. The sampling vessel(s) will be subject to ongoing audits of safety and management systems by Transco. The sampling contractor will provide Transco with reasonable access to any part of the vessel, operational, or maintenance records as appropriate for the audit. Field audits will be coordinated through Transco.

The laboratories will implement a comprehensive program of internal audits to verify compliance of their systems with applicable laboratory-specific standard operating procedures and QA/QC manuals, consistent with their respective certifications (e.g., ASTM, ELAP, or NELAC). External laboratory audits will be performed by the appropriate state agency in accordance with relevant certification programs.

3.1.3 Corrective Action

If field or laboratory QA/QC audits or checks identify any deficiencies, deviations, or nonconformance events, action will be taken to correct any deficiency and minimize the possibility of recurrence. The sampling contractor or laboratory will notify Transco immediately of any potential impacts on the results for the Project.

3.2 Reports to Management

There will be regularly scheduled progress meetings between the Sampling Contractor, consultants and Transco on sampling and testing progress, results, and schedule, as appropriate. Other reports will be provided as detailed in Section 1.6.

4 DATA VALIDATION AND USABILITY

4.1 Data Review, Validation, and Verification Requirements

Data review and validation requirements for geotechnical analyses are identified in the respective ASTM testing methods.

For bulk sediment chemistry analyses, calibration summary, analytical reporting limits and target compounds and QC summary data for surrogates, method blanks, laboratory control samples (LCS), and matrix spike/ matrix spike duplicates (MS/MSD) samples will be compared with the limits listed in Tables 1.4-2, 2.5-1, and 2.5-2.

For evaluation of sediment for HARS disposal, the chemistry analyses, calibration summary, analytical reporting limits and target compounds and QC summary data for surrogates, method blanks, LCS, and MS/MSD samples will be compared with the limits provided in the 2016 Guidance.

4.2 Validation and Verification Methods

The results of the geotechnical field and laboratory analyses will be reviewed for quality by INTECSEA's (and/or Lake Superior Consulting) geotechnical engineers.

Alpha Analytical, the subcontracted analytical laboratory for bulk sediment chemistry analysis, is responsible for internal data review. The QA officer and/or manager for the laboratory will review at least 10% of the data packages. All levels of laboratory review will be fully documented and available for review by Transco if requested or if a laboratory is audited.

ESI, the subcontracted analytical laboratory for evaluation of sediment for HARS disposal, is responsible for internal data review. The QA officer and/or manager for the laboratory will review 100% of the data package prior to reporting. All levels of laboratory review will be fully

documented by the laboratory and available for review by Transco if requested or if a laboratory is audited.

After receipt of sediment chemistry and HARS testing results from the subcontracted laboratories, E & E will evaluate the data for completeness by verifying that the laboratory information matches the field information and that the following items are included in the hard copy data package:

- COC forms and sample summary forms;
- Case narrative describing any out-of-control events and summarizing analytical procedures;
- Data report forms; and
- QA/QC summary forms.

Field data such as sample identifications and sample dates will be checked against the laboratory report. Raw data files from the field and laboratory will not be reviewed unless there is a significant problem noted with the summary information. If the data package is incomplete, the laboratory will be required to provide all missing information within one working day.

The complete Alpha Analytical and ESI laboratory data packages (for both bulk sediment chemistry and evaluation for HARS disposal) will be evaluated for compliance by E & E's lead Project chemist (and/or designee), who will follow EPA Region 2 Data Validation Standard Operating Procedures. Compliance checks will include:

- Review of raw data for any apparent QC anomalies;
- Confirming that all analytical problems and corrections are reported in the case narrative and that appropriate laboratory qualifiers are added;
- If any problems are identified, review of these concerns with the laboratory, obtaining additional information if necessary, and checking all related data to determine the extent of the error; and
- Review of non-analytical data in field logs to help identify potential causes for discrepancies.

The lead Project chemist will alert the E & E Project Manager to any QC problems, obvious anomalous values, or other data discrepancies that may impact data usability. Tables summarizing samples with detectable concentrations of any analytes will be prepared for inclusion

with the Offshore Environmental Sampling and Analysis Summary Report Addendum, along with a summary of data discrepancies and any associated corrective actions taken in accordance with Sections 3.1 and 4.3.

4.3 Reconciliation with User Requirements

The laboratory QA officer and/or manager, as well as Transco's appropriate consultant (E & E or INTECSEA) will work with Transco and relevant agencies in assessing data quality.

If there are discrepancies in the geotechnical data, the laboratory tests will typically be repeated. If there is an inadequate sample to repeat the test, then a determination will be made by an INTECSEA or Transco geotechnical engineer on the validity and use of the original test results, accounting for potential inaccuracies.

The data quality assessment for bulk sediment chemistry analyses may include some or all of the following actions:

- Data that are determined to be incomplete or not usable for the Project will be identified. E & E will discuss resolution of the issue with Transco and agency technical staff and implement appropriate corrective actions (e.g., re-testing if samples are within hold times or considering other sample/analyte results as surrogate data).
- Data that are non-detect but have elevated reporting limits due to blank contamination or matrix interference will be compared with screening criteria values (if available). If reporting limits exceed the screening criteria values, then results will be handled as incomplete data as described above.
- Data that are qualified as estimated will be used for all Project decision-making. Data assessors comparing results with screening criteria will have to account for the higher level of uncertainty in their statistical analysis.

If discrepancies are identified in data results that may affect data usability following submittal of summary reports to agencies, Transco will notify such agencies and identify any proposed measures to correct or resolve resulting data gaps.

It is expected that all data collected for evaluation of sediment for HARS disposal will meet the requirements outlined in this SAP/QAPP and outlined in the 2016 Guidance. All data must be usable for the USACE NYD DMMS to determine whether sediment meet the requirements for HARS disposal.

5 REFERENCES

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Transcontinental Gas Pipe Line Company, LLC

Appendices to Offshore Sampling and Analysis Plan / Quality Assurance Project Plan Version 2.0

Northeast Supply Enhancement Project

January 2018

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Appendix A Sample Site Figures

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Kings County

Lower Bay

Richmond County

Middlesex County

Queens County

Offshore Study Area

Rockaway Delivery Lateral

25.00

35.00

35.49

20.00

New York

New Jersey

30.00

Latitude/Longitude:
N 40.523683, W 73.861886

15.00

12.00

Raritan Bay

Latitude/Longitude:
N 40.464547, W 74.264950

Middlesex County

Monmouth County

Sandy Hook Bay

Atlantic Ocean

Milepost for Offshore Study Area
Offshore Study Area (5,000-ft)
NY/NJ Boundary
Lower NY Bay Lateral
Rockaway Delivery Lateral
Maintained Navigation Channel

0 1 Miles

0 1,000 Meters

Latitude/Longitude Coordinates - GCS North American Datum 1983
Bathymetric elevation contours are at 20-foot intervals
Sources: ESRI 2015; E&E 2003 Williams 2015; NOAA ENC 2013 (Chart # 12327 and # 12326); NJDEP Digital Data Series Bathymetric Data 2006

NOTES

NOTE: All sampling will remain within the depicted study area.

SITE LOCATION

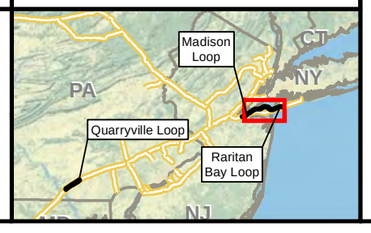
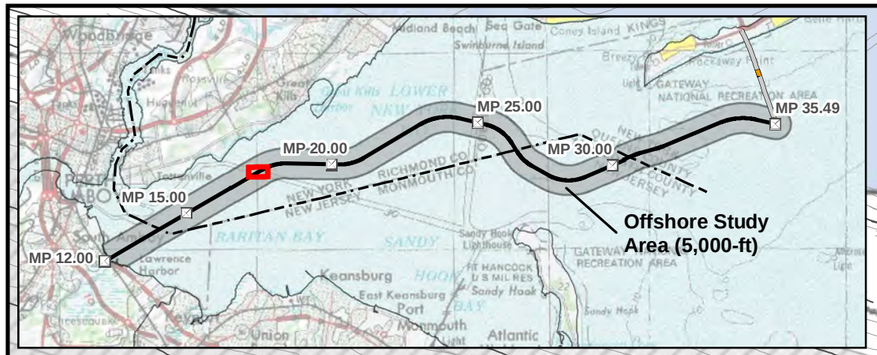


FIGURE 1
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
RARITAN BAY AND LOWER NEW YORK BAY STUDY AREA
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY, NEW YORK

Williams

Revision Date: 1/19/2018

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16

17

22

26

28

13

17

MP 17.90

MP 17.80

MP 17.70

MP 17.60

MP 17.50

MP 17.40

BHA-24

VC18

BHA-23

VC17

BHA-22

BHA-21

VC16

BHA-20

VC15

RARITAN BAY WEST REACH

G

C "25"

-20

-25

-30

-40

-45

-35

Latitude/Longitude Coordinates - GCS North American Datum 1983

Existing Sample Site

New Sample Site (Borehole)

100-ft Buffer

75-ft Buffer

Maintained Navigation Channel

Milepost for Proposed Pipeline

Proposed Raritan Bay Loop

Bathymetric Contour (5-ft interval)

Bathymetric Contour (1-ft interval)

0 100 200 300 Feet

0 50 100 Meters

Bathymetry from Rogers Surveying, PLLC. 2016

NOTES

NOTE: Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigation charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

SITE LOCATION

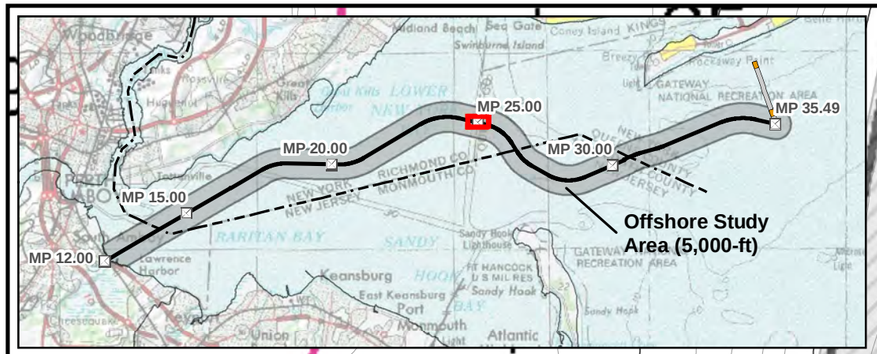


FIGURE 2
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
NON-HARS SUPPLEMENTAL SAMPLING SITES AT CHAPEL HILL CHANNEL
RARITAN BAY LOOP
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW YORK

Williams

Revision Date: 1/19/2018

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MP 24.70
BHA-25
MP 24.80
BHA-26
VC38
MP 24.90
BHA-27
VC39
MP 25.00
VC40-ALT
BHA-28
MP 25.10
BHA-29
MP 25.20
VC41-ALT
BHA-30
MP 25.30
22
24
28 Obstns
28 Obstns
28
25
17
16
24 Rks Obstn
-20
-25
-30
-35
-40

Latitude/Longitude Coordinates - GCS North American Datum 1983

Existing Sample Site
New Sample Site (Borehole)
100-ft Buffer
75-ft Buffer
Maintained Navigation Channel
Milepost for Proposed Pipeline
Proposed Raritan Bay Loop
Bathymetric Contour (5-ft interval)
Bathymetric Contour (1-ft interval)

0 100 200 300 Feet
0 50 100 Meters

Bathymetry from Rogers Surveying, PLLC. 2016

NOTES

NOTE: Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigation charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

SITE LOCATION

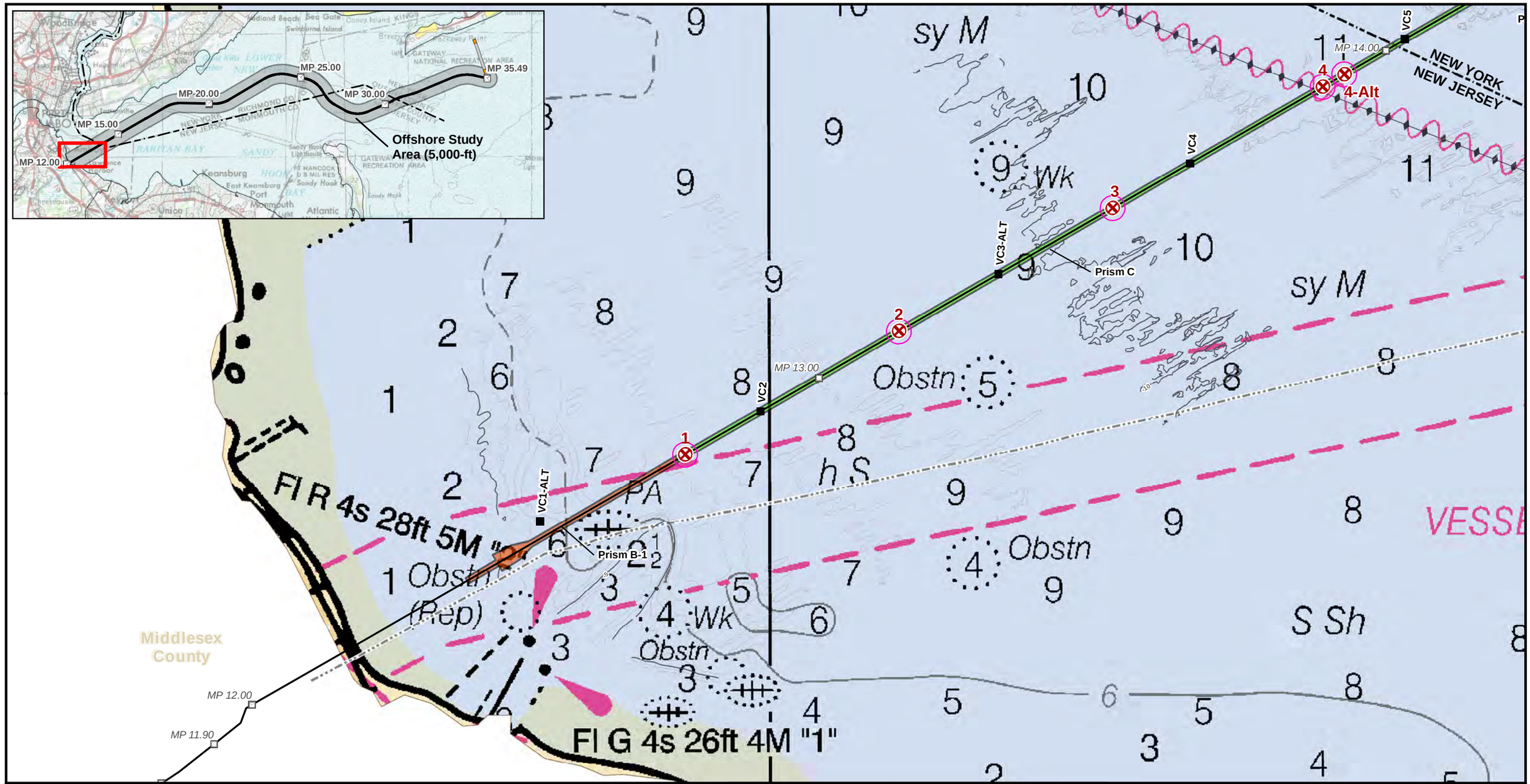


FIGURE 3
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
NON-HARS SUPPLEMENTAL SAMPLING SITES AT RARITAN CHANNEL
RARITAN BAY LOOP
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW YORK

Williams

Revision Date: 1/19/2018

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HARS Sampling Locations
100-ft Buffer
Prism B*
Prism C

Existing Sample Sites (shown for reference only)
Milepost for Proposed Pipeline
Proposed Raritan Bay Loop
Lower NY Bay Lateral
Neptune Transmission Line
NY/NJ Boundary

Bathymetric Contour (5-ft interval)
Bathymetric Contour (1-ft interval)

0 200 400 600 Feet
0 200 400 Meters

HARS Location ID	Latitude	Longitude	Prism
1	40.469943	-74.252452	C
2	40.472662	-74.246242	C
3	40.475381	-74.240034	C
4	40.478052	-74.233933	C
4-Alt	40.478325	-74.233309	C

Bathymetry from Rogers Surveying, PLLC. 2016

NOTES

Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigation charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

Latitude/Longitude Coordinates - GCS North American Datum 1983

** Prism B includes four separate segments. Between MP 24.46 and MP 24.91 (Segment B-4) only the upper 3 feet of sediment is included in Prism B.*

SITE LOCATION

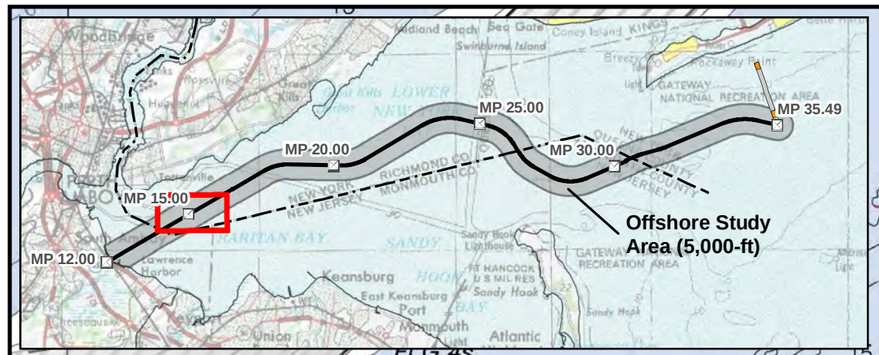


FIGURE 4
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
HARS SAMPLING LOCATIONS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY

Williams

Revision Date: 1/22/2018

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NEW YORK VESSEL TRAFFIC SERVICES AREA (see note C)

NORTH LIMIT FISH TRAP AREA

ROUND

RARITAN

BAY

New York

New Jersey

VC10

VC9

VC8

VC7

VC6

VC5

Prism A-1

Prism D

Prism B-2

Prism C

MP 16.00

MP 15.00

MP 14.00

10

8

9

7

6

5

Obstn

Wk

bk Sh

M Sh

Fl G 4s

G "1"

C "1"

HARS Sampling Location
100-ft Buffer
Prism A
Prism B*
Prism C
Prism D

Existing Sample Sites (shown for reference only)
Milepost for Proposed Pipeline
Proposed Raritan Bay Loop
NY/NJ Boundary
Maintained Navigation Channel

Bathymetric Contour (5-ft interval)
Bathymetric Contour (1-ft interval)

0 200 400 600 Feet

0 100 200 Meters

HARS Location ID	Latitude	Longitude	Prism
5	40.480316	-74.228758	C
6	40.486153	-74.215418	D
7	40.488658	-74.209692	D
8	40.491465	-74.203272	D
9	40.491671	-74.202797	A-1
10	40.493368	-74.198922	A-1

Bathymetry from Rogers Surveying, PLLC. 2016

NOTES

Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigation charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

Latitude/Longitude Coordinates - GCS North American Datum 1983

** Prism B includes four separate segments. Between MP 24.46 and MP 24.91 (Segment B-4) only the upper 3 feet of sediment is included in Prism B.*

SITE LOCATION

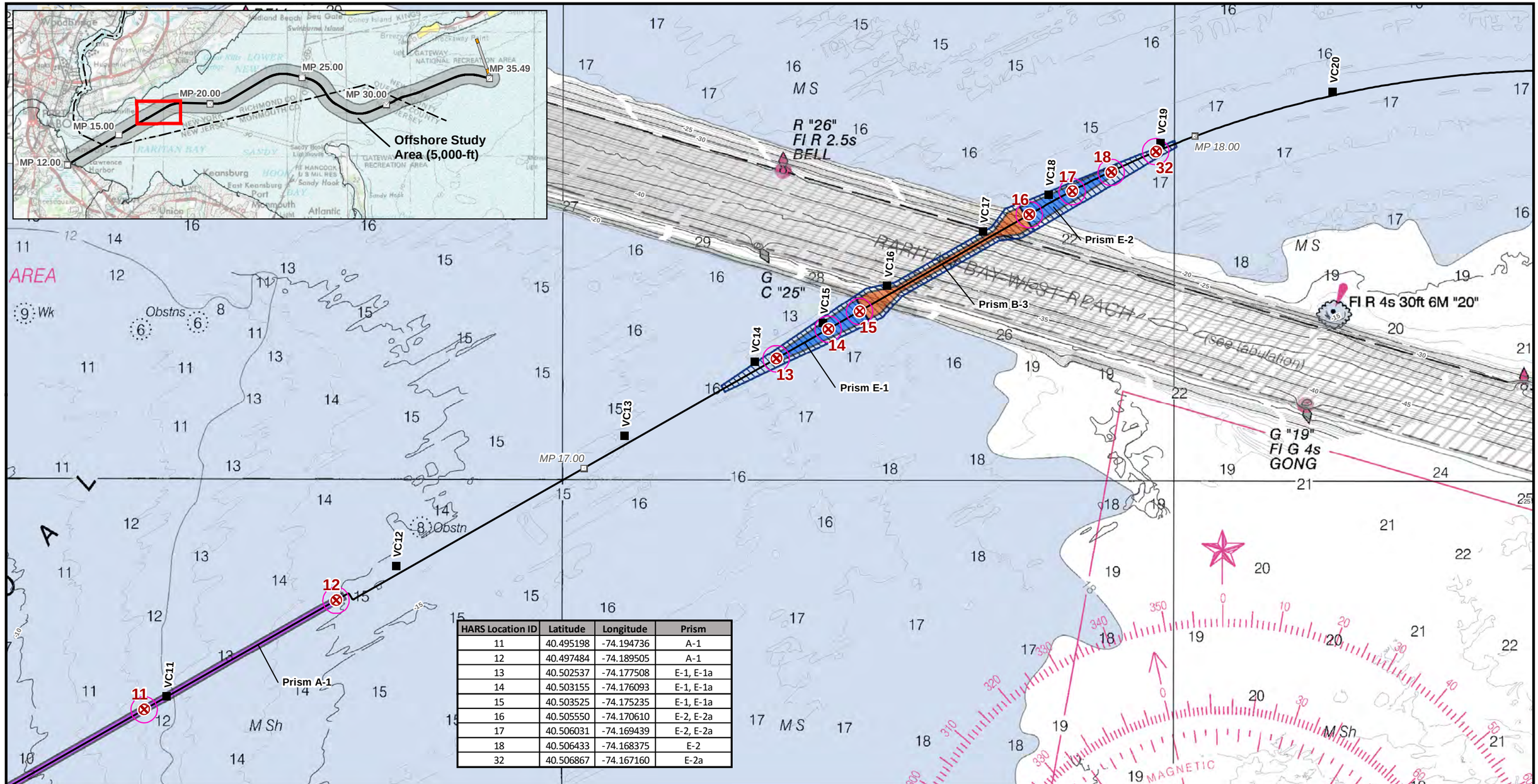


FIGURE 5
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
HARS SAMPLING LOCATIONS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW YORK

Williams

Revision Date: 1/19/2018

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HARS Location ID	Latitude	Longitude	Prism
11	40.495198	-74.194736	A-1
12	40.497484	-74.189505	A-1
13	40.502537	-74.177508	E-1, E-1a
14	40.503155	-74.176093	E-1, E-1a
15	40.503525	-74.175235	E-1, E-1a
16	40.505550	-74.170610	E-2, E-2a
17	40.506031	-74.169439	E-2, E-2a
18	40.506433	-74.168375	E-2
32	40.506867	-74.167160	E-2a

HARS Sampling Location
100-ft Buffer
Prism A
Prism B*
Prism E
Deep Crossing Prism Extent (B-3a, E-1a, E-2a)

Existing Sample Sites (shown for reference only)
Milepost for Proposed Pipeline
Proposed Raritan Bay Loop
Maintained Navigation Channel

Bathymetric Contour (5-ft interval)
Bathymetric Contour (1-ft interval)

0 200 400 600 Feet
0 100 200 Meters

Bathymetry from Rogers Surveying, PLLC. 2016

NOTES

Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigation charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

Latitude/Longitude Coordinates - GCS North American Datum 1983

** Prism B includes four separate segments. Between MP 24.46 and MP 24.91 (Segment B-4) only the upper 3 feet of sediment is included in Prism B.*

SITE LOCATION

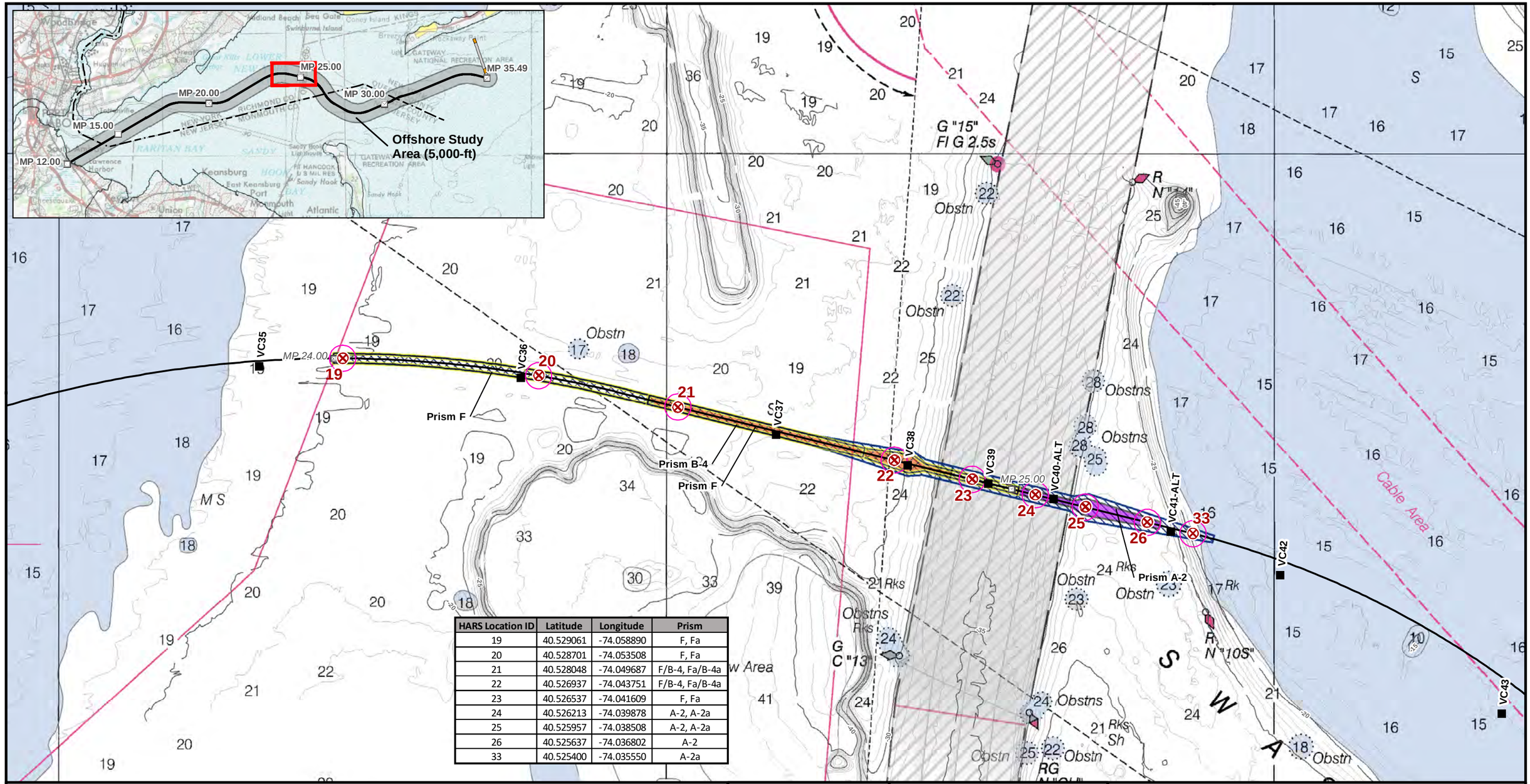


FIGURE 6
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
HARS SAMPLING LOCATIONS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW YORK

Williams

Revision Date: 1/29/2018

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HARS Location ID	Latitude	Longitude	Prism
19	40.529061	-74.058890	F, Fa
20	40.528701	-74.053508	F, Fa
21	40.528048	-74.049687	F/B-4, Fa/B-4a
22	40.526937	-74.043751	F/B-4, Fa/B-4a
23	40.526537	-74.041609	F, Fa
24	40.526213	-74.039878	A-2, A-2a
25	40.525957	-74.038508	A-2, A-2a
26	40.525637	-74.036802	A-2
33	40.525400	-74.035550	A-2a

HARS Sampling Location
100-ft Buffer
Prism A
Prism B*
Prism F
Deep Crossing Prism Extent (A-2a, B-4a, Fa)

Existing Sample Sites (shown for reference only)
Milepost for Proposed Pipeline
Proposed Raritan Bay Loop
Maintained Navigation Channel

Bathymetric Contour (5-ft interval)
Bathymetric Contour (1-ft interval)

0 200 400 600 Feet
0 100 200 Meters

Bathymetry from Rogers Surveying, PLLC. 2016

NOTES

Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigation charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

Latitude/Longitude Coordinates - GCS North American Datum 1983

** Prism B includes four separate segments. Between MP 24.46 and MP 24.91 (Segment B-4) only the upper 3 feet of sediment is included in Prism B.*

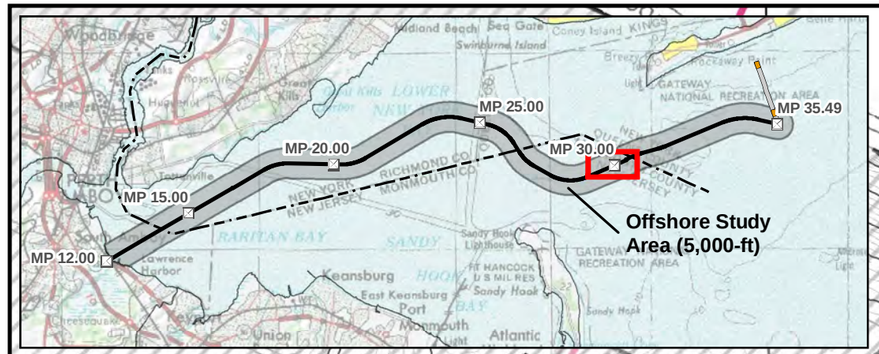
SITE LOCATION



FIGURE 7
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
HARS SAMPLING LOCATIONS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW YORK

Revision Date: 1/29/2018

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NEW YORK VESSEL TRAFFIC SERVICES AREA (see note C)

AMBROSE CHANNEL REACH B (see tabulation)

MP 30.00

VC52 VC53 VC54 VC55 VC56

NEW YORK
NEW JERSEY

27 28 29 30 31

Prism G

Prism A-3

R "8" Fl R 4s

16 Rks

HARS Sampling Locations
100-ft Buffer
Prism A
Prism G
Maintained Navigation Channel

Existing Sample Sites (shown for reference only)
Milepost for Proposed Pipeline
Proposed Raritan Bay Loop
Lower NY Bay Lateral
NY/NJ Boundary

Bathymetric Contour (5-ft interval)
Bathymetric Contour (1-ft interval)

0 150 300 450 Feet
0 100 200 Meters

HARS Location ID	Latitude	Longitude	Prism
27	40.503435	-73.968270	G
28	40.503587	-73.967382	G
29	40.508026	-73.954367	A-3
30	40.507626	-73.954128	A-3
31	40.508083	-73.953500	A-3

Bathymetry from Rogers Surveying, PLLC. 2016

NOTES

Soundings are in feet Mean Lower Low Water (MLLW) as depicted on NOAA navigation charts. MLLW = -0.20 feet MLW at the nearest NOAA tidal station (Sandy Hook, NJ).

Latitude/Longitude Coordinates - GCS North American Datum 1983

SITE LOCATION



FIGURE 8
TRANSCONTINENTAL GAS PIPE LINE COMPANY LLC
HARS SAMPLING LOCATIONS
NORTHEAST SUPPLY ENHANCEMENT PROJECT
NEW JERSEY

Williams

Revision Date: 1/19/2018

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Appendix B Sample Site Coordinate Tables

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Table 1A: Sediment Sample Sites in New York Waters (Non-HARS)[a]

Sample Site [b,c,d]	Latitude	Longitude	Coring Depth Range (ft)	Sample Site [b,c,d]	Latitude	Longitude	Coring Depth Range (ft)
B-20	40.503400	-74.176250	0 - 87.0	VC15	40.503286	-74.176237	15.0 - 25.4
B-21	40.504109	-74.174607	0 - 87.0	VC16	40.504057	-74.174471	15.0 - 27.9
B-22	40.504819	-74.172964	0 - 72.0	VC17	40.505196	-74.171867	15.0 - 24.1
B-23	40.505528	-74.171320	0 - 87.0	VC18	40.505968	-74.170079	15.0 - 30.2
B-24	40.506237	-74.169679	0 - 87.0	VC38	40.526834	-74.043371	15.0 - 26.1
B-25	40.527385	-74.044643	0 - 82.0	VC39	40.526449	-74.041158	12.0 - 21.2
B-26	40.527015	-74.042666	0 - 72.0	VC40	40.526125	-74.039373	15.0 - 22.8
B-27	40.526643	-74.040677	0 - 77.0	VC41-ALT	40.525453	-74.036149	12.0 - 19.4
B-28	40.526271	-74.038690	0 - 77.0				
B-29	40.525899	-74.036699	0 - 82.0				
B-30	40.525503	-74.034715	0 - 87.0				
B-32	40.503335	-74.175054	0 - 112.0				
B-33	40.505172	-74.172038	0 - 82.0				

Reference datum: NAD 1983.

Notes:

(a) Sample Sites as of February 2, 2018.

(b) Sample Sites labeled "B" indicate where boring sampling will occur. Sample Sites labeled "VC" indicate where vibracore sampling will occur.

(c) Sample Sites labeled "-ALT" indicate shifts that occurred based on a Phase I archaeological survey.

Key:

ft = feet

Table 1B: Sediment Sample Sites for Evaluation for HARS Disposal[a]

Sample Site [b]	Location [c]	Dredge Prism[d]	Latitude	Longitude	Coring Depth	Number of Cores Collected	State	Maximum Sampling Offset (ft)[h]
HC-01	1	C	40.469943	-74.252452	7.5	2	NJ	25
HC-02	2	C	40.472662	-74.246242	7.5	2	NJ	25
HC-03	3	C	40.475381	-74.240034	7.5	2	NJ	25
HC-04	4-ALT	C	40.478325	-74.233309	7.5	2	NJ	25
HC-05	5	C	40.480316	-74.228758	7.5	2	NY	25
HD-06	6	D	40.486153	-74.215418	7.5	3	NY	25
HD-07	7	D	40.488658	-74.209692	7.5	3	NY	25
HD-08	8	D	40.491465	-74.203272	7.5	3	NY	25
HA1-09	9	A1	40.491671	-74.202797	7.5	2	NY	25

Table 1B: Sediment Sample Sites for Evaluation for HARS Disposal[a]

Sample Site [b]	Location [c]	Dredge Prism[d]	Latitude	Longitude	Coring Depth	Number of Cores Collected	State	Maximum Sampling Offset (ft)[h]
HA1-10	10	A1	40.493368	-74.198922	7.5	2	NY	25
HA1-11	11	A1	40.495198	-74.194736	7.5	2	NY	25
HA1-12	12	A1	40.497484	-74.189505	7.5	2	NY	25
HE1-13	13	E1	40.502537	-74.177508	8.2	1	NY	28
HE1-14	14	E1	40.503155	-74.176093	19.2	1	NY	63
HE1-15	15	E1	40.503525	-74.175235	25.7	1	NY	85
HE2-16	16	E2	40.505550	-74.170610	25.4	2	NY	78
HE2-17	17	E2	40.506031	-74.169439	14.5	2	NY	48
HE2-18	18	E2	40.506433	-74.168375	9.3	2	NY	26
HF-19	19	F	40.529061	-74.058890	10.5	2	NY	36
HF-20	20	F	40.528701	-74.053508	10.5	2	NY	36
HF-21	21	F	40.528048	-74.049687	6.5[e]	2	NY	34
HF-22	22	F	40.526937	-74.043751	9.2[f]	2	NY	57
HF-23	23	F	40.526537	-74.041609	7.8	2	NY	25
HA2-24	24	A2	40.526213	-74.039878	8.5	2	NY	26
HA2-25	25	A2	40.525957	-74.038508	12.6	2	NY	58
HA2-26	26	A2	40.525637	-74.036802	8.5	2	NY	31
HG-27	27	G	40.503435	-73.968270	17.3	2	NJ	43
HG-28	28	G	40.503587	-73.967382	11.0	2	NJ	31
HA3-29	29	A3	40.508026	-73.954367	13.0	2	NJ	37
HA3-30	30	A3	40.507626	-73.954128	11.0	2	NJ	39
HA3-31	31	A3	40.508083	-73.953500	21.5	2	NJ	69
HE1a-13a	13a	E1a	40.502537	-74.177508	19.3	1	NY	61
HE1a-14a	14a	E1a	40.503155	-74.176093	30.3	1	NY	95
HE1a-15a	15a	E1a	40.503525	-74.175235	36.7	1	NY	100
HE2a-16a	16a	E2a	40.505550	-74.170610	36.4	1	NY	100
HE2a-17a	17a	E2a	40.506031	-74.169439	28.4	1	NY	68
HE2a-32	32	E2a	40.506867	-74.167160	10.3	1	NY	25
HFa-19a	19a	Fa	40.529061	-74.058890	10.5	1	NY	36
HFa-20a	20a	Fa	40.528701	-74.053508	10.5	1	NY	36
HFa-21a	21a	Fa	40.528048	-74.049687	6.5[d]	1	NY	34
HFa-22a	22a	Fa	40.526937	-74.043751	14.4[g]	1	NY	85

Table 1B: Sediment Sample Sites for Evaluation for HARS Disposal[a]

Sample Site [b]	Location [c]	Dredge Prism[d]	Latitude	Longitude	Coring Depth	Number of Cores Collected	State	Maximum Sampling Offset (ft)[h]
HFa-23a	23a	Fa	40.526537	-74.041609	18.7	1	NY	59
HA2a-24a	24a	A2a	40.526213	-74.039878	19.3	2	NY	59
HA2a-25a	25a	A2a	40.525957	-74.038508	22.5	2	NY	94
HA2a-33	33	A2a	40.525400	-74.03555	10.2	2	NY	36

Reference datum: NAD 1983

Notes:

(a) Sample Sites as of January 26, 2018.

(b) Sample Sites labeled "-ALT" indicate shifts that occurred based on utility considerations.

(c) Locations correspond with those outlined in the USACE and EPA Sampling and Testing Requirements for NAN-2016-00908-EHA. An "a" has been added to each location used in overlapping dredge prisms.

(d) An "a" has been added to dredge prisms included in the second USACE and EPA Sampling and Testing Requirements for NAN-2016-00908-EHA, received January 18, 2018.

(e) From 4'-10.5' (under Prism B4)

(f) From 4'-13.2' (under Prism B4)

(g) From 4'-18.4' (under Prism B-4)

(h) Maximum approved distance based on either the nearest prism edge or the Phase I archaeological survey.

Key:

HARS = Historic Area Remediation Site
NJ = New Jersey
NY = New York

Table 1C: Water Sample Sites for Evaluation for HARS Disposal[a]

Sample Site [b]	Location [c]	Dredge Prism	Latitude	Longitude	Maximum Sampling Offset (ft)[d]	State
HC-03	3	C	40.475381	-74.240034	25	NJ
HD-07	7	D	40.488658	-74.209692	25	NY
HA1-11	11	A1	40.495198	-74.194736	25	NY
HE1-15	15	E1	40.503525	-74.175235	85	NY
HE2-16	16	E2	40.505550	-74.170610	78	NY
HF-22	22	F	40.526937	-74.043751	57	NY
HA2-25	25	A2	40.525957	-74.038508	58	NY
HG-27	27	G	40.503435	-73.968270	43	NJ
HA3-31	31	A3	40.508083	-73.953500	69	NJ
HE1a-15	15	E1a	40.503525	-74.175235	117	NY
HE2a-16	16	E2a	40.505550	-74.170610	107	NY
HFa-22	22	Fa	40.526937	-74.043751	85	NY
HA2a-25	25	A2a	40.525957	-74.038508	94	NY

Table 1C: Water Sample Sites for Evaluation for HARS Disposal[a]

Sample Site [b]	Location [c]	Dredge Prism	Latitude	Longitude	Maximum Sampling Offset (ft)[d]	State

Reference datum: NAD 1983

Notes:

(a) Sample Sites as of January 18, 2018.

(b) Sample Sites labeled "-ALT" indicate shifts that occurred based on a Phase I archaeological survey.

(c) Locations correspond with those outlined in the USACE and EPA Sampling and Testing Requirements for NAN-2016-00908-EHA

(d) Maximum approved distance based on either the nearest prism

Key:

ft = feet
HARS = Historic Area Remediation Site
NJ = New Jersey
NY = New York

Appendix C Sediment Chemistry Test Tables

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Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Table A: Sediment Chemistry Tests for New York								
Vibracore Site Numbers [Total of 8]: VC15 through VC18; AND VC38 through VC41-ALT								
Test Analyte	**EPA Method**	**NYSDEC Required Method Detection Limits (mg/kg, ppm)**	**NJDEP or USACE Required Reporting Limits[1] (mg/kg, ppm)**	**NYSDEC No Appreciable Contamination Threshold Values[2] (mg/kg, ppm)**	**NJDEP Low Effects Range[3] (mg/kg, ppm)**	**Listed in NYSDEC TOGs 5.1.9**	**Listed in USACE Reg2 Guidance**	**Listed in NJDEP 1997 Manual**
METALS								
Arsenic	EPA 6020A	3^	1	<14 (<8.2)	8.2	X	X	X
Aluminum	EPA 6020A	N/A	40	N/A	18000*			X
Antimony	EPA 6020A	N/A	12	N/A	9.3*			X
Barium	EPA 6020A	N/A	40	N/A	48*			X
Beryllium	EPA 6020A	N/A	1	N/A	N/A			X
Cadmium	EPA 6020A	1	1	<1.2 (<1.2)	1.2	X	X	X
Calcium	EPA 6020A	N/A	1,000	N/A	N/A			X
Chromium	EPA 6020A	N/A	1	(<81)	81		X	X
Cobalt	EPA 6020A	N/A	10	N/A	10*			X
Copper	EPA 6020A	5^	1	<33 (<34)	34	X	X	X
Iron	EPA 6020A	N/A	20	N/A	N/A			X
Lead	EPA 6020A	2^	0.6	<33 (<47)	47	X	X	X
Magnesium	EPA 6020A	N/A	1,000	N/A	N/A			X
Manganese	EPA 6020A	N/A	3	N/A	260*			X
Mercury	EPA 7474	0.2^	0.1	<0.17 (<0.15)	0.15	X	X	X
Molybdenum	EPA 6020A	N/A	N/A	N/A	N/A			
Nickel	EPA 6020A	N/A	1	(<21)	21		X	X
Potassium	EPA 6020A	N/A	1,000	N/A	N/A			X
Selenium	EPA 6020A	N/A	1	N/A	1*			X
Silver	EPA 6020A	N/A	1	(<1.0)	1		X	X
Sodium	EPA 6020A	N/A	1,000	N/A	N/A			X
Strontium	EPA 6020A	N/A	N/A	N/A	N/A			
Thallium	EPA 6020A	N/A	2	N/A	N/A			X
Tin	EPA 6020A	N/A	N/A	N/A	N/A			
Titanium	EPA 6020A	N/A	N/A	N/A	N/A			
Vanadium	EPA 6020A	N/A	10	N/A	57*			X
Zinc	EPA 6020A	N/A	4	(<150)	150			X

Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Test Analyte	EPA Method	NYSDEC Required Method Detection Limits (mg/kg, ppm)	NJDEP or USACE Required Reporting Limits[1] (mg/kg, ppm)	NYSDEC No Appreciable Contamination Threshold Values[2] (mg/kg, ppm)	NJDEP Low Effects Range[3] (mg/kg, ppm)	Listed in NYSDEC TOGs 5.1.9	Listed in USACE Reg2 Guidance	Listed in NJDEP 1997 Manual
PCB AROCLORS (Aroclor list from 1997 NJDEP manual applied for NY sum.)								
Aroclor-1016	EPA 8082A	N/A	0.033	N/A	0.007			(X)
Aroclor-1221	EPA 8082A	N/A	0.067	N/A	N/A			(X)
Aroclor-1232	EPA 8082A	N/A	0.033	N/A	N/A			(X)
Aroclor-1242	EPA 8082A	N/A	0.033	N/A	N/A			(X)
Aroclor-1248	EPA 8082A	N/A	0.033	N/A	0.03			(X)
Aroclor-1254	EPA 8082A	N/A	0.033	N/A	0.06			(X)
Aroclor-1260	EPA 8082A	N/A	0.033	N/A	0.005			(X)
PCBs (sum of aroclors)	EPA 8082A	0.033	<0.1	0.023 (<0.10)	0.023	X		
DIOXINS AND FURANS								
2,3,7,8 -TCDD	EPA 1613B	N/A	0.000001	(<0.0000005)	N/A	X	X	X
1,2,3,7,8-PeCDD	EPA 1613B	N/A	0.0000025	N/A	N/A	X	X	X
1,2,3,4,7,8-HxCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,6,7,8-HxCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,7,8,9-HxCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,4,6,7,8-HpCDD	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
OCDD	EPA 1613B	N/A	0.00001	N/A	N/A	X	X	X
2,3,7,8-TCDF	EPA 1613B	N/A	0.000001	N/A	N/A	X	X	X
1,2,3,7,8-PeCDF	EPA 1613B	N/A	0.0000025	N/A	N/A	X	X	X
2,3,4,7,8-PeCDF	EPA 1613B	N/A	0.0000025	N/A	N/A	X	X	X
1,2,3,4,7,8-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,6,7,8-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
2,3,4,6,7,8-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,7,8,9-HxCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,4,6,7,8-HpCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
1,2,3,4,7,8,9-HpCDF	EPA 1613B	N/A	0.000005	N/A	N/A	X	X	X
OCDF	EPA 1613B	N/A	0.00001	N/A	N/A	X	X	X
Dioxins/Furans - Total Toxicity Equivalency Factor	EPA 1613B	0.000002	N/A	<0.0000045	0.00000365	X	X	X

Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Test Analyte	EPA Method	NYSDEC Required Method Detection Limits (mg/kg, ppm)	NJDEP or USACE Required Reporting Limits[1] (mg/kg, ppm)	NYSDEC No Appreciable Contamination Threshold Values[2] (mg/kg, ppm)	NJDEP Low Effects Range[3] (mg/kg, ppm)	Listed in NYSDEC TOGs 5.1.9	Listed in USACE Reg2 Guidance	Listed in NJDEP 1997 Manual
PESTICIDES								
alpha-BHC	EPA 8081B	N/A	0.0019	N/A	N/A			X
beta-BHC	EPA 8081B	N/A	0.0033	N/A	N/A			X
delta-BHC	EPA 8081B	N/A	0.0017	N/A	N/A			X
gamma-BHC (Lindane)	EPA 8081B	N/A	0.002	(<0.001)	N/A			X
Heptachlor	EPA 8081B	N/A	0.001	(<0.071)	0.0003*		X	X
Aldrin	EPA 8081B	N/A	0.001	N/A	0.002		X	X
Heptachlor epoxide	EPA 8081B	N/A	0.001	(<0.015)	0.005		X	X
Endosulfan I	EPA 8081B	N/A	0.001	(<0.0001)	N/A		X	X
Endrin	EPA 8081B	N/A	0.0036	(<0.006)	0.003			X
Endosulfan II	EPA 8081B	N/A	0.001	N/A	N/A		X	X
Endosulfan sulfate	EPA 8081B	N/A	0.001	N/A	N/A		X	X
Methoxychlor	EPA 8081B	N/A	0.017	(<0.059)	N/A			X
Endrin ketone	EPA 8081B	N/A	0.0033	N/A	N/A			X
Endrin aldehyde	EPA 8081B	N/A	0.0033	N/A	N/A			X
alpha-Chlordane	EPA 8081B	N/A	0.001	N/A	N/A		X	X
gamma-Chlordane	EPA 8081B	N/A	0.0017	N/A	N/A			X
Toxaphene	EPA 8081B	N/A	0.17	(<0.054)	N/A			X
Trans nonachlor	EPA 8081B	N/A	0.001	N/A	N/A		X	X
4,4'-DDE	EPA 8081B	N/A	0.001	N/A	0.0022		X	X
2,4'-DDE	EPA 8081B	N/A	0.001	N/A	N/A		X	
4,4'-DDD	EPA 8081B	N/A	0.001	N/A	0.002		X	X
2,4'-DDD	EPA 8081B	N/A	0.001	N/A	N/A		X	
4,4'-DDT	EPA 8081B	N/A	0.001	N/A	0.001		X	X
2,4'-DDT	EPA 8081B	N/A	0.001	N/A	N/A		X	
Sum of DDT+DDE+DDD	EPA 8081B	0.0033	N/A	<0.003 (<0.044)	0.0016	X	X	X
Mirex	EPA 8081B	0.189	N/A	<0.0014 (<0.12)	0.007	X	X	X
Chlordane (Total/Technical)	EPA 8081B	0.0017	N/A	<0.003 (<0.063)	0.007	X	X	X
Dieldrin	EPA 8081B	0.0033	0.001	<0.11 (<0.006)	0.002	X	X	X

Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Test Analyte	EPA Method	NYSDEC Required Method Detection Limits (mg/kg, ppm)	NJDEP or USACE Required Reporting Limits[1] (mg/kg, ppm)	NYSDEC No Appreciable Contamination Threshold Values[2] (mg/kg, ppm)	NJDEP Low Effects Range[3] (mg/kg, ppm)	Listed in NYSDEC TOGs 5.1.9	Listed in USACE Reg2 Guidance	Listed in NJDEP 1997 Manual
PAHs								
Acenaphthene (LMW)	EPA 8270D	N/A	0.1	N/A	0.016	X	X	X
Acenaphthylene (LMW)	EPA 8270D	N/A	0.1	N/A	0.044	X	X	X
Anthracene (LMW)	EPA 8270D	N/A	0.1	N/A	0.085	X	X	X
Benz(a)anthracene (HMW)	EPA 8270D	N/A	0.1	N/A	0.261	X	X	X
Benzo(b)fluoranthene (HMW)	EPA 8270D	N/A	0.1	N/A	1.8*	X	X	X
Benzo(k)fluoranthene (HMW)	EPA 8270D	N/A	0.1	N/A	0.24	X	X	X
Benzo(g,h,i)perylene (HMW)	EPA 8270D	N/A	0.1	N/A	0.17	X	X	X
Benzo(a)pyrene (HMW)	EPA 8270D	N/A	0.1	N/A	0.43	X	X	X
Chrysene (HMW)	EPA 8270D	N/A	0.1	N/A	0.384	X	X	X
Dibenz(a,h)anthracene (HMW)	EPA 8270D	N/A	0.1	N/A	0.063	X	X	X
Fluoranthene (HMW)	EPA 8270D	N/A	0.1	N/A	0.6	X	X	X
Fluorene (LMW)	EPA 8270D	N/A	0.1	N/A	0.019	X	X	X
Indeno(1,2,3-c,d)-pyrene (HMW)	EPA 8270D	N/A	0.1	N/A	0.2	X	X	X
Naphthalene (LMW)	EPA 8270D	N/A	0.1	N/A	0.16	X	X	X
Phenanthrene (LMW)	EPA 8270D	N/A	0.1	N/A	0.24	X	X	X
Pyrene (HMW)	EPA 8270D	N/A	0.1	N/A	0.665	X	X	X
Total PAH (Sum of Target Compound List above)	EPA 8270D	0.33	N/A	<4 (<4)	4	X		X
Sum of Low-Molecular Weight (LMW) PAHs	EPA 8270D	N/A	0.1	N/A	N/A		X	
Sum of High-Molecular Weight (HMW) PAHs	EPA 8270D	N/A	0.1	N/A	N/A		X	
MISCELLANEOUS								
Total Organic Carbon (TOC)	EPA 9060A	Not established	Not established	N/A	N/A	X	X	X

KEY:

N/A = Not available

1. Unshaded cells present reporting limits from USACE/EPA *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal* (2014). Shaded Cells present reporting limits from *The Management*

2. Primary threshold value from NYS TOGS 5.1.9 (2004). Value in parathesis from NYS *Screening and Assessment of Contaminated Sediment* (2014).

3. NJDEP Ecological Screening Criteria (2009). Values denoted with * are Effects Range Medium.

^Use the laboratory reporting limit if the value is less than the method detection limit shown in the table.

Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Table B. Sediment Chemistry Tests for HARS Analysis			
Test Analyte	**USACE/EPA Required Reporting Limits[1]**		
	Tissue Reporting Limit (wet)	**Water Reporting Limit**	**Sediment Reporting Limit (dry)**
METALS	**(µg/g)**	**(µg/L)**	**(µg/g)**
Arsenic	4.2	N/R	1
Cadmium	0.10	14	1
Chromium	3.9	350	1
Copper	3.2	1.0	1
Lead	0.43	70	1
Mercury	0.067	0.70	1
Nickel	1.3	25	1
Silver	0.47	0.80	1
Zinc	510	32	1
PCB Congeners	**(ng/g)**	**(µg/L)**	**(ng/g)**
PCB 8	0.4	0.002	1
PCB 18	0.4	0.002	1
PCB 28	0.4	0.002	1
PCB 44	0.4	0.002	1
PCB 49	0.4	0.002	1
PCB 52	0.4	0.002	1
PCB 66	0.4	0.002	1
PCB 87	0.4	0.002	1
PCB 101	0.4	0.002	1
PCB 105	0.4	0.002	1
PCB 118	0.4	0.002	1
PCB 128	0.4	0.002	1
PCB 138	0.4	0.002	1
PCB 153	0.4	0.002	1
PCB 170	0.4	0.002	1
PCB 180	0.4	0.002	1
PCB 183	0.4	0.002	1
PCB 184	0.4	0.002	1
PCB 187	0.4	0.002	1
PCB 195	0.4	0.002	1
PCB 206	0.4	0.002	1
PCB 209	0.4	0.002	1

Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Test Analyte	USACE/EPA Required Reporting Limits[1]		
	Tissue Reporting Limit (wet)	Water Reporting Limit	Sediment Reporting Limit (dry)
DIOXINS AND FURANS	**(pg/g)**	**(µg/L)**	**(pg/g)**
2,3,7,8 -TCDD	0.33	N/R	1
1,2,3,7,8-PeCDD	1	N/R	2.5
1,2,3,4,7,8-HxCDD	1	N/R	5
1,2,3,6,7,8-HxCDD	1	N/R	5
1,2,3,7,8,9-HxCDD	1	N/R	5
1,2,3,4,6,7,8-HpCDD	10	N/R	5
OCDD	100	N/R	10
2,3,7,8-TCDF	1	N/R	1
1,2,3,7,8-PeCDF	1	N/R	2.5
2,3,4,7,8-PeCDF	1	N/R	2.5
1,2,3,4,7,8-HxCDF	1	N/R	5
1,2,3,6,7,8-HxCDF	1	N/R	5
2,3,4,6,7,8-HxCDF	1	N/R	5
1,2,3,7,8,9-HxCDF	1	N/R	5
1,2,3,4,6,7,8-HpCDF	10	N/R	5
1,2,3,4,7,8,9-HpCDF	10	N/R	5
OCDF	100	N/R	10
PESTICIDES	**(ng/g)**	**(µg/L)**	**(ng/g)**
Heptachlor	2.7	0.010	1
Aldrin	4.3	0.4	1
Heptachlor epoxide	2.7	0.010	1
Endosulfan I	0.32	0.010	1
Endosulfan II	0.32	0.010	1
Endosulfan sulfate	0.32	0.010	1
alpha-Chlordane	2.7	0.030	1
Trans nonachlor	2.7	0.010	1
4,4'-DDE	1	4.0	1
2,4'-DDE	1	0.010	1
4,4'-DDD	1	0.010	1
2,4'-DDD	1	0.010	1
4,4'-DDT	1	0.04	1
2,4'-DDT	1	0.010	1
Dieldrin	1.5	0.20	1

Table B: Sediment Chemistry Tests for New York Sites WITH Potential Clamshell Dredging AND ALL New Jersey Sites

Test Analyte	USACE/EPA Required Reporting Limits[1]		
	Tissue Reporting Limit (wet)	Water Reporting Limit	Sediment Reporting Limit (dry)
SEMI-VOLATILES/PAHs	**(ng/g)**	**(µg/L)**	**(ng/g)**
Acenaphthene (LMW)	50	N/R	100
Acenaphthylene (LMW)	50	N/R	100
Anthracene (LMW)	50	N/R	100
Benz(a)anthracene (HMW)	50	N/R	100
Benzo(b)fluoranthene (HMW)	50	N/R	100
Benzo(k)fluoranthene (HMW)	50	N/R	100
Benzo(g,h,i)perylene (HMW)	50	N/R	100
Benzo(a)pyrene (HMW)	50	N/R	100
Chrysene (HMW)	50	N/R	100
Dibenz(a,h)anthracene (HMW)	50	N/R	100
Fluoranthene (HMW)	50	N/R	100
Fluorene (LMW)	50	N/R	100
Indeno(1,2,3-c,d)-pyrene (HMW)	50	N/R	100
Naphthalene (LMW)	50	N/R	100
Phenanthrene (LMW)	50	N/R	100
Pyrene (HMW)	50	N/R	100
1,4-Dichlorobenzene	3900	N/R	100
MISCELLANEOUS			
Total Organic Carbon (TOC)	N/R	N/R	N/A

SOURCE: USACE NYD and EPA Region 2 Final Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal (2016).

KEY:
EPA = Environmental Protection Agency
N/A = Not applicable
N/R = Not required
USACE NYD = U.S. Army Corps of Engineers New York District

NOTES:
1. All concentrations must be reported to three significant figures.

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Appendix D Chemical Laboratory Standard Operating Procedures

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TITLE: Sample Handling and Electronic Login

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	L. DeFrancesco	07/2009
1	Reformat and Update, Title change from "Documentation and Tracking of Subdividing and Combining of Samples" to "Sample Handling and Electronic Login"	L. DeFrancesco	04/2011
2	Reformat document, paragraph spacing and font size, plus add copies of lab forms to file.	K. A. Simon	01/2012
3	Made edits to grammar and formatting Updated to address sample holding time for unpreserved trace metals samples after preservation at the laboratory	K. Cram M. Scott	02/2013 09/2014
4	Update	R. A. McIsaac	01/2016

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1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) covers acceptable electronic log-in procedures for the receipt, handling and disposal of samples, including ESI's documentation system for uniquely identifying the items to be tested, to ensure that there can be no confusion regarding the identity of samples at any time. This system includes identification for all samples, subsamples and subsequent extracts and digestates. A unique identification code is assigned to each sample container received in the laboratory and the ID code is placed on the container in indelible ink. This SOP also covers ESI's electronic documentation system for the tracking of samples that have been subdivided or combined to form a sample(s) distinct from the original sample or to be used for a purpose distinct from the original sample.

2.0 Definitions

Chain of Custody (COC): A form which accompanies samples during shipment. This record serves to document client information, field collection information, and the transfer of samples.

Custody Seal: A clearly labeled, signed and dated closure of a sample shipping container, the integrity of which may be used to signal the absence of tampering.

Lab ID Number: A unique number assigned to each sample during the receiving process. This number consists of the Study Number followed by a hyphen and three digit numerical suffix ("dash-number"). Numerical identifiers in "# of total #" format are added to replicate sample containers.

Project File: The documents held by management which relate to the receipt, tracking, results and reporting of a unique Sample Delivery Group.

Sample: In general, a unit of matrix enclosed by a single container (or replicate, interchangeable containers) representing a single location at a unique point in time.

Sample Delivery Group (SDG): One or more samples comprising a set, as defined by a client's request or purchase order, usually received and logged-in as a unit. One Study Number is assigned to one sample group.

Sample Receipt Record (SRR): A form initiated during the receiving process which identifies the sample condition at the time of receipt. This record includes communications resolving any sample discrepancies.

Study Number: A five digit serialized number which identifies a sample group. This number also identifies a project for report production purposes.

3.0 Applicable Documents and References

Department of Defense (DoD), Department of Energy (DoE). 2013. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.0.

The NELAC Institute (TNI). 2009. *Environmental Laboratory Sector Volume 1: Management and*

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4.0 Materials and Apparatus

Billing Report Forms
Chain of Custody (COC)
Compositing Forms
"L" Drive on a network-linked computer
Sample Receipt Record (SRR) forms
Sample Storage and Usage Log
Study Number Log Book
Thermometer

5.0 Methods and Procedures

5.1 Health Hazards

All samples received by ESI should be treated as having some potential level of health hazard. The majority of the samples evaluated for acute and chronic toxicity are wastewater treatment plant effluents. These samples, while appearing "clean", may contain pathogens which could lead to illness. Other samples may include experimental use materials with limited health effects' information. Exposure may lead to illness and health complications. See Section 9.0 for proper health and safety precautions.

5.2 Sampling Kits

Based upon the specific request, the appropriate containers for the necessary volume and quantity of sample(s) are prepared for shipment to the client. In general, sampling kits are comprised of the following:

Chain of custody forms
Custody seals
Packing material
Preservative
Preservative labels
Sample container(s)
Sample labels
Shipping container(s)
Temperature blank(s)

5.2.1 Sample Containers

Sample containers provided by ESI are made of teflon, virgin polyethylene or glass, and are provided by reputable vendors (such as QEC or Nalgene). Containers are of suitable size and quality as to not cause contamination or reaction with samples. Sample containers are selected to meet project-specific or program-specific data quality objectives, including: minimum reporting limits, sample amount and field

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durability. As such, selected containers should not require pre-cleaning or any other modifications. Suitability of any new or current source of containers may be verified through initial and ongoing analysis of laboratory bottle blanks and field reagent water blanks.

Project-specific quality assurance plans may require special pre-cleaning, serial-numbering, or sample container chain of custody documentation. These will be provided upon request and at additional cost.

5.2.2 Assembly of Sample Kits

Sampling kits generated are based on the needs of the client. Typically, chains of custody and bottle labels (blank or pre-populated with project specific information) are provided. Temperature blanks are always provided. Chains of custody are always placed in a zipper sealed plastic bag to minimize damage during shipping. If blank, bottle labels are also placed in this bag. Pre-populated labels are adhered to their corresponding bottles prior to shipment. The appropriate number and type of containers are then placed in a cooler of appropriate size for transport. Glass containers are surrounded with packing material to minimize damage during shipping. If necessary, appropriate preservatives are provided and accompanied by guidance documents describing their usage.

5.3 ESI's Sample Acceptance Policy

At the time of sample receipt all samples are to be inspected to ensure that they meet minimum protocol and method requirements. This shall include, but not be limited to the following:

Presence of chain of custody (COC)
All pertinent sections of COC are complete and document is signed
Custody seal if present, is intact
Samples are properly labeled
Samples are in acceptable condition (intact, proper container and preservative)
Sample volume is sufficient
Samples received within holding time

The above information shall be entered on the Sample Receipt and Condition Document (see Section 5.4.5). A copy of this document will be included in the final report. If samples received do not meet EnviroSystems' sample receipt policies, the sample is retained and every attempt is made to resolve the discrepancy. Minor discrepancies may be resolved by contacting the client and noting the deviation and corrective actions. Sample temperature, incomplete COC records and sample preservation may be resolved through these means. In these cases, the discrepancy does not immediately threaten the integrity of the data, and testing will usually continue unless otherwise requested by the client. In situations where the discrepancy jeopardizes the integrity of the data, the client will be contacted immediately and testing will be temporarily suspended until an appropriate course of action is determined. In cases where a sample appears not to meet program criteria, the client will be notified and consulted on how to proceed. If the client elects to have the samples analyzed, then data will be reported with the appropriate qualifiers. In cases where the client indicates that they do

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not wish to have samples analyzed, the laboratory will make the appropriate notations and handle the samples as specified by the client.

5.4 Sample Receipt and Processing

5.4.1 Upon sample receipt, sample shipping containers are inspected for general condition and the presence of custody seals. If custody seals are present on the sample shipping container, they are removed from the container, taped onto blank paper, and placed with the project documents. If there are air bills present, they are also taped onto blank paper, and placed with the project documents.

5.4.2 Record the sample receipt temperature (ideally from a temperature blank). All samples which require thermal preservation shall be considered acceptable if the arrival temperature is within 2°C of either the required temperature or the method specified range. For samples with a specified temperature of 4°C, a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable. Samples that are delivered to the laboratory on the same day that they are collected may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice. If a temperature blank is not available, other temperature measurement procedures may be used, such as the use of an IR gun to monitor the surface temperature of a sample. The temperature measured is recorded on the Sample Receipt Record (Figures 1 and 2). If there are multiple shipping containers, the temperature for each shipping container should be noted. Be aware that if you take a temperature blank out of a cooler to measure, the temperature will be impacted by the ambient conditions and should be recorded immediately.

5.4.3 Chain of Custody

The National Enforcement Investigations Center (NEIC) of EPA defines evidence of custody in the following manner:

It is in your actual possession, or, It is in your view, after being in your physical possession, or It was in your possession and then you locked or sealed it up to prevent tampering, or It is in a secure area.

Field personnel or client representatives must complete the chain of custody form(s) and samples received by the laboratory must be accompanied by this form(s). The chain of custody may be the means by which the courts, in some types of legal proceedings, require proof of custody of samples from time of receipt to completion of analysis. (See Figure 3 for an example chain of custody.)

Samples may be physical evidence for litigation and should be handled according to certain procedural safeguards.

The chain of custody should contain the following information:

Analysis requested
Client project name

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Container size and type
Date and time sampled
Date and time samples received
Date and time samples relinquished
Field sample identification (Field ID)
Matrix
Preservative (if present)
Sampler initials
Signature of person receiving samples
Signature of person relinquishing samples

The record is filled out completely and legibly in ink. Correction of errors is made by drawing a single line through the error and entering initials and date. The correct information is then recorded with indelible ink. All transfers of samples, except to and from commercial couriers, must be recorded on the chain of custody via the "relinquished" and "received by" sections. All information except signatures may be printed.

Verify that all samples delivered match the information provided on the chain of custody. Any sample discrepancies should be recorded and the client should be notified. This information must be documented on the sample receipt record. Record the Study Number on the chain of custody and assign Lab ID Numbers to the samples listed. (Assign a Study Number to the project if it not already, see Figure 4.)

If necessary, an internal custody record may be generated to track the transport and status of each sample within the laboratory. Further details on internal laboratory custody procedures are provided in subsequent sections.

5.4.4 Sample Identification

Samples must be labeled with the Lab ID Number and it must be recorded with indelible ink. When multiple sample containers are received for the same analysis, an additional numerical identifier is written on each container replicate. The identifiers always start with “1” and increase by 1 for each container. Both the identifier and total number of sample containers are recorded on each container in “# of total #” format.

Cubitainers for Bioassays:

Cubitainers are also labeled with a sample type identifier. The letter “E” is used for all effluents, the letter “D” is used for all receiving waters. The renewal number is identified with “0" for the start sample, “1" for the first renewal, and “2" for the second renewal. For example, the start effluent sample would be labeled as “E0".

5.4.5 Sample Receipt Records

Document the following information on the sample receipt record: completeness of the chain of custody; sample condition; sample temperature at receipt; presence of intact custody seals (if applicable); date and time of receipt; initials of person

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receiving and logging in samples. The sample receipt record will remain with the project documents and a copy will be included in the final report. If samples have been received not in proper condition, clearly document the status of the impacted samples. A manager should then be notified and based on professional judgement, corrective actions should be initiated. Minor issues, such as the loss of one aliquot in a set, may not require much action, whereas loss of sample integrity would require new collection.

Sample Receipt Records for chronic bioassays that use renewal water samples follow a specialized format (Figure 1). All other samples will use an electronic sample receipt record (Figure 2). This form will be generated using a template found on the server during the sample login procedure.

5.4.6 Sample Preservation

Chemical preservation must be checked at the time of sample receipt for all samples, unless it is not technically acceptable to do so. The parameter list should be consulted if the correct preservation is unknown, the file can be found on the network (L:\DATA2\LOGIN\paramlst.wb1). Samples that have their pH verified or require adjustment need to be recorded in the Preservation Logbook (Figure 5) or on the Sample Receipt Record.

If a sample for metals analysis arrives unpreserved, then it needs to be preserved. The preservation is recorded and the bottle is to be “flagged” to alert the analyst. To “flag” the bottle, apply a label to the cap with the preservation date and time. The method specifies that prior to analysis, samples are held 24 hours after preservation. At the request of the client, the sample may be analyzed without the 24 hour holding period, such data will be appropriately qualified.

5.4.7 Billing Reports

A billing report is filed when samples are received for bioassay, chemistry or microbiological studies. Staple a copy of the chain of custody to the completed billing report form and place in the accounts payable inbox. Examples of the Bioassay Billing Report Form, Chemistry Billing Report Form, and Microbiological Billing Report Form can be found in the appendix (Figures 6-8). A billing report form is not filed for Ecological Risk studies.

5.4.8 Immediate Analysis

Some parameters have short holding times and require immediate analysis, which is defined as less than 48 hours from sampling time. The parameter list (Section 5.4.6) should be consulted if the holding time is unknown for an analysis. If a sample requires immediate analysis, then the appropriate staff members need to be notified.

5.4.9 Sub-samples

If an aliquot must be sub-sampled for analysis, it may be assigned a new Lab ID Number for ease of tracking. Sub-samples that receive a new Lab ID are entered into

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the electronic login database table with the same Field ID, date and time sampled as the parent sample. The date and time that the sub-sample was taken should be recorded as the date and time received in the electronic login table. There may be times when a sample will need to be sub-sampled for multiple analyses. In such a case, the analysis requested code would be filled in as "Subsample: test A, test B, test C;".

5.4.10 Subcontracting

Every effort is made to perform analyses within the ESI laboratory. There are, however, instances where subcontracting of analytical services is necessary.

Subcontracting a testing service will not be done without written approval from the client. Written approval will include an email confirmation of the approval to use a subcontractor, and a record of communications between ESI and the client will be included in the project file. Whenever possible, a NELAP or DoD accredited laboratory will be chosen to accompany the analytical service request. In order to be compliant with DoD requirements, subcontract laboratories must be DoD accredited and have project-specific written approval prior to sample analysis. If no such laboratory exists, the client will be notified. If the client approves the use of a non-accredited laboratory, such reports will be clearly identified as being from a non-approved laboratory.

When a sample that must be subcontracted arrives, it is received like any other sample, it is billed, assigned a Lab ID Number, and logged into the electronic login database table. Samples being sent to a subcontract laboratory are transferred under chain of custody. The ESI Study Number is entered as the project name, and the Lab ID Number is entered as the Field ID. There should be no reference to the client's identification, project name, or location on the chain of custody, unless specifically requested by the client. Any client identifying information that exists on the sample bottle should be blacked out before being sent. A copy of the chain of custody is placed in the project file. The original chain of custody will remain with the samples.

Analyses that are generally subcontracted by ESI are:

Fluoride
Total Phenols
Total Kjeldahl Nitrogen (for the state of Massachusetts)
Mercury (performed for the state of Maine)
Grain Size
Volatile Fatty Acids
Herbicides
Dioxins
Furans

All subcontracted sample data reports are sent to ESI management for review, before being reported to the client. The subcontract laboratory will be clearly identified in ESI's final report to the client. A copy of the subcontractors report may

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be included in the final report.

5.4.11 Altered and Composite Sample Login

ESI occasionally receives samples for an analysis that requires altering the state of the original sample before further analysis can be performed on the sample. If a sample is altered from it's original state, it becomes a different sample. The altered sample is given a new Lab ID Number and a new Field ID. The date and time created then becomes the sampled date and time.

A composited sample is a type of altered sample. When a sample is received for compositing, it is logged into the electronic login database table with the Analysis Requested: "Composite". The composited sample is then logged into the laboratory as a new sample with a unique Lab ID Number, new Field ID, and Analysis Requested information. Compositing Form(s) must be filled out for sample tracking purposes. The completed Compositing Forms are retained in the project file.

5.5 Sample Storage

5.5.1 To ensure sample integrity, a chain of custody must be maintained and all samples must be logged into the laboratory's electronic database table (see Section 5.7). Samples logged into the laboratory and not immediately used are stored under appropriate conditions specified by the method, SOP or client. Samples are stored in areas with limited access or under locked conditions. Samples requiring secure storage or reference samples being retained for archival purposes are stored under locked conditions and may require the use of a Sample Storage and Usage Log (Figure 9). When samples are no longer required for testing or retention, they will be disposed of properly.

5.5.2 Sample Storage and Usage Logs

A Sample Storage and Usage Log will be generated for samples that require usage documentation. Requirements for the storage and usage log include: study number, client name, storage location, sample identification, date, time and initials of staff members removing and returning samples, reason for removal and approximate volumes removed. Completed sheets will be kept with the study data and may be included in the study data package.

5.6 Sample Disposal

Unused portions of samples are disposed of only after completion of sample analysis, submission of the final report, and contracted sample retention times have been achieved. All conditions of disposal and all records and correspondence concerning the final disposition of the physical sample shall be recorded and retained. Records shall indicate the date of disposal, the nature of disposal and the name of the individual who performed the task.

Samples considered as hazardous waste are handled by state and federally licensed hazardous waste disposal firms. Storage of waste samples and associated waste materials will follow applicable rules and regulations. Waste material stored in the storage shed will be

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inspected at least once each week to ensure container integrity and accessability. Inspections will be documented by a Hazardous Waste Coordinator. Waste storage containers filled with liquid materials are placed on spill pallets with sufficient volume to accommodate the containers. A list of contact persons, with day and home telephone numbers, can be found in the storage shed.

5.6.1 Aqueous Samples

After testing, aqueous samples will be disposed of through ESI's aqueous waste UV treatment system located on the first floor. This includes all unused samples, diluted samples and dilution waters that are not preserved. Access points for aqueous waste disposal are located throughout the laboratory, second floor's access "waste chute" is located in the dishwashing area. ESI's Federal and State NPDES Permit allows waste disposed of in this manner to be discharged to the Taylor River.

5.6.2 Solid Materials

Waste storage, transfer and disposal will be in compliance with NH DES and US EPA waste disposal regulations. Non-hazardous solid materials, sediments and soils will be disposed of through an appropriate waste disposal site. All non-hazardous solid waste will be drained of as much free liquid as possible. The remaining sample is removed from its original container and placed with other non-hazardous waste samples in a 55 gallon metal drum. Once full, these containers will then be transferred to an approved waste disposal facility. This transfer is recorded on a waste manifest and they are retained for a minimum of 3 years.

5.6.3 Chemically Preserved Samples (Characteristically Hazardous Only)

Chemically preserved samples should have a pH of <2 or >12 SU. The pH alone causes them to be classified as characteristically corrosive hazardous wastes. ESI has a Limited Permit granted by the NH DES that allows for the use of an elementary base neutralization unit to render characteristically hazardous wastes non-hazardous. Prior to disposal, samples are neutralized allowing them to be safely added to ESI's aqueous waste UV treatment "pit" system for discharge. The approximate volume of neutralized waste, date of disposal and initials of person performing the disposal, are recorded in a logbook. Samples considered hazardous for any other reason can not be neutralized and disposed of in this fashion.

5.6.4 Hazardous Waste Samples

Hazardous waste samples must be handled by properly trained employees. Only the ESI hazardous waste coordinator may train ESI employees on handling hazardous waste. There are 3 main hazardous waste streams at ESI, "dilute metals acidic", "solvents" and "Lachat wastes". When a satellite container needs to be emptied, it is properly transported to the waste shed and added into the appropriately labeled container for that waste stream. Logbooks are kept as a record for volumes added to each drum of any specific waste stream and shipping manifests reflect when they are picked up for transport.

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5.7 Electronic Sample Login

Data for each sample is entered into an electronic login database table after the sample has been deemed acceptable and the proper information has been recorded on previously mentioned records (i.e. Sample Receipt Record, sample preservation, billing report). Samples can be logged into ESI's electronic data system by appending the login table located at: "L:\Data2\LOGIN\login.wb1" or by using a template found at: "L:\Data2\startup_.wb1". On the primary template page, the button titled "Sample Receipt and Login" is where the template can be accessed. The template acts as a prompt and helps one fill in the necessary information for sample login.

The login table has multiple records (rows). Each record (row) has 22 fixed fields (columns, listed below). The first record in the table has in each of its fields a fixed "field name". There is a subsequent record (row) for each unique sample. The following table lists the login table field names, character length, format examples and descriptions.

Field Name	Length	Format Example	Description
LabID	9	*99999-999*	All numbers and hyphen are required. The first 5 numbers is the Study Number and the last 3 digits after hyphen is the Sample Number. Multiple portions get the same 3 digit sample number if they can be used interchangeably.
Field ID	64	alphanumeric	This is how the sample is identified on the chain of custody. The same Field ID may be assigned to more than one Lab ID Number. (For example, when portions are in different containers for different analyses.)
Matrix	5	"Water" or "Solid"	The matrix is used to format calculations. Any other entry causes errors.
Analyses Requested	240	"test1", "test1, test2", "analyte1,analyte2;"	This lists what analyses are to be performed on a sample. Analysis codes are taken verbatim from a list of analysis names found in L:\data2\Login\paramlst.wb1. Tests are separated by commas. Multi-analyte tests are terminated by a semicolon.
SAMPLED	8	'09/02/02 2345	This text string starts with a prime symbol. It is the date and time a sample was taken or originated. Time is reported with 4 digits, in 24 hour format and without a colon.
CUST	5	ESI_1	This is a code used to identify customers. It contains 5 characters; 3 or 4 alpha characters and 1 numeric character. If 3 alpha characters are used, an underscore is placed before the numeric character.
PROJ	55	First Quarter Permit	This string is used to identify project names.
PROTOCOL	12	NPDES	This is used to identify the appropriate test protocol.
PRIORITY	8	'09/02/02	This starts with a prime symbol and is the report due date.

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Field Name	Length	Format Example	Description
INVOICE	12	'11234	This starts with a prime symbol and identifies the invoice number assigned to the project.
RECEIVED	8	'09/02/02 2345	This starts with a prime symbol and identifies the date and time a sample was received. Time is reported with 4 digits, in 24 hour format and without a colon.
PONum	24	'12345	This starts with a prime symbol and is the customer's Purchase Order Number.
Mailed	8	'09/02/02 or '09/02/02 WEB	This entry starts with a prime symbol and is when the report was mailed or delivered. If initials are necessary, the date string is followed by a space and up to 4 characters. This field is not currently in use.
SComment	240	'HOLD Total Metals Cd,Hg;	This is the sample comment field and where special requests are noted.
Comment2	240	WB or WB SJ	The initials of who received and logged the sample. If not the same person, then initials of the receiver are placed before the logger, and 2 spaces are placed between the initials.
Sample_Delivery_Group	40	-	This identifies samples that are part of a group. It can be a study number or a name.
CTR	8	'250mL P or '2x40mL G	Entry starts with prime symbol and is the sample container size and type. Size is followed by a type identifier, "P" for plastic, "G" for glass. Multiple containers are noted by container number than "x" in front of entry (second example).
PRES	5	'H2SO4 or 'HCL	This is where the sample preservation is noted.
FieldFiltered	13	-	If a sample has already been filtered, it is noted here.
SITE	240	-	In cases where samples may be derived, this field identifies the original sample.
PROJ_NO	5	'P00000	For in-house project table reference, or for inclusion of EPA Case Number for CLP reporting purposes.
COC_COM	-	-	This is where any chain of custody notes are reported.

6.0 Quality Control Requirements

As required, all appropriate state and federal reports associated with the disposal of hazardous waste material will be filed with the appropriate agencies.

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All other sample documents will be retained by the laboratory's QA system for a period of at least five years.

7.0 Calculations and Reporting

This section is not applicable to this Standard Operating Procedure.

8.0 Corrective Actions

This section is not applicable to this Standard Operating Procedure.

9.0 Health and Safety

9.1 Gloves and safety glasses should be worn when handling samples and chemicals. It is advisable to wear a lab coat to protect clothing.

9.2 Strong acids and bases should be added to containers and solutions under the hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

9.3 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding MSDS sheets.

10.0 Responsibilities

10.1 It is the Lab Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedures outlined in this SOP.

10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities.

11.0 Waste Management and Pollution Prevention

11.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the biohazard and hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from bench operations. Compliance with all sewage discharge permits and regulations is also required.

11.2 Assays and sample disposal will be conducted using procedures to minimize potential

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pollution of surface waters, ground waters, soils and sediments.

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APPENDIX

Figure 1. Chronic Exposure WET Test Sample Receipt Record
Figure 2. Electronic Sample Receipt Record
Figure 3. Chain of Custody Record
Figure 4. Study Number Log Book
Figure 5. Sample Preservation Log
Figure 6. Bioassay Billing Report Form
Figure 7. Chemistry Billing Report Form
Figure 8. Microbiology Billing Report Form
Figure 9. Sample Storage and Usage Log

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Figure 1. Chronic Exposure WET Test Sample Receipt Record

SAMPLE RECEIPT RECORD
FOR CHRONIC TOXICITY EVALUATIONS

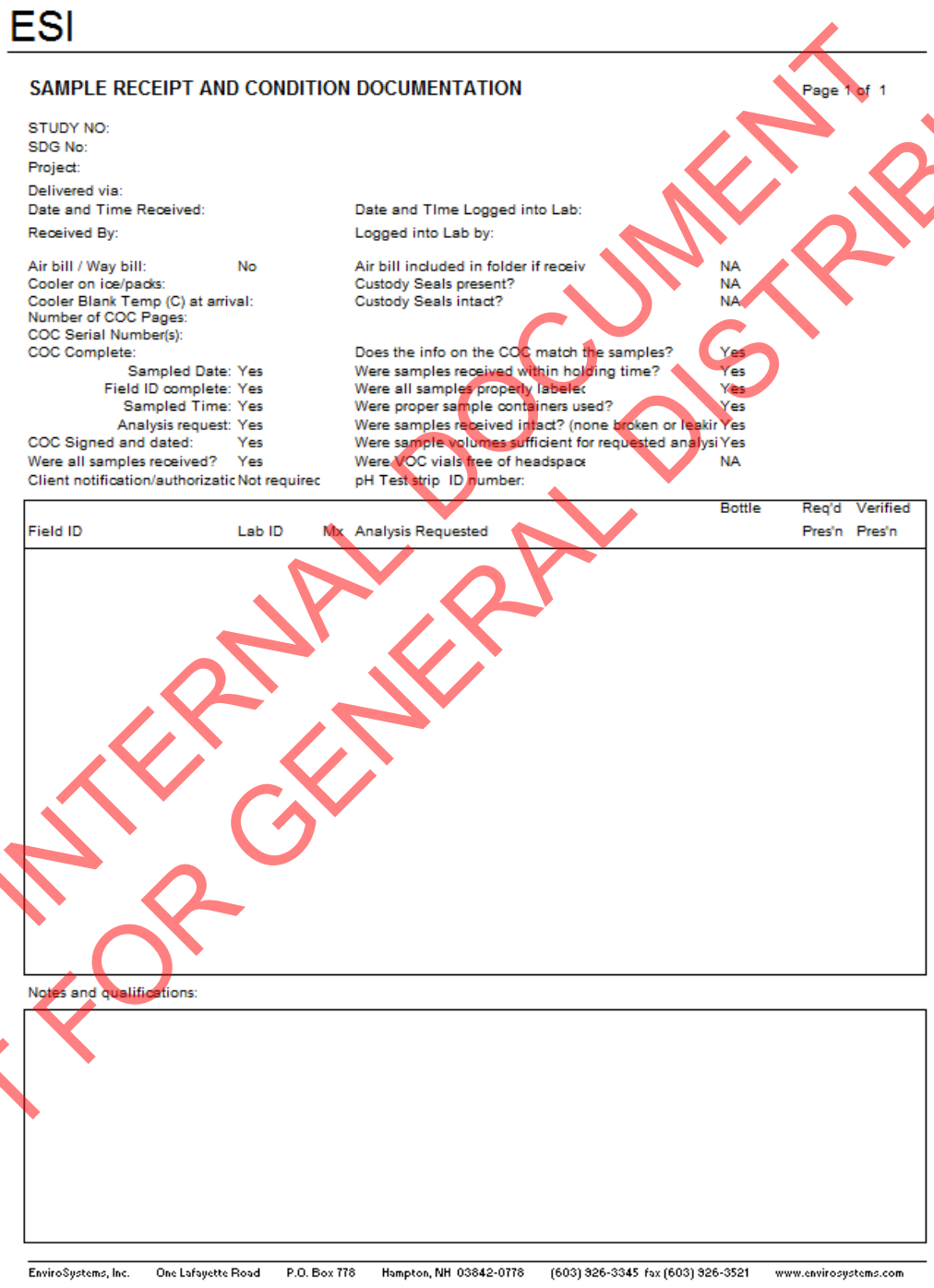
STUDY #:		CLIENT:	
SAMPLE RECEIPT INFORMATION			
	Start Sample	First Renewal	Second Renewal
Sample Receipt Date & Time:			
Received By:			
Delivered Via:	Fed Ex UPS Client Courier ESI	Fed Ex UPS Client Courier ESI	Fed Ex UPS Client Courier ESI
Logged Into Lab By:			
Date &Time Logged In:			
SAMPLE CONDITION INFORMATION			
Chain of Custody?	Yes or No	Yes or No	Yes or No
Chain of Custody Signed?	Yes or No	Yes or No	Yes or No
Chain of Custody Complete?	Yes or No	Yes or No	Yes or No
Sample Date?	Yes or No	Yes or No	Yes or No
Sample Time?	Yes or No	Yes or No	Yes or No
Sample Type?	Yes or No	Yes or No	Yes or No
Custody Seal in Place?	Yes NA No	Yes NA No	Yes NA No
Shipping Container Intact?	Yes or No	Yes or No	Yes or No
Temp Blank Temperature:			
DOES CLIENT NEED NOTIFICATION OF TEMP?	Yes or No	Yes or No	Yes or No
Sample Arrived on Ice?	Yes or No	Yes or No	Yes or No
COMMENTS:			

P:\GENERAL PROJECTS\FORMS\LABFORMS\Sample Receipt Record - Chronic 2013.wpd

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Figure 2. Electronic Sample Receipt Record



ESI

SAMPLE RECEIPT AND CONDITION DOCUMENTATION

Page 1 of 1

STUDY NO:
SDG No:
Project:
Delivered via:
Date and Time Received:
Received By:

Date and TIme Logged into Lab:
Logged into Lab by:

Air bill / Way bill:	No	Air bill included in folder if receiv	NA
Cooler on ice/packs:		Custody Seals present?	NA
Cooler Blank Temp (C) at arrival:		Custody Seals intact?	NA
Number of COC Pages:			
COC Serial Number(s):			
COC Complete:		Does the info on the COC match the samples?	Yes
Sampled Date:	Yes	Were samples received within holding time?	Yes
Field ID complete:	Yes	Were all samples properly labelec	Yes
Sampled Time:	Yes	Were proper sample containers used?	Yes
Analysis request:	Yes	Were samples received intact? (none broken or leakir	Yes
COC Signed and dated:	Yes	Were sample volumes sufficient for requested analysi	Yes
Were all samples received?	Yes	Were VOC vials free of headspac	NA
Client notification/authorizatic	Not requirec	pH Test strip ID number:	

Field ID	Lab ID	Mx	Analysis Requested	Bottle	Req'd Pres'n	Verified Pres'n

Notes and qualifications:

EnviroSystems, Inc. One Lafayette Road P.O. Box 778 Hampton, NH 03842-0778 (603) 926-3345 fax (603) 926-3521 www.envirosystems.com

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Figure 3. Chain of Custody Record

CHAIN OF CUSTODY RECORD

ENVIROSYSTEMS, INCORPORATED
P.O. Box 778, Hampton, New Hampshire 03842

ESI Study Number:_____

Customer Services: Phone # (603) 926-3345
Fax # (603) 926-3521

PAGE OF

CLIENT: | CONTACT: | PROJECT NAME: | **P.O. #**

REPORT TO: | ADDRESS: | PHONE:

INVOICE TO: | ADDRESS: | SAMPLED BY:

Program Requirements: ☐ NPDES ☐ RCRA ☐ USACE ☐ EPA ☐ OTHER

SAMPLE #	YOUR FIELD IDENTIFICATION (MUST AGREE WITH CONTAINER)	DATE SAMPLED	TIME SAMPLED	COMPOSITE /GRAB	E-EFFLUENT D-DILUENT O-OTHER	CONTAINER #/VOL/TYPE	FIELD PRESERVED	ANALYSIS REQUESTED (SPECIAL INSTRUCTIONS, CAUTIONS, ETC.)

RELINQUISHED BY: DATE: TIME: RECEIVED BY: DATE: TIME:

Figure 4. Study Number Log Book

Project #	Date Started	Client Name	Assay Type
26517			
26518			
26519			
26520			
26521			
26522			
26523			

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Figure 5. Sample Preservation Log

pH Strips A-

SAMPLE PRESERVATION LOG

Nitric Acid A-

Sulfuric Acid A-

Today's Date	Init	ESI #	Client	Cube #	Date CHEM Sampled	Analysis	H_2SO_4 (✓)	HNO_3 (✓)	Other *Indicate*	Client (✓)	Bottle #	pH <2	pH neutral	pH >12

Figure 6. Bioassay Billing Report Form

BIOASSAY BILLING REPORT

ESI STUDY NUMBER: _____ **SAMPLE RECEIPT DATE:** _____

CLIENT: _____ **PROJECT:** _____

P.O. NUMBER: _____

# of TESTS	ASSAY TYPE	UNIT PRICE	Extension
	C.dubia ☐24 ☐48 ☐AS ☐AD		
	D.pulex ☐24 ☐48 ☐AS ☐AD		
	D.magna ☐24 ☐48 ☐AS ☐AD		
	C.dubia 7 Day Chronic Assay		
	P.promelas ☐24 ☐48 ☐AS ☐AD		
	P.promelas 7 day Chronic Assay		
	Trout 10 Day Chronic Assay		
	A.bahia ☐24 ☐48 ☐AS ☐AD		
	A.bahia 7 day Chronic Assay		
	M.beryllina ☐24 ☐48 ☐AS ☐AD		
	M.beryllina 7 day Chronic Assay		
	A. puctulata Fertilization Assay		
	A. puctulata Developmental Assay		
	Standard Trace Metals - Effluent		
	Standard Trace Metals - Diluent		
	TOC		

TASK	UNIT PRICE	Extension
Pick-up & Delivery of Samples:		
Fuel Charge:		
Sampling Services:		
Analytical Support:		
Renewals:		
Additional Charges or Comments		
TOTAL:		

Provide the number of samples received for each type of analysis to be conducted as part of this study. Also attach a copy of the chain of custody. Deliver the completed form to Patti for billing purposes. Patti will take care of all pricing. **IF YOU HAVE ANY QUESTIONS PLEASE CONSULT A MANAGER.**

INITIAL: _____ DATE: _____

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Figure 7. Chemistry Billing Report Form

CHEMISTRY BILLING REPORT

ESI STUDY NUMBER: _____ **SAMPLE RECEIPT DATE:** _____

CLIENT: _____ **PROJECT:** _____

P.O. NUMBER: _____

# SAMPLES	ANALYSIS PERFORMED	UNIT PRICE	EXTENSION
	SAMPLE DELIVERY & RECEIPT		
TOTAL:			

Provide the number of samples received for each type of analysis to be conducted as part of this study. Also attach a copy of the chain of custody. Deliver the completed form to Patti for billing purposes. Patti will take care of all pricing. **IF YOU HAVE ANY QUESTIONS PLEASE CONSULT A MANAGER.**

INITIAL: _____ DATE: _____

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Figure 8. Microbiology Billing Report Form

MICROBIOLOGY
Billing Report

ESI STUDY NUMBER: _____ **SAMPLE RECEIPT DATE:** _____
CLIENT: _____ **PROJECT:** _____
P.O. NUMBER: _____

# SAMPLES	ANALYSIS PERFORMED	UNIT PRICE	EXTENSION
	Fecal Coliform		
	Total Coliform		
	Enterococcus		
	Heterotrophic Plate Count		
	E. coli		
	ADDITIONAL ANALYSIS		
	TS		
	Conductivity		
	SAMPLE DELIVERY & RECEIPT		
TOTAL:			

Provide the number of samples received for each type of analysis to be conducted as part of this study. Also attach a copy of the chain of custody. Deliver the completed form to Patti for billing purposes. Patti will take care of all pricing. **IF YOU HAVE ANY QUESTIONS PLEASE CONSULT A MANAGER.**

INITIAL: _____ DATE: _____

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Figure 9. Sample Storage and Usage Log

LABORATORY SAMPLE STORAGE/USAGE LOG

ESI Study: Client:		Project:		
Sample ID	**Sample Removed from Storage Date / Time / By**	**Sample Returned to Storage Date / Time / By**	**Reason for Sample Removal**	**Sample Volume Removed**

PAGE ___ of ____

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TITLE: Handling and Storage of Compressed Gases

Approved By:

QA Officer:_____ Date:_____

Technical Director:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
1	Review and Update	P. Karbe	10/1999
2	Review and Update	S. Dionne	06/2000
3	Review and Update	M. Scott	01/2013

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TITLE: Handling and Storage of Compressed Gases

1.0 Purpose and Applicability

This document was generated and provided by WESCO of Billerica, Massachusetts. The document provides a comprehensive program of gas handling, storage and guidance for general use and distribution of compressed gases.

2.0 Definitions

See Attached Document

3.0 Applicable Documents/References

WESCO. 1995. Safe Storage and Handling of Gases. WESCO, Billerica, MA. 18 pages.

4.0 Materials and Apparatus

See Attached Document

5.0 Methods/Procedures

See Attached Document

6.0 Quality Control Requirements

See Attached Document

7.0 Calculations/Reporting

NA

8.0 Corrective Actions

See Attached Document

9.0 Health and Safety

See Attached Document

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TITLE: Handling and Storage of Compressed Gases

10.0 Responsibilities

It is the lab manager's responsibility to insure technicians performing this procedure are properly trained and the training is documented in the technicians training file. It is the lab supervisor's responsibility to make sure standard operating procedures are up to date.

It is the analyst's responsibility to read and follow this standard operating procedure.

11.0 Waste Management and Pollution Prevention

NA

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TITLE: Compositing of Sediment Samples

Approved By:

QA Officer:_____ Date:_____

President:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	R.A.McIsaac	09/10

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TITLE: Compositing of Sediment Samples

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) defines procedures for the preparation of a composite sample from multiple samples for subsequent testing. This SOP is specific to compositing of sediment samples for bioassay testing. Compositing of other samples for alternative purposes may require alternative methods.

2.0 Definitions

Composite Sample: A single sample prepared from two or more discrete samples. Composites will be prepared based on client instructions.

3.0 Applicable Documents/References

ASTM. 2010. *Standard Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing and for Selection of Samplers Used to Collect Benthic Invertebrates.* E 1391-03, West Conshohocken, PA.

4.0 Materials and Apparatus

- Sediment Samples
- Volume Measuring equipment
- Non-contaminating container of sufficient volume to accomplish compositing
- Mixing equipment made of a non-contaminating material
- ESI SOP 1113 - Cleaning and Maintenance of General Laboratory Glassware

5.0 Methods/Procedures

5.1 Confirm client's specific compositing requirements; samples may be composited on equal volumes or on volume provided. Determine total volume of composite sample and select appropriate mixing container. Acceptable containers include containers constructed of high density polyethylene (HDPE), glass or Teflon.

5.2 Insure that the mixing chamber and equipment used for compositing are clean. Beakers and buckets will be washed, acid soaked and rinsed, see SOP 1113. Larger containers will be washed and rinsed with deionized water prior to use.

5.3 In some cases settling may have occurred and there may be overlying water in the sample container. In most cases this is included in the composite, but if the technician believes this volume to be excessive it may be decanted. In all cases the project file should be consulted to assure that there are not client specific requirements.

5.4 If the project sediments require sieving, this is usually done during the compositing process. The Project Manager should be consulted as to whether or not samples need to be sieved and what size screen should be applied.

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TITLE: Compositing of Sediment Samples

5.5 Once appropriate portions of each discrete sample have been added to the mixing chamber, the sample should be either mechanically stirred using a non-contaminating material or paddle to achieve complete mixing. Mixing should be performed as quickly and efficiently as possible.

5.6 Once samples are completely mixed, the composite may be placed in the original sample containers or in another appropriate container. Composite containers should be clearly labeled with indelible ink on the sides of the container.

6.0 Quality Control Requirements - Not Applicable

7.0 Calculations/Reporting

7.1 When samples are composited, a Laboratory Composite Preparation Documentation form needs to be completed and filed in the project folder.

8.0 Corrective Actions - Not Applicable

9.0 Health and Safety

9.1 As with all samples, gloves and safety glasses should be worn when handling samples. It is advisable to wear a lab coat to protect clothing.

9.2 At the end of compositing, test material is stored at 2-4°C in a secure location. Sediments can usually be disposed of once clients have had a three week comment period to accept the results of an assay. It is recommended that the project file and the Project Manager are consulted before sediment is disposed.

9.3 If the material is classified as non- hazardous, the material may be disposed in an appropriate waste container. Sample disposal will be conducted using procedures to minimize potential pollution of surface and ground waters, soils and sediments.

10.0 Responsibilities

10.1 It is the Lab Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedures outlined in this SOP.

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Technical Information

Reference Number:	ASTM D854
Test Method Title:	Standard Test Method for Specific Gravity of Soil Solids by Water Pycnometer
Test Property:	Specific gravity
Test Specimen Size:	Specific gravity of soil passing the #4 sieve

Specimen dry mass in grams when using a 500 ml pycnometer:

SP, SP-SM	100 ± 10
SP-SC, SM, SC	75 ± 10
Silt or clay	50 ± 10

Number of Test Specimens: 1 (representative sample obtained by quartering)

Test Equipment:

500 ml volumetric pycnometer
Drying oven capable of maintaining 110 ± 5 °C
Balance readable to 0.01 gram with 1,000 gram capacity
Thermometer readable to 0.1 °C
System to apply heat and/or agitation
Vacuum system
Mortar and pestle
Funnel
Distilled water
De-aired distilled water
Squirt bottle
Dessicator
Insulated container

Standard Operating Procedure

METHOD A - Procedure for Moist Specimens

1a. Check the dry weight of your pycnometer against its calibrated dry weight. If it is off by more than 0.06g do not use pycnometer until a recalibration is performed. Determine recommended dry mass for test specimen. Collect sufficient wet mass of sample to satisfy dry mass criteria. Add soil to 100 ml of distilled water. Disperse the soil in the distilled water using the blender. Using a funnel, pour the slurry into a calibrated pycnometer. Rinse any soil particles remaining on the funnel into the pycnometer using a wash/spray squirt bottle of distilled water. Add distilled water into the pycnometer until the water level is about ½ the depth of the main body of the pycnometer. Rinse any soil adhering to the inside neck of the pycnometer into the slurry.

Proceed to Step 2.

METHOD B – Procedure for Oven-Dried Specimens

1b. Check the dry weight of your pycnometer against its calibrated dry weight. If it is off by more than 0.06g do not use pycnometer until a recalibration is performed. Dry the specimen to a constant mass in an oven maintained at 110+/- °5C. Break up any clods of soil using a mortar and pestle. If the soil will not easily disperse after drying or has changed composition, use Test Method A. Place funnel into pycnometer. The stem of the funnel must extend past the calibration mark. Spoon the soil solids directly into the funnel. Record the weight of the dry soil solids in the pycnometer. Add water until the water level is about ½ the depth of the main body of the pycnometer. Agitate the water until a slurry is formed. Rinse any soil adhering to the inside neck of the pycnometer into the slurry.

Proceed to Step 2.

2. Entrapped air needs to be removed by using a vacuum, boiling or both vacuum and boiling. The pycnometer needs to be continually agitated for at least two hours under vacuum or boiling alone. (This means the silt/clay particles need to remain in suspension and slurry is in constant motion.) The vacuum must remain relatively constant and be sufficient to cause bubbling at the beginning of the de-airing process. If both boiling and vacuum method is used, this must last a minimum of 1 hour.

3. Add de-aired distilled water to the pycnometer until it nearly reaches the calibration mark. Place pycnometer(s), a bottle of de-aired distilled water and thermometer in a closed insulated container and allow them to reach thermal equilibrium overnight. When thermal equilibrium is achieved, remove pycnometer by only touching the top and placing it on an insulated surface near the scale you will be using to weigh it on. Top the pycnometer up so the calibration mark is at the mid-height of the meniscus of the water in the pycnometer. Wipe drips above the calibration mark on the inside of the pycnometer and wipe and dry the entire outside of the pycnometer. Measure and record the mass of pycnometer, soil, and water. Measure and record the temperature of the slurry/soil-water mixture.

4. For both Method A and B –Weigh out an empty, clean dish for drying specimen in oven. Transfer the slurry to a dish using distilled water (be sure all soil particles are transferred from pycnometer to dish). Dry the specimen to a constant mass in an oven maintained at 110+/- 5 ºC. After the soil is oven-dried record the dry weight of soil and dish. Calculate and record the mass of dry solids to the nearest 0.01g.

5. Calculate the mass of the pycnometer and water at the test temperature:

$$M_{pwt} = M_p + (V_p \bullet P_{wt})$$

Where:

M_{pwt} = Mass of the pycnometer and water at the test temperature
M_p = the average calibrated mass of the dry pycnometer
V_p = the average calibrated volume of the pycnometer
P_{wt} = the density of water at the test temperature

6. Calculate the specific gravity as follows:

$$G_t = M_s / (M_{pwt} – (M_{pwst} - M_s)$$

Where:

G_t = specific gravity
M_s = weight of oven-dry soil, g
M_{pwt} = mass of water and pycnometer at test temperature
M_{pwst} = mass of water pycnometer, water, and soil solids at test temperature

7. Calculate the specific gravity of soil solids at 20 ºC as follows:

$$G_{20} = K \bullet G_t$$

Where:

K = the temperature coefficient given in Table 2

8. Report: sample identification, specific gravity at 20 °C to the nearest 0.01, test method used, maximum particle size of the specimen, if any portion of the original sample was not used for the test.

9. NOTE: If porosity is required, use results of a density test and specific gravity (ASTM D 854) to calculate as follows:

$$n = 1 - (\gamma_d / G \times \gamma_w)$$

Where:

n = porosity
γ_d = dry density of soil, lbs/ft^3
G = specific gravity of soil
γ_w = specific gravity of water

TABLE 2 Density of Water and Temperature Coefficient (K) for Various Temperatures[A]

Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)	Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)	Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)	Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)
15.0	0.99910	1.00090	16.0	0.99895	1.00074	17.0	0.99878	1.00057	18.0	0.99860	1.00039
.1	0.99909	1.00088	.1	0.99893	1.00072	.1	0.99876	1.00055	.1	0.99858	1.00037
.2	0.99907	1.00087	.2	0.99891	1.00071	.2	0.99874	1.00054	.2	0.99856	1.00035
.3	0.99906	1.00085	.3	0.99890	1.00089	.3	0.99872	1.00052	.3	0.99854	1.00034
.4	0.99904	1.00084	.4	0.99888	1.00067	.4	0.99871	1.00050	.4	0.99852	1.00032
.5	0.99902	1.00082	.5	0.99886	1.00066	.5	0.99869	1.00048	.5	0.99850	1.00030
.6	0.99901	1.00080	.6	0.99885	1.00064	.6	0.99867	1.00047	.6	0.99848	1.00028
.7	0.99899	1.00079	.7	0.99883	1.00062	.7	0.99865	1.00045	.7	0.99847	1.00026
.8	0.99898	1.00077	.8	0.99881	1.00061	.8	0.99863	1.00043	.8	0.99845	1.00024
.9	0.99896	1.00076	.9	0.99879	1.00059	.9	0.99862	1.00041	.9	0.99843	1.00022
19.0	0.99841	1.00020	20.0	0.99821	1.00000	21.0	0.99799	0.99979	22.0	0.99777	0.99957
.1	0.99839	1.00018	.1	0.99819	0.99998	.1	0.99797	0.99977	.1	0.99775	0.99954
.2	0.99837	1.00016	.2	0.99816	0.99996	.2	0.99795	0.99974	.2	0.99773	0.99952
.3	0.99835	1.00014	.3	0.99814	0.99994	.3	0.99793	0.99972	.3	0.99770	0.99950
.4	0.99833	1.00012	.4	0.99812	0.99992	.4	0.99791	0.99970	.4	0.99768	0.99947
.5	0.99831	1.00010	.5	0.99810	0.99990	.5	0.99789	0.99968	.5	0.99766	0.99945
.6	0.99829	1.00008	.6	0.99808	0.99987	.6	0.99786	0.99966	.6	0.99764	0.99943
.7	0.99827	1.00006	.7	0.99806	0.99985	.7	0.99784	0.99963	.7	0.99761	0.99940
.8	0.99825	1.00004	.8	0.99804	0.99983	.8	0.99782	0.99961	.8	0.99759	0.99938
.9	0.99823	1.00002	.9	0.99802	0.99981	.9	0.99780	0.99959	.9	0.99756	0.99936
23.0	0.99754	0.99933	24.0	0.99730	0.99909	25.0	0.99705	0.99884	26.0	0.99679	0.99858
.1	0.99752	0.99931	.1	0.99727	0.99907	.1	0.99702	0.99881	.1	0.99676	0.99855
.2	0.99749	0.99929	.2	0.99725	0.99904	.2	0.99700	0.99879	.2	0.99673	0.99852
.3	0.99747	0.99926	.3	0.99723	0.99902	.3	0.99697	0.99876	.3	0.99671	0.99850
.4	0.99745	0.99924	.4	0.99720	0.99899	.4	0.99694	0.99874	.4	0.99666	0.99847
.5	0.99742	0.99921	.5	0.99717	0.99897	.5	0.99692	0.99871	.5	0.99665	0.99844
.6	0.99740	0.99919	.6	0.99715	0.99894	.6	0.99689	0.99868	.6	0.99663	0.99842
.7	0.99737	0.99917	.7	0.99712	0.99892	.7	0.99687	0.99866	.7	0.99660	0.99839
.8	0.99735	0.99914	.8	0.99710	0.99889	.8	0.99694	0.99873	.8	0.99657	0.99836
.9	0.99732	0.99912	.9	0.98707	0.99887	.9	0.99681	0.99860	.9	0.99654	0.99833
27.0	0.99652	0.99831	28.0	0.99624	0.99803	29.0	0.99595	0.99774	30.0	0.99585	0.99744
.1	0.99649	0.99628	.1	0.99621	0.99800	.1	0.99592	0.99771	.1	0.99562	0.99741
.2	0.99646	0.99825	.2	0.99618	0.99797	.2	0.99589	0.99768	.2	0.99559	0.99738
.3	0.99643	0.99822	.3	0.99615	0.99794	.3	0.99586	0.99765	.3	0.99556	0.99735
.4	0.99641	0.99820	.4	0.99612	0.99791	.4	0.99583	0.99762	.4	0.99553	0.99732
.5	0.99638	0.99817	.5	0.99609	0.99788	.5	0.99580	0.99759	.5	0.99550	0.99729
.6	0.99635	0.99814	.6	0.99607	0.99785	.6	0.99577	0.99756	.6	0.99547	0.99726
.7	0.99632	0.99811	.7	0.99604	0.99783	.7	0.99574	0.99753	.7	0.99544	0.99723
.8	0.99629	0.99808	.8	0.99601	0.99780	.8	0.99571	0.99750	.8	0.99541	0.99720
.9	0.99627	0.99806	.9	0.99598	0.99777	.9	0.99568	0.99747	.9	0.99538	0.99716

[A]Reference: CRC Handbook of Chemistry and Physics, David R. Lide, Editor-in-Chief, 74th Edition, 1993–1994.
[B]mL = cm^3

Technical Information

Reference Number:	ASTM D2216
Test Method Title:	Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock Mass
Test Property:	Moisture content
Test Specimen Size:	As per test method based on maximum particle size
Number of Test Specimens:	1 or, as required by other testing representative based on type of sample: Undisturbed: mix sample by hand or take random samples throughout test sample, combine and mix Disturbed: take a representative cross-section of material for test
Test Equipment:	Oven capable of maintaining 60 or 110 ±5 °C Balance readable to 0.01 gram with 200 gram capacity or readable to 0.1 grams with >200 gram capacity Numbered specimen containers Desiccator

Standard Operating Procedure

1. Determine and record mass of a clean, dry specimen container.

2. Select a representative test specimen based on largest particle size. (See chart in test standard for guidance.) Place moist test specimen into container. Determine and record mass of container and moist test specimen using proper balance based specimen size.

3. Place the container with moist specimen in the oven at 110 °C (unless highly organic, in which case use 60°C). Dry material to constant mass.

4. Once specimen has dried to constant mass, remove from oven and place into desiccator. Allow cooling to room temperature (or until container can be held comfortably in hands).

5. Measure and record mass of dried specimen and container with the same type of balance used to measure the before drying mass.

6. Calculate the moisture content of the material as follows:

$$w = [(M_{cws} - M_{cs}) / (M_{cs} - M_c)] \times 100$$

where:

w = moisture content, %
M_{cws} = mass of container and wet specimen, g
M_{cs} = mass of container and dry specimen, g
M_c = mass of container, g

7. Report: sample identification, moisture content of specimens to the nearest 0.1% or 1 % based on sample size used, description of material, temperature of drying oven if other than 110 °C, size and amount of material (if any) that was excluded from the test specimen.

Technical Information

Reference Number:	ASTM D4318
Test Method Title:	Standard Test Method for Liquid Limit, Plastic Limit and Plasticity Index of Soils
Test Property:	Plasticity of soils
Test Specimen Size:	150-200 grams of material passing the #40 sieve 100 grams of as received soil for Moisture Content Determination
Number of Test Specimens:	1 (representative sample obtained by quartering, mixing or splitting)
Test Equipment:	Liquid limit device Flat grooving tool Drying containers Balance readable to 0.01 gram with 500 gram capacity Ground glass plate Spatula #40 sieve Wash bottle Distilled Water Drying oven capable of maintaining 110 ± 5 °C Drying Plate/vacuum pump apparatus Soil Blender with mixing cup

Standard Operating Procedure

The Atterberg Limit Test requires the As Received Moisture content determination to be reported. If no other assigned testing requires the As Received Moisture content determination, follow ASTM D 2216 and obtain the result for the sample.

GTX requires a daily IRM check of the equipment to include, but is not limited to: the wear of the grooving tool, the drop height of the Liquid Limit device cup and the wear spot on the base under the cup at the point of impact of the Liquid Limit device. Do not use equipment that is not comply with the IRM tolerances, found on the IRM log.

SAMPLE PREPARTION (wet method)

1. If sample has little or no material retained on the #40 sieve, prepare specimen of 150-200 g by mixing thoroughly with distilled water on glass plate or mixing dish using a spatula. If desired, soak the material in a mixing/storage dish with a small amount of water to soften the material before the start of mixing. Adjust the water content of the soil and mix again to bring to a consistency that would require between 25-35 blows. Remove >#40 sieve material by hand if small percentage is present. If water has been added to specimen place in a covered dish and allow to cure for a minimum of 16 hours.

2. If large percentage of >#40 sieve material is present, obtain a large enough specimen which will contain 150-200 g of material passing the #40 sieve. Place specimen in mixing cup and cover with distilled water. Mix into slurry, pass specimen through a #40 sieve and discard >#40 material. Reduce the amount of water using the drying plate/vacuum pump apparatus. Do not allow sample to over dry. Dry until sample is at a consistency that would require between 25-30 blows. Water has been added to the specimen so place in a covered dish and allow to cure for a minimum of 16 hours.

SAMPLE PREPARATION (dry method)

1. If sample is received dry, pass the material through a #40 sieve, and adjust the moisture content and mix thoroughly to bring the soil to a consistency that would require between 25-35 blows. Place specimen in a covered dish and allow to cure for a minimum of 16 hours.

LIQUID LIMIT DETERMINIATION (multipoint method)

1. Before starting test, remix thoroughly in its dish adjusting the water content slightly, as necessary.

2. Fill the liquid limit cup even with front rim, to a depth that allows a full height cut with grooving tool. Keep the unused material covered with a wet towel in the mixing dish.

3. Form a groove in the soil from the highest point to the lowest point on the rim of the cup using the beveled edge facing forward of the grooving tool. Keep the tool perpendicular to cup from back to front by using an arc type motion. If the groove cannot be made in one stroke without tearing the soil, cut the groove in layers a little deeper with each cut, without tearing the soil.

4. Inspect the underside of the Liquid Limit Device, making sure the underside of the cup and base are clean. Lift and drop the cup at a rate of 1.9-2.1 drops per second until the two halves of the soil pat come in contact at the bottom of the groove along a distance of 0.5 inch. The base of the machine shall not be held with a hand while the crank is being turned. Record number of blows required. Take approximately 10 g of material from the closed area, perpendicular to the groove and place into a pre-weighed drying container. Measure and record mass to nearest 0.01 g. Cover the container.

5. Remove balance of material from cup and remix with soil in dish. Clean the cup.

6. By wetting the remaining material, adjust the blow counts so a distribution of blow counts is obtained. One point between 35 and 25 blows, another point between 20 and 30 blows, and a third point between 25 and 15 blows. For each point repeat steps 1 through 6.

PLASTIC LIMIT DETERMINATION

1. Obtain about 20 grams of soil prepared for the Liquid Limit test and dry by blotting with paper towels until it can be rolled without sticking to the glass plate.

2. To determine plastic limit, roll three to four threads of 1.5-2.0 grams of material down to a 1/8 inch diameter for a total sample weight of at least 6 grams.

3. Roll the material on a glass plate with the palm of your hand until a thread of uniform diameter of 1/8 inch is obtained (80-90 strokes per minute). This should take no longer than 2 minutes.

4. Ball up and repeat step 3 again. Repeat until 1/8 inch diameter cannot be obtained (thread crumbles under the pressure required for rolling). The only requirement for continuing is that the threads are able to be reformed into a ball and rolled out again.

5. Gather portions of the crumbled thread together and place into a pre-weighed container. Measure and record mass to nearest 0.01 g.

6. Repeat steps 2 through 5 one more time.

7. Dry all materials to a constant mass in oven with the temperature required for the material type being tested. Weigh again to the nearest 0.01 g.

8. Calculate the plasticity index as follows:

   PI = LL-PL

   where:

   PI = Plasticity Index
   LL = Liquid Limit
   PL = Plastic Limit

9. If either the liquid limit or plastic limit could not be determined, or if the plastic limit is equal to or greater than the liquid limit, report the soil as non-plastic.

10. Report: sample identification, as received moisture content, sample preparation procedure, liquid limit, plastic limit and plasticity index to the nearest whole number, and non-plastic if applicable.

Technical Information

Reference Number: ASTM D6913

Test Method Title: Standard Test Methods for Particle-Size Distribution Gradation) of Soils Using Sieve Analysis

Test Property: Grain Size Analysis for small samples that do not need to be split on the large Gilson screen shaker

Test Specimen Size: Depends on test samples maximum particle size. See ASTM D6913 Table 2 below. Sample size is generally on the order of 2 to 10 kg (3 to 22 lbs.)

D 6913 – 04$^{\varepsilon 2}$

TABLE 2 Minimum Mass Requirement for Specimen

Maximum Particle Size of Material (99 % or more passes)		Minimum Dry Mass of Specimen, g or kg[A]	
Alternative Sieve Designation	Maximum Particle Size, mm	Method A Results Reported to Nearest 1 %	Method B Results Reported to Nearest 0.1 %
No. 40	0.425	50 g	75 g
No. 10	2.00	50 g	100 g
No. 4	4.75	75 g	200 g[B]
⅜ in.	9.5	165 g[C]	[D]
¾ in.	19.0	1.3 kg[C]	[D]
1 in.	25.4	3 kg[C]	[D]
1-½ in.	38.1	10 kg[C]	[D]
2 in.	50.8	25 kg[C]	[D]
3 in.	76.2	70 kg[E]	[D]

[A] Specimen masses should not significantly exceed (by more than about 50 %) the presented values because excessively large specimens may result in sieve overloading, (see 11.3) and increase the difficulty of specimen processing.

Number of Test Specimens: 1 representative sample obtained by quartering, mixing or splitting

Test Equipment:

Coarse Sieve Set:
3 in., 2in., 1.5 in., 1 in., ¾ in., ½ in., 3/8 in.
Fine Sieve Set:
3/8 in., No. 4, No. 10, No. 20, No. 40, No. 60, No.100, No. 140, No.200
Washing Sieve, No. 200 (75-µm)
Washing Sink with Spray Nozzle
Mechanical Sieve Shaker
Balances (readable to 1 g and 0.01 g)
Drying Oven 110° ± 5° C
Sample and Specimen Containers
Sieve Brushes
Wash bottle
Spatula
Stirring Rod

Optional Items
Splitter/Riffle Box or Quartering equipment
Mortar and Rubber-Covered Pestle (for breaking up aggregations of dried soil.)
Low Temperature Drying Oven not to exceed 60°C
Sodium hexametaphosphate dispersant solution:
Make a solution by using 40 g of sodium hexametaphosphate and 1,000 g distilled, deionized, or demineralized water

Standard Operating Procedure

ASTM D6913 Method A – Composite Sieving (Use for samples containing particles larger than a #4 sieve.)

Sample Splitting:

1. Split sample on a 3/8 in. sieve.
2. Record the wet weight of the minus 3/8 in. fraction.
3. Record the weight of an empty tare.
4. Collect a 100–200 g representative sub-sample of the minus 3/8 in. fraction and place it in the tare weighed in step 3 above. This sample will be used as a moisture content sample and the fine sieve test sub-specimen. Record the wet weight of the [sub-specimen + tare] and oven dry at 110° ± 5° C.

Coarse Sieves:

1. Oven-dry the plus 3/8 in. fractions from Sample Splitting step 1 at 110° ± 5° C.

2. Sieve the plus 3/8 in. fraction on the standard coarse sieve set, which includes the following screen sizes:

   3 in., 2in., 1.5 in., 1 in., ¾ in., ½ in., 3/8 in.

   Additional sieves may be added as requested by the client.

3. Avoid overloading individual sieves. Refer to ASTM Table 3 below for maximum sieve loading. If necessary, shake overloaded sieves in multiple batches.

4. Shake the plus 3/8 in. fraction sample for a minimum 10 minutes. (The "Standard Shaking Period" is verified annually based on the procedure in ASTM D 6913.)

5. Cumulatively record the weights of the oven-dried material retained on each of the coarse sieves. Place the material, sieve by sieve, into a tared weighing container and record the cumulative weight. At least one sieve in the sieving process shall have 100 percent passing.

Fine Sieves:

1. Record the weight of the [oven-dried minus 3/8 in. sub-specimen + tare] dried in Sample Splitting step 4 above.

2. Disperse the minus 3/8 in. sub-specimen by soaking in water for at least 5 minutes before washing. Difficult samples can be dispersed using a sodium hexametaphosphate solution and/or an ultrasonic bath.

3. Wash the dispersed specimen on a #200 wash sieve, using tap water. Collect the wash water and dispose of the fine soil properly. Do not let the fines run down the drain.

   The material in the wash sieve may be lightly manipulated by hand, to facilitate the washing, taking care not to lose any of the soil. Do not exert any downward pressure on the wash sieve as this may damage the sieve.

   Fine material can clog the screen. Care should be taken not to let the wash sieve fill with too much water when clogging occurs. Prevent clogging by lightly tapping the sides of the sieve with your hand or tapping on the bottom of the screen with a fingertip.

4. Transfer the washed +200 in. fraction of soil back into the same tare and oven dry at $110^{o} \pm 5^{o}$ C.

5. Sieve the dried specimen using the standard fine sieve set, which includes the following sieve sizes:

   3/8 in., No. 4, No. 10, No. 20, No. 40, No. 60, No.100, No. 140, No.200

   Additional screens may be added as requested by the client.

6. Shake the sieve sample for a minimum 10 minutes. (The "Standard Shaking Period" is verified annually based on the procedure in ASTM D 6913.)

7. Avoid overloading individual sieves. Refer to ASTM Table 3 below for maximum sieve loading. If necessary, shake overloaded sieves in multiple batches.

8. Cumulatively record the weights of the oven-dried material retained on each of the fine sieves. Place the material, sieve by sieve, into a tared weighing container and record the cumulative weight. At least one sieve in the sieving process shall have 100 percent passing.

TABLE 3 Overloading Limits for Standard Sieve Set

Alternative Sieve Designation	Standard Sieve Designation	Number of Grain Layers on Given Sieve	Maximum Mass Retained on 200-mm (8-in.) Diameter Sieve, g[A]	Maximum Mass Retained on 305-mm (12-in.) Diameter Sieve, g	Maximum Mass Retained on 370- by 580-mm (14.6- by 22.8-in.) Sieve, g
3 in.	75 mm	0.8	2 700	6 100	18 000
2 in.	50 mm	0.9	2 000	4 500	13 000
1-½-in.	37.5 mm	0.9	1 500	3 400	10 000
1 in.	25 mm	1	1 100	2 500	7 000
¾ in.	19.0 mm	1	900	2 000	6 000
⅜ in.	9.5 mm	1.25	550	1 200	3 600
No. 4	4.75 mm	1.5	325	730	2 000
No. 10	2.00 mm	2	180	410	1 000
No. 20	850 µm	3	115	260	800
No. 40	425 µm	4	75	170	500
No. 60	250 µm	5	60	140	400
No. 100	150 µm	6	40	90	300
No. 140	106 µm	6	30	70	200
No. 200	75 µm	6	20	50	100

Calculations:

1. Use initial moisture content and initial wet weight of test specimen to calculate initial dry weight of test specimen. Use reporting software to enter data and calculate % passing and retained for each sieve size.

Report:

1. The datasheet shall include the following:

    - Project identification, boring number, sample number, depth,
    - Visual classification of the soil being tested
    - Test method used (Method A)
    - Sample preparation - moist, air dried, or oven dried, etc.
    - Any material excluded from the specimen.
    - Indicate that composite sieving was used and the size of the designated separating sieve.
    - If material is retained on the designated separating sieve size in finer sieve set, then document that the percent retained does not exceed 2 % criterion.
    - Indicate if the ultrasonic bath or shaking apparatus or both were used during the dispersion process.
    - Any prior testing performed on specimen
    - All mass measurements.
    - Tabulation of percent passing for each sieve to 1%.

2. The report shall include the following:

    - Project identification, boring number, sample number, depth,
    - Classification or visual classification of the soil being tested
    - Any material excluded from the specimen.
    - Any prior testing performed on specimen
    - A graph of the percent passing versus log of particle size in mm

ASTM Comments:

Some materials that are not soils, but are made up of particles may be tested using this method.

This test method is ***not*** applicable for the following soils:

1. Soils containing fibrous peat that will change in particle size during the drying, washing, or sieving procedure.
2. Soils containing extraneous matter, such as organic solvents, oil, asphalt, wood fragments, or similar items. Such extraneous matter can affect the washing and sieving procedures.
3. Materials that contain cementitious components, such as cement, fly ash, lime, or other stabilization admixtures.

This test method may **not** produce consistent test results within and between laboratories for the following soils:

1. Friable soils in which the sieving processes change the gradation of the soil. Typical examples of these soils are some residual soils, most weathered shales and some weakly cemented soils such as hardpan, caliche or coquina.
2. Soils that will not readily disperse such as glauconitic clays or some dried plastic clays.

To test these soils, this test method must be adapted, or altered, and these alterations documented.

Technical Information

Reference Number:	ASTM D7263-09
Test Method Title:	Standard Test Method for Laboratory Determination of Density (Unit Weight) of Soil Specimens
Test Property:	Density
Test Specimen Size:	Minimum dimension of 1.3 inches and largest particle should be smaller than 1/10 of the specimens (container) smallest dimension.
Number of Test Specimens:	1
Test Equipment:	Drying Oven capable of maintaining 110 ± 5 °C, Balance, readable to 0.01 gram with 500 gram capacity Balance, readable to 1 gram with 12000 or more gram capacity for larger samples Ruler Calipers pi tape Trimming & carving tools Wire saw Soil miter box Containers of known volume, for remolding Wax, non-shrinking type, of known specific gravity Containers to melt wax (water bath type) Paintbrush Hot Plate Thermometer Wire Basket, with hanging apparatus Container for specimen submerging

Standard Operating Procedure

METHOD A: Water Displacement

Intact Specimens

1. Determine the density of the Wax to be used to four significant digits, if unknown.

2. Prepare specimen by trimming to a fairly regular shape. Cavities present should be patched with material from the trimmings, when necessary. Handle specimen carefully in a high humidity environment.

3. Record the mass of the specimen to four significant digits.

4. Cover the specimen with a thin coat of melted Wax, either with a paint brush or by dipping. Apply a second coat when first coat has hardened. Keep the Wax melted but not too hot, so as not to dry out the specimen.

5. Record the mass of the Wax-coated specimen to four significant digits.

6. Attach wire basket underneath the balance to be used and submerge the wire basket in water and zero out the balance.

7. Place Wax-coated specimen in wire basket and record the submerged mass of the Wax-coated specimen to four significant digits.

8. Record the temperature of the water to one decimal place, which is used to submerge the specimen.

9. Remove the Wax from the specimen.

10. Determine the water content of specimen (by ASTM D2216), do not include any Wax in your water content specimen.

Calculations:

11. Calculate the moist density to four significant digits as follows:

$$\rho_m = M_t/[((M_c - M_{sub})/\rho_w) - ((M_c - M_t)\rho_\pi)]$$

Where:

M_t = mass of moist/total soil specimen, g
M_c = mass of wax-coated specimen, g
M_{sub} = mass of submerged wax-coated specimen, g
ρ_p = density of wax, g/cm^3
ρ_w = density of water corrected for the temperature (see ASTM D854 for values)
ρ_m = density of total (moist) soil specimen, g/cm^3

12. Calculate the dry density to four significant digits as follows:

$$\rho_d = (\rho_m / (1+w))$$

Where:

ρ_d = dry density of soil specimen, g/cm^3
w = moisture content in decimal form

METHOD B: Direct Measurement

<u>Intact Specimens</u>

1. Prepare intact specimens from a block or similar sample or obtain specimen from undisturbed tube. If carving from a block, use hand tools to obtain a cylindrical specimen. Handle samples and specimens carefully to avoid disturbance. May also perform test on an un-extruded tube sample as long as ends are cut plane and perpendicular to the longitudinal axis. The height and inner dimensions of the tube may be used as the specimen dimensions.

2. Trim specimens in an enclosure that minimizes loss of moisture. If removal of gravel is necessary, fill voids with remolded soil taken from trimmings.

3. Once uniform dimensions are achieved, place cylindrical specimen in miter box and cut the specimen to a uniform height with wire saw or knife.

4. Use trimmings to obtain water content (by ASTM D2216).

5. Determine mass of specimen to 4 significant digits and record.

6. Measure the height of the specimen at three locations (rotating 120° each time). Record each reading and average.

7. Measure the diameter of the specimen at three locations (top, middle and bottom). Record each reading and average.

Remolded/Reconstituted Specimens

8. Prepare specimen as prescribed by test method, client or both.

9. After specimen is made, trim ends perpendicular to the longitudinal axis (if necessary).

10. Remove specimen from mold.

11. Determine mass, height and diameter as prescribed above in steps 5, 6 and 7. The inner dimensions of the mold may be used as the specimen dimensions.

12. For specimens that bleed water, containers of known mass and volume should be used.

13. Compact soil into container of the size that will satisfy the max particle size requirement and strike off excess material. Use compaction force as requested.

Calculations

14. Calculate dry mass of the soil sample in grams with the following equation:

$M_3 = [(M_1-M_2)/(1 + w/100)]$

where:

M_1 = mass of the cylinder and wet soil sample, g (if cylinder was not used, use only mass of wet soil)
M_2 = mass of the cylinder, g (if cylinder not used, value should be 0.00)
M_3 = mass of the dry soil sample, g
w = moisture content, % as dry mass basis

15. Calculate the dry density of the sample in lbs/ft^3 as follows:

$\rho_d = (M_3/V)(3.810)$

where:

ρ_d = dry density of soils, lbs/ft^3
V = volume of the cylinder, in^3

16. Calculate the bulk (wet) density of the sample in lbs/ft^3 as follows:

$\rho_w = (\rho_d \times (1+w))$

where:

ρ_w = wet density of soils, lbs/ft^3

15. Report: sample identification, depth below ground surface, bulk density, dry density, moisture content, dimensions and volume of cylinder, and visual description of soil.

Standard Operating Procedure **ASTM D7928**

Technical Information

Reference Number:	ASTM D7928
Test Method Title:	Test Method for Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis
Test Property:	Grain Size Analysis
Test Specimen Size:	Passing #10 sieve at least 50 g soil, containing no more than 60 g and no less than 15 g of minus #200 material. Amount varies see test standard Table 1 (based on largest particle size)
Number of Test Specimens:	Two representative samples obtained by quartering, mixing or splitting. One for grain size analysis and one for a representative moisture content determination.
Test Equipment:	Hydrometer (ASTM 152H –reads in grams of soil in suspension) Sedimentation Cylinder Sedimentation Cylinder Covers Stirring Apparatus (blender) Dispersion Cup Drying containers Balance readable to 0.01 gram Thermometer readable to 0.5 ºC Timing device Various sieves 250 mL glass beaker Drying oven capable of maintaining a temperature of 110 ± 5 ºC Dispersing agent mixture (40 g/L of Sodium Hexametaphosphate solution) Distilled Water Isopropyl alcohol Spray Bottle Wash pan Spatula

Standard Operating Procedure

Sampling

1. Collect a representative sample and perform a moisture content test in accordance with ASTM D2216. Enter data into the unique LIMS test file for this sample.
2. Collect another representative sample to be used for the particle size analysis. Base specimen size on test standard (based on largest particle size). Record empty beaker ID and weight. Record the weight of the wet specimen and beaker. Enter data into the unique LIMS test file for this sample.

   (Note: the two samples described above must be similar in grain size distribution and moisture content)

Splitting / Washing sample on #200 sieve

3. Add 125 ml of dispersing agent mixture or 5 g +/- 0.1 g of dry dispersing agent with at least 100 ml of distilled water to the test sample. Stir with spatula until all of the soil aggregations are broken-up.

   (Note: do not spill or lose any soil particles from the sample, rinse all surfaces off and back into sample)

4. Rinse sample into dispersion cup and use blender to further disperse sample for 1 minute.

5. Wash the test specimen from the dispersion cup, using distilled water, over the No. 200 sieve into a container. Be sure to collect all washings in the container. Use only 800 ml of distilled water for the washing operation.

6. Transfer the portion retained on the No. 200 into a tare and place in a drying oven. The dry weight will be needed for data entry and used in calculations for reporting. In addition, this portion could continue on for further grain size testing following any of a number of test standards for a full grain size analysis, upon request.

7. Wash the minus No. 200 sieve material into a Sedimentation cylinder and record ID of cylinder in the unique LIMS test file for this sample.

8. Add distilled water so the bottom of the meniscus is at the 1000 mL line of the Sedimentation cylinder. Place a rubber stopper securely (so no leaks or spills occur) over the open end and turn the cylinder upside down and shake in a manner that loosens all of the soil from the bottom and forms a slurry. Turn the cylinder upside down and back for a period of 1 minute (should be 60 turns per minute). Set the cylinder down.

9. Cover the cylinder, to prevent evaporation and allow the test specimen to sit overnight. This is the conditioning period which allows the temperature to equilibrate and the specimen to deflocculate.

Hydrometer Analysis of portion passing #200 sieve:

10. Using the Hydro Read Program of GTX LIMS system bring up this sample's unique test file and prepare to start this portion of the test.

11. Place a rubber stopper over the open end of the Sedimentation Cylinder and give the sample a pre-shake to loosen it from the bottom and make a slurry of the entire sample again.

12. Start the clock in the Hydro Read LIMS software. This will countdown the shake and time the test. Now turn the cylinder upside down and back for a period of 1 minute (should be 60 turns per minute) in coordination with the LIMS timer prompts. Set the cylinder down for sedimentation on a stable surface free from jarring and vibrations.

13. Remove stopper but DO NOT wash any adhering soil into the cylinder, let drips fall is necessary. If foaming on the surface occurs, spray with isopropyl alcohol to remove.

14. Begin to take and record hydrometer readings at the following intervals, using the LIMS timing prompts in the software: 1, 2, 4, 15, 30, 60, 120, 240, and approximately 1440 minutes. Enter your data into the unique LIMS test file for this sample

15. After the hydrometer reading, the temperature of the solution should be recorded. You may use the same temperature for the first few readings, and then for 30 min and those following take the temperature each time a reading is taken. Enter your data into the unique LIMS test file for this sample.

Report:

16. Enter all data required in the Hydrometer Header Data portion of the unique LIMS test file for this sample. The LIMS software will generate a complete grain size distribution report with tabulated and graphical data representations.

17. Record the following in the unique LIMS test file for this sample: Sample ID information including: Boring, Sample and Depth, Visual Description of sample including Munsell Book color and an initial "best guess" of grain sizes, Technician that performed each part of test, Balance ID, Oven ID, Thermometer ID, Separating Sieve ID and Sieve size, Wash Sieve ID, Hydrometer ID and type, Specimen Prep Method, Type of dispersant, Describe any excluded material, Describe any problems encountered and Indicate any prior testing on this specimen and if foam inhibitor was used.

18. Update your initial "best guess" sample description to correctly reflect the grain size descriptors corresponding to the as-tested data set following ASTM D2487 as guidance.

19. Calculate the % finer than the #200 sieve and add a sample comment to the unique LIMS test file as follows: Percent Passing #200 Sieve = 92%

TITLE: Manual Chromatographic Peak Integration

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Draft Preparation	R.D. Foster	02/2007
1	Update and Revision	R.D. Foster	02/2007
2	Review and SOP number change from 8000A to 1132	M. Scott	04/2011
3	Review and Update	M. Scott	01/2013
4	Review, update, and reformatting	A. MacKinnon	06/2016

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1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) summarizes integration processes analysts use to integrate peak areas manually using chromatographic software. This procedure is used by analysts involved with Gas Chromatography (GC), High Performance Liquid Chromatography (HPLC), Ion Chromatography (IC) and Gas Chromatography / Mass Spectrometry (GC/MS) data, and occasionally in flow-injection analysis. This procedure applies to any original integration of data by the analyst or any re-integration of data initially processed automatically.

The purpose of the SOP is to emphasize the need to oversee automated integration and assure that both manual and automated integration results in technically defensible data only. Modification of integration that is not technically defensible and is solely for the purpose of meeting quality control criteria are strictly not allowed.

2.0 Definitions

Analyte Response Peak: Analytical response versus time produces an analyte response peak. The height or area of the peak is the basis of quantitation for that analyte.

Automated Integration: The process by which algorithms in computer chromatographic software are used to detect peak start point and endpoint and determine the peak area or height used for quantitation.

Manual Integration: The process by which an analyst sets the start and stop points for the baseline of a peak during reprocessing and quantitation.

Chromatogram: A plot relating the GC detector response verses time from injection.

Baseline: The detector response vs time due only to detector background and column bleed.

Noise: Fluctuations in detector response not due to analytes.

See ESI's Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

US EPA. 2014. *Test Methods for Evaluating Solid Waste, Method 8000D Determinative Chromatographic Separations.* SW-846. 5th Edition.

Department of Defense (DoD), Department of Energy (DoE). 2013. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.0.

4.0 Materials and Apparatus

Agilent Technologies. 1989-2001. *G1701DA Version D.00.00.38 19-Nov 2001.* Environmental

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ChemStation.

Agilent Technologies. 1989-2001. *Rev. A.10.01 [1635] 1990-2003.* GC ChemStation.

Microsoft Corp. 1990-1995. *Version 4.00.950.* PeakNet.

5.0 Methods and Procedures

Due to the complex nature of both organic analytes and environmental samples, it is ESI's policy to assure a skilled analyst assigns peak areas in all cases.

5.1 Peak Integration

Whenever reasonable, the analyst may allow the analytical systems software automatic integration to assign area response values. The analyst must inspect any automatically assigned values for correctness. If automatically assigned values are deemed incorrect then manual integration may be preformed to accurately represent the area of the analytes present. If manual integration is deemed necessary, a file containing the automatic integration values must be generated and saved.

5.2 Manual Integration

5.2.1 The following scenarios describe why manual verification or reassignment of peak area are required:

- It is expected that sample chromatograms are complex and have irregular baselines that require a skilled analyst's interpretation.
- Suitable system software is not available or is unreliable.
- Peaks are split or improperly defined by chromatography software.
- Complex peak patterns exist, such as partially resolved peaks, or peaks that are shoulders on other peaks.
- Baseline noise prevents proper peak identification.
- There are negative or positive upsets in the baseline.
- There are rising or falling baselines.
- The wrong peak is identified by the chromatographic software.
- Other cases should be approved by the Laboratory Manager prior to use.

5.2.2 Manual integration must consider the chromatographic baseline and the start and stop points of the peak along the baseline. Estimate the average noise level of the baseline. Set the start and stop points for the peak at the average noise level of the baseline.

5.2.3 Each case of manual integration should be flagged in the raw data with the corresponding qualifier:

- m - A baseline or shoulder adjustment
- m1 - The peak was missed
- m2 - The wrong peak was assigned

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- m3 - The peak is co-eluding

5.2.4 Manual integrations must be implemented using a consistent approach.

5.3 Integration Techniques

Examples of improperly and properly integrated peaks with descriptions and appropriate qualifier codes are diagramed and attached as Figures 1-4. Examples of improperly and prohibited integrated peaks are diagramed and attached as Figure 5.

6.0 Quality Control Requirements

6.1 The manually integrated peak must be qualified with the corresponding qualifier on the quantitation report for peer review. The qualifiers are listed above in section 5.2.3.

6.2 Each quantitation report including both manual and automatic integrations should be initialed and dated by the analyst.

6.3 The automatic integrations must be maintained so that comparisons between manual and automatic integrations can be reviewed.

7.0 Calculations and Reporting

Manually integrated peaks must be qualified on the quantitation report for peer review.

8.0 Corrective Actions

This section is not applicable to this Standard Operating Procedure.

9.0 Health and Safety

This section is not applicable to this Standard Operating Procedure.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptable criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts preforming this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities.

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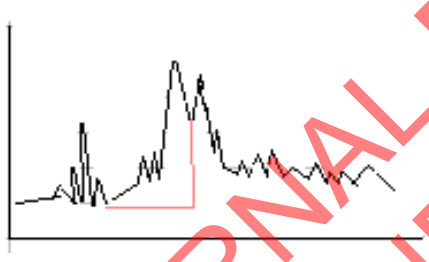
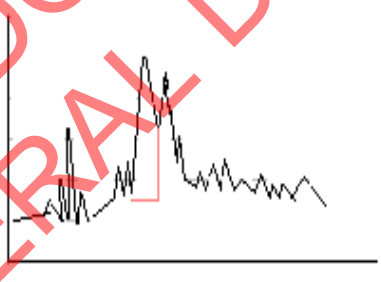
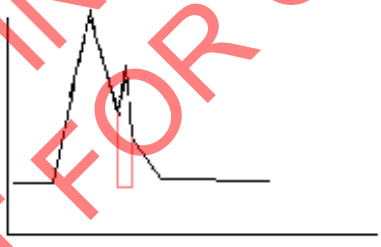
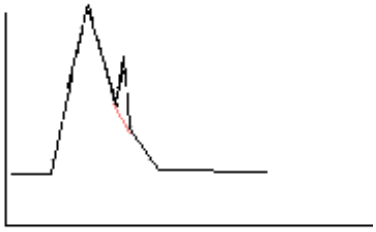
10.3 Caution is required when using automated integration, particularly when the integration affects method compliance. Each automated integration must be verified by the analyst, corrected as necessary, and documented in the data.

10.4 Quality Assurance personnel will examine chromatographic data during internal method audits to determine compliance with the procedures in this SOP.

11.0 Waste Management and Pollution Prevention

This section is not applicable to this Standard Operating Procedure.

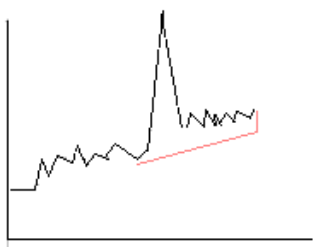
Figure 1. Qualification: m - baseline or shoulder adjustment

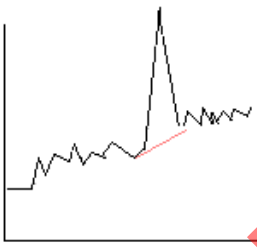
Incorrect Integration	Correct Integration
	
Description: Baseline correction for noise interferences	
	
Description: Baseline correction for peak shoulders	

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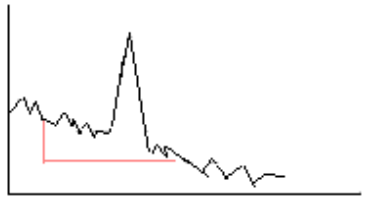
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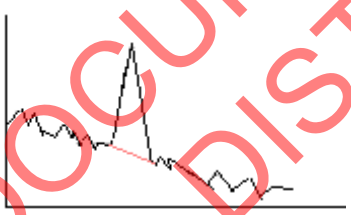
TITLE: Manual Chromatographic Peak Integration



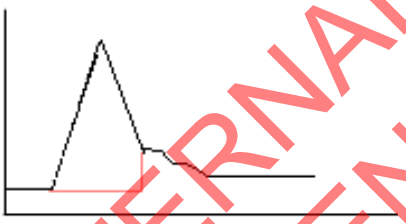


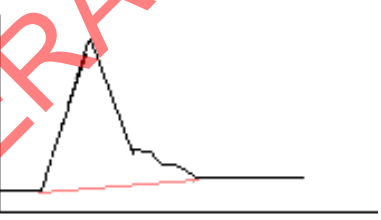
Description: Baseline correction for rising noise



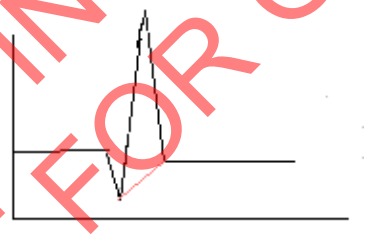


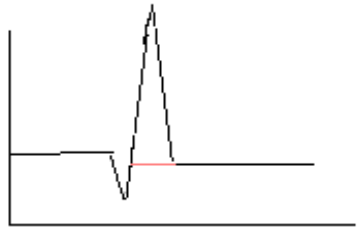
Description: Baseline correction for falling noise





Description: Baseline correction for excessive peak tailing.





Description: Baseline correction for negative peak.

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Figure 2. Qualification: m1 - the peak was missed

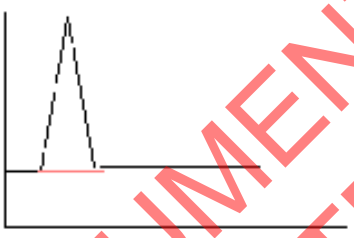
Incorrect Integration	Correct Integration
	

Figure 3. Qualification: m2 - the wrong peak was identified

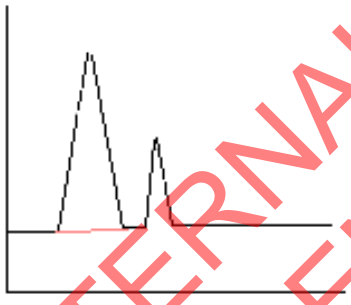
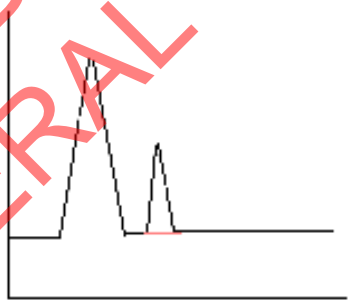
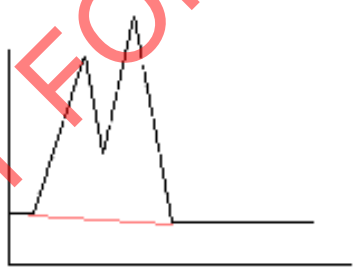
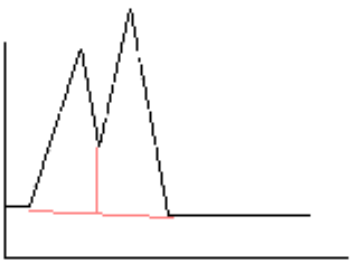
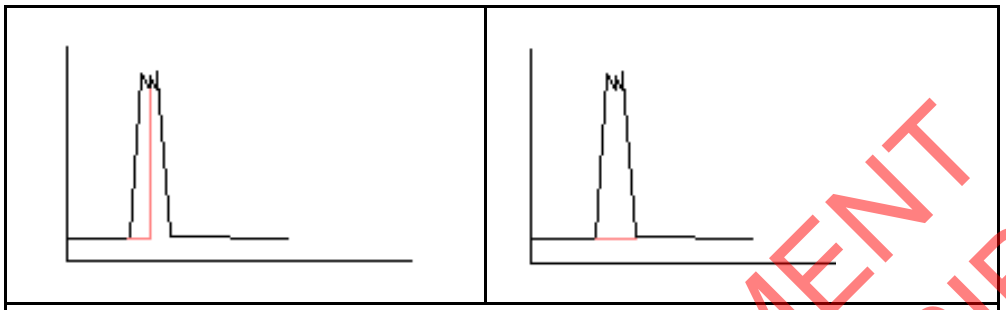
Incorrect Integration	Correct Integration
	

Figure 4. Qualification: m3 - co-eluting peaks

Incorrect Integration	Correct Integration
	
Description: Overlapping peaks	

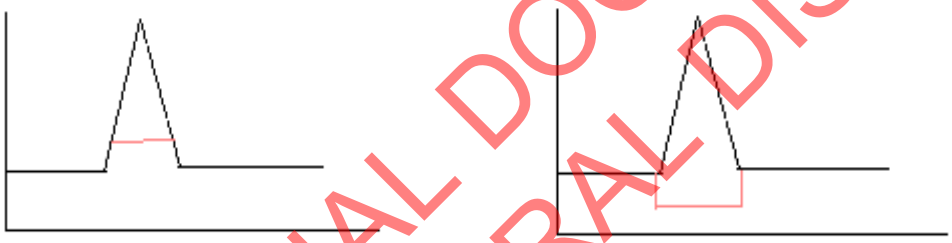
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Description: Split peaks (due to noise)

Figure 5. Incorrect Prohibited Manual Integrations



Integration Description: Above the baseline Integration Description: Below the baseline

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TITLE: Analysis of LOD and LOQ Quality Control Samples

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	M. Scott	11/2010
1	Revision adding statement regarding use of data qualifier for values below LOQ plus general formatting update	K.A. Simon	01/2016

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TITLE: Analysis of LOD and LOQ Quality Control Samples

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) outlines the method for analyzing and evaluating Limit of Detection (LOD) and Limit of Quantitation (LOQ) quality control samples in any matrix.

The LOD is a quality control sample spiked at a level above the method detection limit but below the reporting limit to verify the method detection limit. The LOQ is a quality control sample spiked at a level no more than one to two times the reporting limit. Both quality control samples are processed through the method the same as a field sample would be treated. The LOD must be sufficiently higher than the signal-to-noise ratio or the mean method blank concentration. The LOQ must demonstrate precision and bias at the LOQ by meeting the acceptance criteria.

2.0 Definitions

Limit of Detection (LOD): The smallest concentration of a substance that must be present in a sample in order to be detected at the MDL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. An LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.

Limit of Quantitation (LOQ):The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range. LOQ acceptance criteria are based on limits set by the method or in the cased of DoD projects they may be found in Appendix C of QSM 5.0. Program specific LOQs may also be set in the SOP.

Method Detection Limit (MDL): The minimum concentration of a substance that can be measured and reported with 95% confidence that the analyte concentration is greater than the blank and is determined from analysis of a sample in a given matrix containing the analyte.

Reporting Limit: The lowest value that can be reported while maintaining QC criteria; a responsive value that is the greater of the MDL, lowest calibration value, or lowest value required by the protocol or program.

3.0 Applicable Documents and References

Department of Defense (DoD), Department of Energy (DoE). 2013. *DoD Quality Systems Manual (QSM) for Environmental Laboratories.* Version 5.0.

The NELAC Institute (TNI). 2009. *Environmental Laboratory Sector Volume 1: Management and Technical Requirements for Laboratories Performing Environmental Analysis* (TNI Standard).

4.0 Materials and Apparatus

Appropriate standards
Milli-Q® water, or other appropriate substance

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5.0 Methods and Procedures

5.1 Limit of Detection (LOD) Determination

An LOD sample shall be prepared using Milli-Q® water, or another substance known to be free of contaminants for matrices other than water, and a standard containing the analyte of concern.

For a single-analyte standard, the LOD sample shall be spiked at a concentration approximately 2 to 3 times the determined method detection limit (MDL) and no higher than the reporting limit. For a multi-analyte standard, the LOD sample shall be spiked at a concentration approximately 1 to 4 times the MDL and no higher than the reporting limit.

The sample shall be processed through the selected method as if it were a field sample and evaluated against the acceptance criteria. Where more than one instrument or method is used, the LOD must be verified on each instrument or method.

5.2 Limit of Quantitation (LOQ) Confirmation

An LOQ sample shall be prepared by using Milli-Q® water, or another substance known to be free of contaminates for matrices other than water, and a standard containing the analyte of concern. The LOQ sample shall be spike at a concentration no more than 1 to 2 times the reporting limit.

The sample shall then be processed through the selected method as if it were a field sample and evaluated against the acceptance criteria. Where more than one instrument or method is used the LOQ must be verified on each instrument or method.

6.0 Quality Control Requirements

QC elements may be added or removed to meet specific program or project requirements.

6.1 Method Detection Limit (MDL)

The MDL is calculated using the appropriate Student's t-parameter value multiplied by the standard deviation of 7 or more replicates of spiked samples or standards. MDLs are performed annually and with changes in instrument or method. The t-parameter value is from a one-tailed Student's t-Test at the 99% confidence interval using n-1 degrees of freedom, with "n" being the number of replicates.

The calculated MDL must be lower than the reporting limit.

6.2 Limit of Detection (LOD)

LOD determination will be conducted on a quarterly basis. The LOD is a single quality control sample performed according to the customer specified protocol. The apparent signal-to-noise ratio at the LOD must be at least a factor of 3. For methods that do not produce a signal-to-

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noise ratio, the LOD result must be at least 3 standard deviations greater than the mean method blank.

6.3 Limit of Quantitation (LOQ)

LOQ confirmation will be conducted on a quarterly basis. The LOQ is a single quality control sample performed according to the customer specified protocol. If the analysis is analyzed under DoD ELAP requirements, then it is performed within the limits specified in Appendix C, QSM 5.0, for the specific parameter/method.

The LOQ must pass the acceptance criteria listed in the SOP or as specified by the client for the analyte being tested. For studies conducted under DoD ELAP guidance, acceptance will be as specified in Appendix C of QSM 5.0.

6.4 See individual method SOPs for possible interferences.

7.0 Calculations and Reporting

7.1 Unless otherwise requested, ESI reports values below the LOQ as Not Detected ("ND") or as the reporting limit qualified with Undetected ("U"). If a client or program requests or requires reporting of values between the LOQ and the LOD, then values falling in the range between the LOQ and LOD will be qualified appropriately and defined in the report.

7.2 See individual method SOPs for a list of all data recorded for the analyte being evaluated.

7.3 Calculations are performed as defined in the individual method SOPs.

8.0 Corrective Actions

8.1 If an MDL, LOD or LOQ does not meet acceptance criteria, then the analysis should be repeated until acceptance criteria have been met.

8.2 In the event that a LOD continually fails the acceptance criteria, then repeat the MDL and the LOD studies at higher concentrations. Alternatively, perform two LOD samples at a higher concentration (if it will be below the reporting limit) and set the LOD to that higher concentration.

9.0 Health and Safety

As with all samples, gloves and safety glasses should be worn when handling samples and chemicals. It is advisable to wear a lab coat to protect clothing.

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10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP.

10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities.

11.0 Waste Management and Pollution Prevention

Sample disposal is conducted using procedures to minimize potential pollution of surface and ground waters, soil and sediments. Unused portions of samples are disposed of only after completion of sample analysis, submission of the final report and contracted sample retention times have been achieved. All conditions of disposal and all records and correspondence concerning the final disposition of the physical sample shall be recorded and retained. Records shall indicate the date of disposal, the nature of disposal and the name of the individual who performed the task.

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TITLE: Analysis of LOD and LOQ Quality Control Samples

Appendix. Example data for Hexavalent Chromium

	A	B	C	D	E	F	G	H
	LAB ID	MATRIX	RESULT	TRUE	QLIMIT	QUAL	UNITS	METHOD
1	MDLA2010	Water	0.012	0.01	0.005		mg/L	SM 3500-Cr B
2	MDLB2010	Water	0.014	0.01	0.005		mg/L	SM 3500-Cr B
3	MDLC2010	Water	0.016	0.01	0.005		mg/L	SM 3500-Cr B
4	MDLD2010	Water	0.012	0.01	0.005		mg/L	SM 3500-Cr B
5	MDLE2010	Water	0.014	0.01	0.005		mg/L	SM 3500-Cr B
6	MDLF2010	Water	0.012	0.01	0.005		mg/L	SM 3500-Cr B
7	MDLG2010	Water	0.012	0.01	0.005		mg/L	SM 3500-Cr B
8	PB099W	Water	0.0	0.0	0.005		mg/L	SM 3500-Cr B
9	PB098W	Water	0.0	0.0	0.005		mg/L	SM 3500-Cr B
10	PB097W	Water	0.0	0.0	0.005		mg/L	SM 3500-Cr B
11	PB096W	Water	0.002	0.0	0.005		mg/L	SM 3500-Cr B
12	PB095W	Water	0.0	0.0	0.005		mg/L	SM 3500-Cr B
13	LODA2010	Water	0.0038	0.0045	0.005		mg/L	SM 3500-Cr B

- Calculate the MDL as follows: @STD(C1..C7)*3.14

  MDL = @STD(0.012, 0.014, 0.016, 0.012, 0.014, 0.012, 0.012) * 3.14 = 0.004575

- Calculate 3 standard deviations of the blank concentrations as follows: @STD(C8..C11)*3

  @STD(0, 0, 0, 0.002, 0) * 3 = 0.0024

- Calculate the mean blank concentration as follows: @AVG(C8..C11)

  Mean = @AVG(0, 0, 0, 0.002, 0) = 0.0004 mg/L

- Determining the LOD concentration:

  The LOD quality control sample must be prepared at a level greater than 0.0028 mg/L (0.0024 + 0.0004) and less than the reporting limit of 0.005 mg/L. The MDL is 0.004575 mg/L so a standard prepared at 0.0045 mg/L would be an appropriate selection.

- Evaluating the LOD:

  If a standard at 0.0045 mg/L produced a result of 0.0038 mg/L the LOD would be verified. The LOD sample produced a result that was at least three standard deviations greater than the mean blank concentration (0.0028mg/L).

- Determining the LOQ concentration:

  The LOQ must be at least twice the reporting limit. The LOQ is 0.01, the MDL standard.

- Evaluating the LOQ:

  LOQs that fall within +/- 3 Std Dev of historic mean, or other specified method limit, are acceptable. For DoD programs, QSM 5.0 Appendix C provides acceptable LOQ ranges by method and matrix.

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TITLE: Sample Preparation; Trace Metals Digestion

Approved By:

QA Officer: _____ Date: _____

Technical Director: _____ Date: _____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	K. A. Simon	3/1995
1	Review and Update	S. Dionne	06/2000
2	Review and Update	M. Folsom	05/2006
3	Review and Update	R Foster	10/2007
4	Review and Update	M. Scott	05/2009
5	Review and Update	M. Scott	12/2009
6	Reformat and Update	M. Scott	12/2011
7	Update based on audit responses	M. Scott	03/2012
8	Update based on internal audit	M. Scott	01/2015

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TITLE: Sample Preparation; Trace Metals Digestion

1.0 Purpose and Applicability

This SOP describes the method of preliminary digestion to reduce interference by organic material and convert metals associated with particulate that can be determined by inductively-coupled plasma. This procedure uses the least rigorous digestion required to provide complete and consistent recovery compatible with the metal being analyzed.

2.0 Definitions

NA

3.0 Applicable Documents/References

APHA. 1995. Standard Methods for the Examination of Water and Wastewater, 19th Edition. Methods 3030D,3030E, and 3030F.

200.8 Determination of trace elements in waters and wastes by inductively coupled plasma - mass spectrometry 1994.

ESI SOP # 1324 and 1369.

4.0 Materials and Apparatus

4.1 Reagents/Standards

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date.

Milli-Q®

Ultra-Trace Metals Grade Nitric Acid (HNO_3), Concentrated and 1:1

Ultra-Trace Metals Grade Hydrochloric Acid (HCl), Concentrated and 1:1

Commercially Available Single- or Multi-Element Standards, in both “high” and “low” concentrations, or Ultra-High Purity Compounds

4.2 Equipment and Supplies:

See ESI SOP #1324 for proper cleaning techniques

250 mL beakers, Pyrex, Teflon, or polypropylene
50 mL polyethylene digestion tubes
100 mL graduated cylinders polyethylene, or polypropylene

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50 mL graduated cylinders polyethylene, or polypropylene
Adjustable Volumetric pipets
Watch glasses pyrex, teflon, or polypropylene
Funnels, polyethylene, teflon, or polypropylene
Filter paper - Qualitative Grade P5, 12.5 cm, acid washed
Hot plate, Teflon or ceramic coated, calibrated for use at 96 C, for use in non metal-fume hood fitted with particle filter control
Polyethylene laboratory countertop lab furnishing
Pipet tips, verified to be metals-free
Thermometer calibrated in 70° C - 100° C range
Analytical Balance
Filter paper - Qualitative Grade P8, 12.5 cm, acid washed before use.
FilterMate® 2µm PTFE Certified Filter
Trace Metals Grade Hydrogen Peroxide, 30%

5.0 Methods/Procedures

Samples should be collected in 250-500mL plastic disposable bottles. Samples received for metals are preserved with HNO_3 to a pH $\leq$2 SU and have a holding time of 180 days.

5.1 Nitric Acid Digestion

Transfer a measured volume of well-mix, acid-preserved sample to a clean beaker. Record initial volume. General sample size is 100 mL.

Note: If sample contains undissolved solids, an aliquot containing no more than 1g solids should be used.

Add 3 mL of concentrated HNO_3 1 mL of concentrated HCl to sample (alternatively 5 mL of concentrated HNO_3 could be used).

Place beaker on a hot plate, or in a steam bath until the volume is reduced to 20 mL (four to five hours per 100 mL). The sample must not be allowed to boil or go dry. The beaker can be covered with an elevated watch glass.

If suspended solids remain, the sample may be filtered, allowed to settle, or centrifuged.

Cool and dilute to final volume with Milli-Q® water, mix well. Rinse the beaker 3 times as you bring the sample to final volume. Record the final volume. Final volume is typically 100 or 50 mL, but may vary based on the quantitation limit requirements of the program or project-specific data quality objectives.

5.2 Total HNO_3 / HCl

Transfer a measured volume of well-mix, acid-preserved sample to a clean beaker. Record initial volume. Generally, a sample volume of 100 mL is used. If a sample contains suspended solids, an aliquot containing no more than 1g solids should be used.

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Add 3 mL concentrated Nitric Acid to the sample. Add spiking solutions appropriate to program or project-specific data quality objectives.

The beaker can be covered with an elevated watch glass. Place beaker on a hot plate, or in a steam bath until the volume is reduced to near 5 mL The sample must not be allowed to boil or go dry.

Cool the sample and add 5 mL of concentrated HNO_3. Cover with a watch glass and return the beaker to the hot plate. Increase temperature to allow for a gentle reflux action. Continue heating and adding acid until the sample has a light color and does not change in appearance. Evaporate to <5 mL and cool. Add 10 mL of 50% HCl and 15 mL of Milli-Q® water. Heat an additional 15 minutes. Cool, wash down beaker walls and watch glass cover into sample with Milli-Q® water.

Dilute the sample to the required final volume (typically 50-100 mL) with Milli-Q® reagent water and mix well.

Allow any undissolved solids to settle over night, or centrifuge a portion of the sample until clear. If suspended solids remain, the sample may be filtered. Pre-rinse the filter paper with Milli-Q® water and 2-5% HNO_3.

5.3 Solids Sample Preparation

Mix sample thoroughly and transfer a portion containing close to 1 gram dry weight, using the percent moisture determined from a separate aliquot of sample into a 50 mL digestion tube. Record sample initial wet weight taken.

Add 5 mL of concentrated HNO_3 and 5 mL of Milli-Q. Add spiking solutions appropriate to program or project-specific data quality objectives.

Place the digestion tube on a hot plate, or in a steam bath, covered with a watch-glass. Heat the sample for ½ hour then add 5 mL of HCl to the samples.

Heat the sample and gently reflux until the volume has gone down to about 4mL. Gentle boiling may take place, however rapid boiling must be avoided.

If the sample has a high organic content, up to 2 mL of hydrogen peroxide may be added to the sample over the course of digestion.

If suspended solids remain, the sample may be filtered through filter paper pre-rinsed with 1% HNO_3 or a certified FilterMate® may be used.

Cool and bring to final volume with Milli-Q® water. Final volume is based on program or project-specific quantitation limit requirements. Record the final volume.

6.0 Quality Control Requirements

The objective reporting limits vary from 1 to 0.0005mg/L.

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6.1 QC Elements

Demonstration of capability (DOC), four replicates of a prepared quality control sample. Required with a change of instrument, method, or analyst.

Method detection limit (MDL) study, seven replicates (standard deviation * 3.14). Performed annually, with change in instrument, or method.

Limit of quantitation (LOQ), a single quality control sample, no more than 1-2x the reporting limit. Performed annually.

Analyze a blank, laboratory control, sample spike, sample spike duplicate, and if desired a laboratory control sample duplicate every analytical batch or twenty samples, whichever is more frequent.

QC elements may be added or removed to meet specific program or project requirements.

6.2 Acceptance Criteria

Demonstration of capability samples must meet LCS acceptance criteria.

The calculated MDL must be lower than the reporting limit.

The limit of quantitation standard acceptance criteria is 70-130%.

Acceptance criteria for all blanks is less than the reporting limit or less than 1/10 of sample value.

Acceptance criteria for LCS/LCSD recovery is 85%-115%. Acceptance criteria for LCSD percent relative range is less than or equal to 30%.

Acceptance criteria for relative percent difference between sample duplicates is less than or equal to 30%.

Acceptance criteria for spiked sample recovery is 80%-120%.

Alternate acceptance criteria may be set for specific programs or projects.

7.0 Calculations/Reporting

7.1 Record

Record the following:

1. Date
2. Initials of analyst
3. Sample ID
4. Batch
5. Sample Code / Standard Code

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6. Acid lot ID
7. Spike ID and amount , with dilution factor if applicable
8. Initial volume
9. Final volume
10. Method
11. Notes, if any

7.2 Calculations

See ESI SOP #1369 Determination of Trace Metals by ICP-MS Analysis.

8.0 Corrective Actions

8.1 Data Qualification

DOC's, MDL's, and LOQ's should be redone until they meet acceptance criteria.

Samples analyzed with a prep blank not meeting acceptance criteria may be reanalyzed or reported with qualification (B).

If the LCS or LCSD's do not meet percent recovery and/or percent relative difference (RPD) acceptance criteria they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (J).

Samples analyzed in duplicate not meeting percent relative difference (RPD) acceptance criteria may be reported with qualification (J) or reanalyzed. If the sample result or sample duplicate result is less than 5 times the reporting limit, the percent relative difference need not be evaluated.

If the spiked sample recovery is not within acceptance criteria samples may be reported with qualification (J) or reanalyzed. If the sample result is greater than 5 times the spike amount the percent recovery need not be calculated.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified:

1. Quality and expiration date of reagents
2. Cleanliness of containers, equipment, and labware
3. Integrity of standards
4. ICP-MS operation

9.0 Health and Safety

Gloves and safety glasses must be worn when handling samples and chemicals. It is necessary to wear a lab coat to protect clothing.

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Strong acids and bases should be added to containers and solutions under the hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

Analysts should familiarize themselves with any chemical substances which are named along with their corresponding MSDS sheets.

Use precautions to prevent spills. Clean up all spills immediately and notify supervisor.

10.0 Responsibilities

It is the analyst's responsibility to read and follow this standard operating procedure. Successfully perform a demonstration of capability. Record all data as per section 7.0. Review all QC results for compliance with acceptance criteria.

It is the lab supervisor's responsibility to make sure standard operating procedures are up to date and that acceptance criteria have been set.

11.0 Waste Management and Pollution Prevention

Unused non-hazardous sample is neutralized to pH ~7 SU, and then may be disposed of as wastewater.

Waste disposal of prepared metals samples and standards are disposed of in designated dilute acids and metals waste containers.

Minimize reagent use. Prepare reagents in amounts that will be used before expiration. Dispose of samples and wastes in accordance with ESI policy, state, and federal regulation.

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TITLE: Preparation of Glassware for Trace Metals Analysis

Approved By:

QA Officer:_____ Date:_____

President:_____ Date:_____

Revision History:

Revision Number	Changes	Authorization	Date
1	Review and Update	Petra Karbe	10/1999
2	Review and Update	S. Dionne	06/2000
3	Review and Update	M. Folsom	01/2006
4	Review and Update	J. Hobbs	12/2009

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TITLE: Preparation of Glassware for Trace Metals Analysis

1.0 Purpose and Applicability

This SOP describes the method for properly cleaning glassware for trace metals analysis.

2.0 Definitions - NA

3.0 Applicable Documents/References

APHA. 1992. Standard Methods for the Examination of Water and Wastewater. 18th Edition. Method 3010 C.

4.0 Materials and Apparatus

Dishwasher
Trace Metals Grade Nitric Acid, Concentrated, 50%, and 2% - 5%
Trace Metals Grade Hydrochloric Acid, Concentrated and 50%
Aqua Regia
Fume Hood

5.0 Methods/Procedures

5.1 Reagents - All acids and diluents will be 'Trace Metals Grade ' or better.

5.1.1. Aqua Regia - Mix 1 part concentrated hydrochloric acid with 1 part concentrated nitric acid and 2 parts Milli-Q® water.

5.1.2. 50% Nitric acid - Mix 1 part nitric acid to 1 part Milli-Q® water.

5.1.3. 5% Nitric acid - Mix 100 mL concentrated nitric acid in 2 L Milli-Q® water.

5.1.4. 50% Hydrochloric acid - Mix 1 part concentrated hydrochloric acid to 1 part Milli-Q® water.

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5.2 All glassware that comes in contact with test samples must be cleaned before initial use and after each use.

The following is considered as an appropriate procedure for cleaning glassware used in trace metal analysis. **NOTE:** Analysis of samples at the microgram per liter (ppb) level is subject to contamination from a number of sources, attention must be used when preparing glassware for use in these evaluations.

5.3 General glassware and plastic materials:

5.3.1. Wash in hot tap water.

5.3.2. Rinse three times with deionized water.

5.3.3. Soak the materials in a solution of 5% nitric acid. **Note:** This acid bath should not be used for any equipment that is not pre-rinsed in deionized water and no metal equipment should ever be placed into it.

5.3.3.1. If contamination is still visibly present, materials may be soaked in an acid solution of 50% trace metals grade nitric acid and 50% trace metals grade hydrochloric acid or aqua regia. This process must be performed in a fume hood and will melt plastic materials.

5.3.3.2. Rinse thoroughly seven times with deionized water.

5.3.4. Air dry materials in an area free of airborne contaminants.

5.3.5. Glass and plastic materials may be stored in deionized water.

6.0 Quality Control Requirements

Glassware should be checked for contamination periodically by filling with a 5% nitric acid solution, allowing a 2-hour soak and conducting an analysis of the acid to determine if trace metal levels are elevated as compared to the original acid solution.

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TITLE: Preparation of Glassware for Trace Metals Analysis

7.0 Calculations/Reporting - NA

8.0 Corrective Actions

If background checks indicate that the glassware is contaminated, re-wash all glassware where contamination is suspected. Replace all acid solutions. Clean the lab.

9.0 Health and Safety

Gloves must be worn at all times when working with acids. It is advised that safety glasses and lab coats be worn also when using acids. A properly working fume hood must be used when working with aqua regia. Read all relevant MSDS before working with acids.

10.0 Waste Management

All acid solutions and acid baths are disposed of in the dilute metals and acids waste containers. These containers must be properly labled. It is the responsibility of the analyst to ensure that all waste is disposed of according to ESI protocol, as well as state and federal law.

11.0 Responsibilities

It is the lab managers' responsibility to insure technicians performing this procedure are properly trained and the training is documented in the technician's training file. The technician is responsible for following the procedures outlined in this SOP.

12.0 Pollution Prevention

Minimize acid use by only replacing acid baths as often as is necessary. Dispose of acid solutions and baths according to ESI protocol, as well as state and federal law.

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TITLE: Ammonia by Lachat

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
1	Review and Update	S. Dionne	05/2000
2	Review and Update, Addition of NELAC Requirements	S. Dionne	02/2002
3	Review	K. Simon	05/2003
4	Review and Update per NELAC Assessment: Winter 2004	A. Planz	04/2004
5	Update	M. Folsom	09/2006
6	Update	M. Scott	06/2007
7	Correction of typo on page 12	M. Scott	06/2007
8	Review and correction on page 8	M. Scott	05/2009
9	Review and Update	M. Scott	06/2010
10	Update based on audit response	M. Scott	03/2012
11	Update based on audit response	M. Scott	06/2012
12	Update based on annual review	M. Scott	02/2014
13	Update to method reference	M. Scott	03/2015
14	Update in response to DOD Audit	M. Gargas	06/2016

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1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the method for determining ammonia as N in aqueous samples using method SM 4500 NH_3 G. Samples are analyzed using an automated lachat analyzer equipped with an auto-sampler. The applicable range for this method is 0.1-20 mg/L as N.

Samples are processed by the lachat analyzer. Alkaline phenol and hypochlorite react with ammonia in the sample to produce a volume of indophenol blue that is proportional to the sample's ammonia concentration. Sample data is processed into absorbance curve by CPU; results are generated and evaluated against calibration and quality control samples.

2.0 Definitions

See ESI's Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

APHA. 2012. *Standard Methods for the Examination of Water and Wastewater*. 22nd Edition. Method 4500 NH_3 G.

ESI SOP QA-1328 "Preparation of Ammonia Samples by Distillation"

Hach. 2008. QuikChem® Automated Ion Analyzer. *Methods Manual*.

Hach. 2008. QuikChem® Automated Ion Analyzer. *Hardware Installation and System Operation Manual*.

Department of Defense (DoD), Department of Energy (DoE). 2013. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.0.

4.0 Materials and Apparatus

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference, number (obtained from applicable log book), and expiration date.

4.1 Reagents and Standards

4.1.1 Milli-Q® water

4.1.2 Sodium Phenolate

In a 1 L volumetric flask, add approximately 800 mL of Milli-Q® water and then 83 g crystalline phenol (C_6H_5OH). While stirring, slowly add 32 g sodium hydroxide (NaOH). Cool, dilute to final volume with Milli-Q® water, and mix until all solids are dissolved. Store refrigerated at 4±2°C for up to one month.

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4.1.3 Sodium Hypochlorite

In a 1 L volumetric flask, mix 500 mL bleach [5.25% sodium hypochlorite (NaOCl)] with Milli-Q® water to final volume and invert to mix. Store refrigerated at 4±2°C for up to one month.

4.1.4 Sodium EDTA Buffer

In a 1 L volumetric flask add approximately 200 mL of Milli-Q® water. Add 50 g disodium ethylenediamine tetraacetate (Na_2EDTA) and 5.5 g sodium hydroxide (NaOH). Dilute to final volume with Milli-Q® water and mix until all solids are dissolved. Store refrigerated at 4±2°C for up to one month.

4.1.5 Sodium Nitroprusside

In a 1 L volumetric flask, dissolve 3.50 g sodium nitroprusside ($Na_2Fe(CN)_5NO•2H_2O$), also known as sodium nitroferricyanide, in 700 mL of Milli-Q® water. Dilute to final volume with Milli-Q® water and mix until all solids are dissolved. Store refrigerated at 4±2°C for up to one month.

4.1.6 200 mg/L as N Stock Standard

In a 500 mL volumetric flask dissolve 0.3815 g ammonium chloride (NH_4CL) in Milli-Q® water. Add 0.5 mL concentrated sulfuric acid and dilute to final volume with Milli-Q® water. Mix until all solids are dissolved. Store refrigerated at 4±2°C for up to six months.

4.1.7 Secondary Standard

Secondary standards are prepared from a source other than the ammonium chloride used to prepare the stock standard. Check standards may be prepared from commercially obtained quality control solutions, purchased ammonia standards, or another traceable source.

4.2 Equipment and Supplies

630 nm interference filter
Adjustable or fixed volume volumetric pipettes
Ammonia manifold
Analytical balance, capable of weighing to 0.0001 g (1 mg)
Computer and printer
Graduated cylinders
Heating unit
Lachat QuikChem 8500® system unit
Magnetic stir plate
Micro ammonia loop
Pump tubing
- Green - Green => sample
- Orange - Orange => sodiom phenolate

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Orange - Orange => sodiom nitroprusside
Grey - Grey => carrier line
Black - Black => sodium hypochlorite
Red - Red => sodium EDTA buffer
Reagent pump
Software, Omnion Data System Version 3.0
Stir bar
Tubes, borosilicate glass -12x75 mm, 6 mL capacity
Tubes, plastic - 15x100 mm, 15 mL capacity
Tube rack
Volumetric flasks
Weigh boats
0.45 micron syringe filters

5.0 Methods and Procedures

Samples should be collected in plastic disposable 125-500 mL bottles, based on whether or not distillation is required. Samples received for ammonia must be preserved with H_2SO_4 to a pH of <2 SU and stored at 4±2°C for up to 28 days. Samples marked at Login as requiring distilation should be distilled according to ESI SOP QA-1328 "Preparation of Ammonia Samples by Distillation", otherwise no further sample preparation is necessary. Samples with high turbidity should be filtered using a 0.45 micron syringe filter to avoid clogging the capillary tubing on the manifold.

5.1 Lachat Instrumentation Preparation

The manufacturer's hardware installation and system operation manual should be consulted for equipment specifics.

5.1.1 Timing Settings

The following timing protocols are specific to automated ammonia analysis and normally should not need to be changed.

QuikChem® 8500 Protocol	Timing
Sample Throughput	80 samples/hr; 45 s/sample
Pump Speed	35 rpm
Cycle Period	50 seconds
Inject to Start of Peak Period	28 seconds

5.1.2 Ammonia Manifold Preparation

The ammonia manifold is inserted into the Lachat analyzer. The thin tubing on the manifold will already be in place. In the event that this tubing is missing, or needs to be changed, consult the manufacturer's instructions. The colored tubing is connected to the manifold and sampling system as follows.

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- The sample line is pumped from the autosampler needle to port 6 on the injection valve using a Green - Green line.
- The micro ammonia loop connects ports 1 and 4 on the injection valve.
- The carrier line is pumped from the carrier container to port 2 on the injection valve using a Grey - Grey line.
- The manifold line connects from port 3 on the injection valve to the tee fitting labeled "➞from valve" on the left side of the manifold.
- The waste line is connected to port 5 on the injection valve and to the labeled waste container under the counter using a Green - Green line.
- The sodium EDTA buffer line is connected from the buffer container to the tee fitting labeled "Buffer➞" on the right side of the manifold using a Red - Red line.
- The sodium phenolate line is connected from the phenolate container to the tee fitting labeled "➞Phenolate" on the left side of the manifold using an Orange - Orange line.
- The sodium hypochlorite line is connected from the hypochlorite container to the tee fitting labeled "➞Hypochlorite" on the left side of the manifold using a Black - Black line.
- The sodium nitroprusside line is connected from the nitroprusside container to the tee fitting labeled "➞Nitroprusside" on the left side of the manifold using an Orange - Orange line.
- The heater In line comes from the left of the heater and up through the hole in the manifold board connecting to the top of the tee fitting labeled "➞Nitroprusside".
- The heater out line comes from the right of the heater (after use it becomes slightly brown in color) and up through the hole in the manifold board connecting to the left of the fitting labeled "to flow cell ➞630nm".
- The fitting labeled "to flow cell ➞630nm" connects to the flow cell line on the right of the manifold.

5.1.3 Turn the power to the analyzer unit on. the auto-sampler needle should rise to its highest position and the temperature gauge on the left side of the unit will turn on. Turn on the pump and check that it is working properly.

5.1.4 Place all of the lines into the Milli-Q® water rinse vessel to allow water to flush through the entire system for 2 to 5 minutes. The temperature will automatically set via the Omnion 3.0 software.

Caution: The reagent line will melt if empty or allowed to sit containing standing water. Make sure that the pump is on, and that rinse water is flowing throughout preparation.

5.1.5 The 630 nm interference filter is placed right-side-up into the flow cell. It must be kept in a desiccator when not in use.

5.2 Lachat Instrumentation System Startup

5.2.1 Turn the computer on and log into the Omnion 3.0 program. The QuikChem® unit must also be powered up. Click on "Open" and select the ammonia template.

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5.2.2 Enter all of the necessary sample data into the tray spreadsheet. If the sample data has already been entered and saved, then click on "Open" and select the tray to be analyzed.

5.2.3 Go to "File" and "Save Tray As." The tray data is saved as the current date, followed by an A,B,C, etc. to differentiate multiple analyses from the same day.

5.3 Standard and Quality Control Sample Preparation

The preparation of all standards may be modified to meet project specific requirements.

5.3.1 Calibration Standards

To five 100 mL volumetric flasks, add the following amounts of stock standard to Milli-Q® water, bring to final volume, and invert gently to mix. Store refrigerated at 4±2°C for up to one month.

Stock Standard Volume (mL)	10	7.5	5.0	2.5	0.05
Final Concentration as mg/L N	20	15	10	5.0	0.1

5.3.2 The Initial Calibration Verification (ICV) standard should be made from a recent Proficiency Test stock. The suppliers instructions should be followed. Store refrigerated at 4±2°C for up to three months.

5.3.3 The Continuing Calibration Verification (CCV) standard is the 10 mg/L as N Calibration Standard.

5.3.4 For the Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and preparatory blank samples use Milli-Q® water.

5.3.5 For the Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD), spike 5 mL of Milli-Q® water with 263 uL of the 200 mg/L as N Stock Standard and mixed thoroughly. True Value is 10 mg/L as N.

5.3.6 For the Matrix Spike (MS) and Matrix Spike Duplicate (MSD) spike 5 mL of the sample or diluted sample with 263 uL of the 200 mg/L as N Stock Standard and mixed thoroughly. True value of the spike is 10 mg/L as N.

5.4 Calibration Procedure

5.4.1 The auto-sampler recognizes standards to be located in cups 1-S. Fill each cup completely with the appropriate calibration standard and place in the appropriate cup as listed in the tray spreadsheet (20,15, 10, 5, 0.1, 0 mg/L N, respectively).

5.4.2 The calibration equation is quadratic with a 1/X weighing.

5.4.3 An ICV/ICB must be analyzed following calibration. A CCV/CCB must be run every ten samples and at the end of an analytical session.

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5.5 Sample Preparation

5.5.1 Samples are removed from refrigeration immediately prior to sample preparation. Each sample is poured, without prior agitation to the sample container, into a 12x75 mm glass tube. Tubes are placed into the autosampler cups in the order listed in the tray spreadsheet, or placed, in the proper order, into a tube rack, to await analysis. It is imperative that samples are kept in the proper order throughout analysis.

5.5.2 Sample Distillation

Most ammonia samples will not require distillation. However, some samples may require sample distillation prior to analysis. The Laboratory Manager may be consulted to determine which samples require distillation. See SOP QA-1328 "Preparation of Ammonia Samples by Distillation".

5.6 Sample Analysis

5.6.1 Place all lines in the appropriate reagent vessels. Observe the manifold to ensure that the instrument is drawing reagent. Allow reagents to flow for about ten minutes to let the baseline stabilize.

5.6.2 To start a run, click on "Run". The auto-sampler will move to the appropriate location and analyze the samples.

5.6.3 During sample analysis, any sample that is noted to have an ammonia value greater than the highest point on the calibration curve, 20 mg/L as N, it must be diluted and re-analyzed. The diluted sample is added to the end of the tray. The tray must be saved before the auto-sampler reaches the diluted sample or the sample will not be analyzed.

5.7 Shut Down Procedure

5.7.1 When all samples in the tray are analyzed, remove the lines from the reagents and place in rinse water. If reagents are to be reused, store at 4±2°C until the respective expiration dates.

5.7.2 Remove the 630 nm interference filter from the flow cell and place it in the desiccator.

5.7.3 When the session is complete, it is automatically saved under the file name assigned during instrument preparation.

5.7.4 The temperature will automatically reset to 20°C once the run is complete.

When the temperature reads <35°C, the lines may be removed from the rinse water and allowed to pump dry.

5.7.5 Exit the program.

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6.0 Quality Control Requirements

Quality Control elements may be added, removed, or altered to meet specific program or project requirements. Acceptance criteria shall be updated based on the associated control charts once sufficient data points have been collected (at least 20 data points) and yearly thereafter. Alternate acceptance criteria may be set for specific programs or projects. For DoD-specific projects reference QSM 5.0, as appropriate.

6.1 Demonstration of Capability (DOC)

An analyst must complete a DOC before working independently on this procedure and yearly thereafter. A DOC is also required with any changes to instrument, method, or analyst. To perform a DOC, four replicates of a prepared quality control sample must be prepared. DOC samples must meet LCS acceptance criteria. For more information on DOCs see SOP QA-1108 “Training”.

6.2 Method Detection Limit (MDL)

The MDL is calculated using the appropriate student’s t-parameter value multiplied by the standard deviation of 7 or more replicates of spike samples or standards. MDLs are performed annually and with changes in instrument or method. The calculated MDL must be lower than the reporting limit.

The t-parameter value is from a one-tailed Student’s t-Test at the 99% confidence interval using n-1 degrees of freedom, with “n” being the number of replicates.

The most recent MDL study can be found in the Inorganics Quality Control directory. For more information on MDLs see SOP QA-1111 “Determination of Method Detection and Reporting Limits”.

6.3 Limit of Detection (LOD)

LOD determination will be conducted on a quarterly basis. For a single-analyte standard, the LOD is a single quality control sample that is approximately 2 to 3 times the MDL but no greater than the reporting limit. For a multi-analyte standard, the LOD is a single quality control sample that is approximately 1 to 4 times the MDL but no greater than the reporting limit.

The apparent signal to noise ratio at the LOD must be at least 3. For methods that do not produce a signal to noise, the LOD result must be at least 3 standard deviations greater than the mean method blank.

6.4 Limit of Quantitation (LOQ)

LOQ confirmation will be conducted on a quarterly basis. The LOQ is a single control sample no more than 1 to 2 times the reporting limit. The LOQ standard acceptance criteria is 75-125%. For more information on LODs and LOQs see SOP QA-1147 “Determination of LOD and LOQ Quality Control Samples”.

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6.7 Blanks, LCS/LCSD, MS, MSD (if required by customer specific protocol), Sample Duplicate, Relative Percent Difference (RPD) - criteria to be updated yearly from the associated control charts.

6.7.1 A blank, LCS/LCSD, MS and sample duplicate will be analyzed every batch or every 20 samples, whichever is more frequent.

6.7.2 Acceptance criteria for the blank is <0.1 mg/L as N or less than 1/10th of sample value.

6.7.3 Acceptance criteria for LCS/LCSD recovery is 82-118% with a warning limit of 88-112%. Acceptance criteria for LCSD RPD is less than or equal to 18% with a warning limit of 12%.

6.7.4 Acceptance criteria for MS/MSD recovery is 75-125% with a warning limit of 86-114%.

6.7.5 Acceptance criteria for RPD between sample duplicates is less than or equal to 18% with a warning limit of 12%.

6.8 ICV, CCV, ICB, and CCB

6.8.1 Initial calibration consists of a blank and five standards. Acceptance criteria for initial calibration is a relative standard deviation for response factors less than 10%, 25% for the low point, and a correlation coefficient of greater than 0.995.

6.8.2 The ICV and ICB are performed following analysis of initial calibration standards. Acceptance criteria for the ICV must be ±10% of the target value or within calculated limits, whichever is more stringent. Acceptance criteria for the ICB is <0.1 mg/L as N or less than 1/10th of sample value.

6.8.3 CCV and CCB are performed every 10 samples and at the end of an analytical session. Acceptance criteria for CCVs is must be ±10% of the target value or within calculated limits, whichever is more stringent. Acceptance criteria for the CCB is <0.1 mg/L as N or less than 1/10th of sample value.

6.9 The objective reporting limit for the analysis of Ammonia by Lachat is 0.1 mg/L as N.

6.10 Interferences

Air spikes occur when the sampling syringe draws in air, disrupting the sample peak. If an air spike causes erroneous computer integration, manual integration may be performed on the peak. All calculations should be documented.

7.0 Calculations and Reporting

7.1 The following information should be recorded when performing an ammonia analysis.

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- Date
- Analysts initial
- Batch
- Sample ID / Standard ID, with dilution factor if applicable
- ID of reagents
- Amount/ID of standard
- Data file name
- Pipette IDs
- Notes, if any

7.2 Calculations

Calibrate the system software as per the Lachat Instruments "Hardware Installation and System Operation Manual" (see System Operation section). Use the system software to calculate the Ammonia - N values for quality control elements and samples. Review the data to ensure it is correct and that all quality control requirements have been met.

7.2.1 Calculate LCS/ICV/CCV Recovery as follows:

$$\frac{\text{LCS/ICV/CCV Concentration}}{\text{Concentration Added}} \times 100 = \%\text{Recovery}$$

7.2.2 Calculate %RPD as follows:

$$\frac{\text{Abs(Result 1 - Result 2)}}{\text{Average of results}} \times 100 = \%\text{RPD}$$

7.2.3 Calculate MS/MSD Recovery as follows:

$$\frac{\text{Spike P Concentration - Sample P Concentration}}{\text{Concentration added to Spike}} \times 100$$

8.0 Corrective Actions

8.1 Data Qualification

8.1.1 If DOCs, MDLs, LODs, or LOQs do not meet acceptability criteria, then repeat until acceptability criteria is met.

8.1.2 If the initial calibration curve is not within acceptance criteria, then re-calibrate or qualify any positive sample values as estimated (using the "J" qualifier).

8.1.3 If the ICV does not meet acceptance criteria, then prepare a new curve and ICV or qualify any positive sample values as estimated (using the "J" qualifier).

8.1.4 If samples analyzed with blanks (prep, initial, continuing) do not meet acceptance criteria, then samples may be re-analyzed or reported with qualification (using the "B" qualifier).

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8.1.5 If LCS/LCSDs do not meet percent recovery and/or RPD acceptance criteria, then they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (using the "J" qualifier).

8.1.6 If samples analyzed in duplicate do not meet RPD acceptance criteria, then samples may be reported with qualification (using the "J" qualifier) or reanalyzed. If the sample result or sample duplicate result is less than 5 times the reporting limit, then the RPD need not be evaluated.

8.1.7 If the MS/MSD recovery is not within acceptance criteria, then samples may be reported with qualification (using the "J" qualifier) or re-analyzed. If the sample result is greater than 5 times the spike amount, then the percent recovery need not be calculated.

8.1.8 If a CCV does not meet acceptance criteria, then all associated samples must be reanalyzed or qualified as estimated (using the "J" qualifier).

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified

- Quality and expiration date of reagents
- Cleanliness of containers, equipment, and labware
- Integrity of standards
- Condition of tubing
- Lachat operation: see troubleshooting section of Lachat Instruments: Hardware Installation and System Operation Manual

9.0 Health and Safety

9.1 Phenol is known to cause burns and phenol vapors have been known to cause respiratory irritation, headaches and nausea; phenol must be used with extreme caution. Whenever possible, limit exposure by keeping containers capped when not in use, ensuring that any spilled phenol is cleaned immediately, and conducting preparations under a fume hood.

9.2 Gloves and safety glasses should be worn when handling samples and chemicals. It is advisable to wear a lab coat to protect clothing.

9.3 Strong acids and bases should be added to containers and solutions under the hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

9.4 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding Material Safety Data Sheet (MSDS).

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10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptance criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP, recording all data as described in section 7.0, and reviewing all QC results for compliance with acceptance criteria.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities and DOC performance.

11.0 Waste Management and Pollution Prevention

Unused portions of samples are disposed of only after completion of sample analysis, submission of the final report and contracted sample retention times have been achieved. All conditions of disposal and all records and correspondence concerning the final disposition of the physical sample shall be recorded and retained. Records shall indicate the date of disposal, the nature of disposal and the name of the individual who performed the task.

Unused non-hazardous sample is neutralized to pH ~7 SU, and then may be disposed of as wastewater. Waste disposal of reagents and standards are disposed of in the designated lachat waste container. Waste disposal of prepared ammonia samples are disposed of in designated diluted dilute acids and metals waste containers. Minimize reagent use and prepare reagents in amounts that will be used before expiration.

11.1 Chemically Preserved Samples

Chemically preserved samples should have a pH of <2 or >12 SU. The pH alone causes them to express a corrosively hazardous nature that must be neutralized prior to disposal. Once neutralized, samples are no longer viable for testing and are then considered waste. ESI's aqueous waste UV treatment "pit" system is used for discharge. If any samples are hazardous for reasons other than pH, then they must be disposed of according to their specific requirements based on hazard classification. The approximate volume of neutralized waste, date of disposal and initials of person performing the disposal are recorded in a logbook.

11.2 Hazardous Waste Samples

Hazardous waste samples must be handled by properly trained employees. Only the ESI hazardous waste coordinator may train ESI employees on handling hazardous waste. There are 3 main hazardous waste streams at ESI, "dilute metals acidic", "solvents" and "Lachat wastes". When a satellite container needs to be emptied, it is properly transported to the waste shed and added into the appropriately labeled container for that waste stream. Logbooks are kept as a record for volumes added to each drum of any specific waste stream

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and shipping manifests reflect when they are picked up for transport.

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TITLE: Percent Moisture in Solid Samples

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	K. A. Simon	05/1995
1	Review and Update	S. Dionne	06/2000
2	Review and Update	L. Hauthaway	11/2001
3	Review and Update	K. A. Simon	05/2004
4	Editorial Review and Update	N. Roka	06/2009
5	Review and Update	M. Gargas	02/2016

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TITLE: Percent Moisture in Solid Samples

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the procedure for obtaining the Percent (%) Moisture of solid samples by ASTM Method E1756, or as specified by the project scope. Determination of percent moisture is used for reporting data in terms of dry weight. Percent moisture is the percentage of water found in a test sample and is determined by the amount of mass lost during the drying of the sample at 105±3°C to constant mass.

2.0 Definitions

Percent Moisture: The percentage of water (by weight) contained in a given amount of solid sample.

See ESI's Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

ASTM International. 2012. *Annual Book of ASTM Standards*. Volume 11.06. Method E1756-08.

Department of Defense (DoD), Department of Energy (DoE). 2013. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.0.

SOP QA-1124 "Calibration and Use of Balances"

4.0 Materials and Apparatus

Analytical Balance
Desiccator, containing desiccant
Drying Oven (105±3°C)
Sample Scoop
Sand (stored in drying oven)
Tongs
Weighing Dish (aluminum pan, beaker, petri dish, crucible, or watch-glass)

5.0 Methods and Procedures

Samples should be stored in glass jars. If storage of solid samples is required then refrigerate at 4±2°C for up to 14 days.

5.1 Preparation of Weighing Dish

5.1.1 The weighing dish used should be of appropriate material and size for the sample. Commonly used weighing dishes include: disposable aluminum pans, beakers, petri dishes, crucibles, or watch-glasses. Dry the weighing dish in a drying oven maintained at 105±3°C for at least 2 hours. Using tongs, remove the weighing dish from the oven, place in a desiccator and let cool to ambient temperature (this will

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take at least 1 hour).

5.1.2 Using tongs to handle the dish, determine the weight of the weighing dish by using an analytical balance. Record the weight to the nearest 0.001 g as the dish tare weight. See SOP QA-1124 "Calibration and Use of Balances" for more information.

5.2 Obtain a representative subsample and add 5±1 g of sample to the weighing dish. Record to the nearest 0.001 g as the sample wet weight.

5.3 Place the sample dish into an oven maintained at 105±3 °C until sample is dry. This will take at least 3 hours but no longer than 72 hours. If multiple sample dishes are placed in the oven at once, then it is important to keep all dishes on one level to ensure equal heat distribution; do not stack dishes.

5.4 Remove the sample dish from the oven with gloves or tongs, and place in a desiccator and let cool to ambient temperature.

5.5 Record the sample dish to the nearest 0.001 g as the sample dry weight.

5.6 Repeat steps 5.3 to 5.5 until a constant weight is obtained, which is defined as successive weighings that differ less than 0.005 g or 5% of the net dry solid weight.

5.7 Calculate percent (%) moisture as described in Section 7.2.

5.8 Calibration and standardization are not applicable components of this method and procedure.

6.0 Quality Control Requirements

6.1 A duplicate will be analyzed with every batch or every 20 samples, whichever is more frequent. The Relative Percent Difference (RPD) for duplicates is expected to be within 10%.

6.2 A blank will be analyzed with every batch or every 20 samples, whichever is more frequent. A blank may be clean sand that is stored in the drying oven. Blanks should be <0.1% moisture.

6.3 Desiccant used in desiccators should be refreshed on a quarterly basis to ensure the drying agent does not become saturated.

6.4 Interferences and a Method Detection Limit are not applicable quality control requirements for this method and procedure.

7.0 Calculations and Reporting

Report percent moisture to 3 significant figures.

7.1 The following information should be recorded when performing an analysis of percent (%) moisture:

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- Date
- Analyst's initial
- Batch
- Sample ID / code
- Balance ID
- Tare weight, sample wet weight, sample dry weight
- Percent moisture calculation
- Notes, if any

7.2 Calculate percent (%) moisture as follows:

$$\text{Percent (\%) Moisture} = \frac{S_D - D}{S_W - D} * 100$$

Where:

S_W = Dish and Wet Sample
S_D = Dish and Dry Sample
D = Dish Tare Weight

8.0 Corrective Actions

8.1 Data Qualification

8.1.1 If the duplicates RPD is >10%, then all samples in the batch must be re-dried. If calculations show duplicates are still out of range, then the batch should be re-run or qualified.

8.1.2 If blanks contain >0.1% moisture, then all samples in the batch must be re-run. If blanks are still out of range, then the batch should be re-run or qualified.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified.

- Cleanliness of containers, equipment, and labware
- Instrument operation

9.0 Health and Safety

9.1 Gloves and safety glasses should be worn when handling samples. Personal protective equipment should be worn when removing or adding dishes to the drying oven.

9.2 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding material safety data sheets (MSDS).

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10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptance criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP, recording all data as described in Section 7.1, and reviewing all QC results for compliance with acceptance criteria.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities.

11.0 Waste Management and Pollution Prevention

11.1 Unused portions of samples are disposed of only after completion of sample analysis, submission of the final report and contracted sample retention times have been achieved. All conditions of disposal and all records and correspondence concerning the final disposition of the physical sample shall be recorded and retained. Records shall indicate the date of disposal, the nature of disposal and the name of the individual who performed the task. Disposal of solid samples should be determined on a case by case basis through consultation with the Project Manager.

11.2 Solid Materials

Waste storage, transfer and disposal will be in compliance with NH DES and US EPA waste disposal regulations. Non-hazardous solid materials, sediments and soils will be disposed of through an appropriate waste disposal site. All non-hazardous solid waste will be drained of as much free liquid as possible. The remaining sample is removed from its original container and placed with other non-hazardous waste samples in a 55 gallon metal drum. Once full, these containers will then be transferred to an approved waste disposal facility. This transfer is recorded on a waste manifest and they are retained for a minimum of 3 years.

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TITLE: Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry

Approved By:

QA Officer:_____Date:_____

Technical Director:_____Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	M. Folsom	10/2006
1	Review and Update	M. Scott	12/2009
2	Update based on Audit Response	J. Hobbs	04/2010
3	Review and Update	M. Scott	09/2010
4	Update based on audit response	M. Scott	03/2012
5	Update based on audit response	M. Scott	06/2012

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TITLE: Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry

1.0 Purpose and Applicability

This method is for the determination of mercury (Hg) in water by cold vapor atomic fluorescence spectrometry. It is applicable to drinking water, surface and ground waters, marine water, industrial and municipal wastewater, aqueous samples, ranging from marine water to effluent.

The samples are processed by treatment with a bromate/bromide solution followed by a hydroxylamine solution just prior to analysis. The mercury levels are determined by running the samples through a PSA mercury fluorescence system and then evaluated against calibration and quality control samples. This method is used with a range of 10ng/L to 150ng/L.

2.0 Definitions

NA

3.0 Applicable Documents/References

Method 245.7 Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry. Revision 2.0 February 2005. EPA-821-R-05-001.

4.0 Materials and Apparatus

4.1 Reagents/Standards

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date.

Milli Q
Air - low in particulate and gaseous mercury
Argon Gas - high purity (99.998%)

Hydrochloric Acid Solution - add concentrated HCl to Milli-Q in a 1:1 ratio. Prepare as needed.

Sulfuric Acid Solution - add 100mL of concentrated sulfuric acid to Milli-Q for a final volume of 4L. Prepare as needed.

Hydroxylamine Solution - Dissolve 2.4g of Hydroxylamine Hydrochloride in 20.0 mL of Milli Q. Prepare weekly.

Stannous Chloride Solution - Dissolve 20.0g of stannous chloride into 2L of the Sulfuric acid solution or dissolve into 2 L of Milli-Q with 20.0 mL of concentrated HCl. Prepare before each analytical session.

Bromate/Bromide Solution - Dissolve .167g of Potassium Bromate and 0.714g of Potassium Bromide in 30.0 mL of Milli-Q. Prepare as needed.

Mercury Standard, 1.0 mg/L - Add about 50mL of Milli-Q, 0.5mL of the Bromate/Bromide solution, and

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0.25mL of the HCl solution to a 100mL volumetric flask. Add .100mL of the stock mercury standard and dilute to 100mL, transfer to a glass bottle. Stable until expiration date given for stock mercury standard.

Check Mercury Standard, 1.0 mg/L - Add about 50 mL of Milli-Q, 0.5mL of the Bromate/Bromide solution, and 0.25mL of the HCl solution to a 100 mL volumetric flask. Add 0.100 mL of the secondary mercury standard and dilute to 100 mL, transfer to a glass bottle. Stable until expiration date given for stock mercury standard.

Working Mercury Standard, 2000 ng/L - Dilute 0.2mL of the Mercury standard to 100 mL in a volumetric flask with Milli Q. This solution should be made new with each use.

ICV Mercury Standard - Dilute .2mL of the Check Mercury standard to 100mL in a volumetric flask with Milli Q. This solution should be made new with each use.

4.2 Equipment and Supplies:

CVAFS System: PSA Merlin
Software: Avalon P.S. Analytical Version 2.28
40mL glass vials
Adjustable pipettes
Volumetric Flasks
Graduated cylinders
Analytical balance, capable of weighing to 0.0001 g (1 mg)
Stir Bar, magnetic stir plate, and weigh boats
Computer & printer
Vial rack

5.0 Methods/Procedures

Samples should be collected in a 40-125mL glass or fluoropolymer vials with fluoroploymer lined caps. Samples received for mercury are preserved with HCl to a pH $\leq$2 SU and have a holding time of 28 days.

5.1 Sample Preparation

Samples are prepared by first treating empty 40mL vials with 2.25mL of HCl Solution and .45mL of the Bromate/Bromide solution (a dark yellow color should result). Then 20mL of each sample is transfered into a treated 40mL vial. The sample should be shaken and allow to digest for at least 30 minutes. If the yellow color does not remain more Bromate/Bromide should be added until a permanent yellow color is obtained.

After the samples are allowed to digest the caps are removed and 25uL of the Hydroxylamine solution is added to each vial, recapped swirled occasionally over a 5 minute period. The addition of the hydroxylamine should cause the yellow color to disappear. The hydroxylamine should be added at a time the coincides with the start of sample analysis.

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5.2 Calibration Preparation

The curve, ICV, ICB, CCV, and CCB are prepared at the same time and in the same manner as the samples. The curve points are 10, 25,50,100,150ng/L. The ICV and CCV are prepared at 25ng/L. The CCB and ICB consist of 20mL of Milli-Q added to the pretreated vials. The following amounts of the Working Mercury Standard are brought to a final volume of 20mL in pill cups before being added to the pretreated 40mL vials.

Concentration (ng/L)	Volume added (mL)	Std. Source
10	.100	Working Hg Std.
25	.250	Working Hg Std.
50	.500	Working Hg Std.
100	1.00	Working Hg Std.
150	1.50	Working Hg Std.
CCV25	.250	Working Hg Std.
ICV25	.250	ICV Hg Std.

The curve, ICV, ICB, CCV, and CCB are also allowed to digest for at least 30 minutes and then have 25uL of hydroxylamine solution added to them just prior to analysis.

5.3 Instrument Operation

Turn on the CVAFS system, air, and argon. Set the Argon to 400 cc/mn, the air to 2.5 L/mn, and the flow valve to .8 L/mn. Allow the instrument to warm up for 20 minutes with Milli-Q running through the reagent and sample lines. When ready to begin analysis place the reagent line in the Stannous Chloride solution and the sample line into the Sulfuric Acid solution and allow to flow through.

Analyze an instrument blank to verify no contamination is present, then run the curve, ICV, ICB. If the curve and initial calibration standards are acceptable proceed with batch analysis with CCV's and CCB's being run every 10 samples. The sample line is to be in the sample when the program is during its "rise" and "analysis" cycles and in the sulfuric acid solution at all other times.

When ready to shut down allow water to flush through the lines for at least 10 minutes. Then shut off the argon, air, and CVAFS system.

6.0 Quality Control Requirements

The objective reporting limit is 10ng/L.

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TITLE: Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry

6.1 QC Elements

Demonstration of capability (DOC), four replicates of a prepared quality control sample. Required with a change of instrument, method, or analyst.

Method detection limit (MDL) study, seven replicates (standard deviation * 3.14). Performed annually, with change in instrument, or method.

Limit of quantitation (LOQ), a single quality control sample, no more than 1-2x the reporting limit. Performed annually.

Analyze a blank, laboratory control, and if desired a laboratory control sample duplicate every analytical batch or twenty samples, whichever is more frequent. A sample spike and sample spike duplicate must be performed every ten samples.

Initial calibration consisting of a blank and five standards.

Initial calibration verification (ICV) and initial calibration blank (ICB) are performed following analysis of initial calibration standards.

Continuing calibration verification (CCV) and continuing calibration blank (CCB) are performed every ten samples and at the end of an analytical session.

QC elements may be added or removed to meet specific program or project requirements.

6.2 Acceptance Criteria

Demonstration of capability samples must meet LCS acceptance criteria.

The calculated MDL must be lower than the reporting limit.

The limit of quantitation standard acceptance criteria is 50-150%.

Acceptance criteria for initial calibration is a relative standard deviation for response factors less than 15% or a correlation coefficient of greater than 0.995. Also the % recovery of the low point in the curve must lie in the range of 75 - 125%.

Acceptance criteria for all blanks is < 10 ng/L or less than 1/10 of sample value.

Acceptance criteria for the initial calibration verification standard (ICV) must be ±15% of the target value.

Acceptance criteria for the continuing calibration verification standard (CCV) is must be within 76%-113% of the target value.

Recovery of the LCS/LCSD pair must be within 76%-113% of the target value. Additionally, the relative percent difference (RPD) between the LCS and LCSD must not exceed 25%.

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Acceptance criteria for relative percent difference between sample spike and sample spike duplicates is less than or equal to 18%.

Recovery of the spiking compound must be within 63%-111% of the target value.

Alternate acceptance criteria may be set for specific programs or projects.

6.3 Interferences

Gold, silver, and iodine are known interferences. The bromate/bromide digestion reagent overcomes many chloride, sulfide, and molecular absorption interferences.

7.0 Calculations/Reporting

7.1 Record

Record the following:

1. Date
2. Analysts initial
3. Batch
4. Sample ID / Standard ID, with dilution factor if applicable
5. Sample Code / Standard Code
6. Signal
7. Amount/ID of reagent
8. Amount/ID of standard
9. Sample volume
10. Notes, if any

7.2 Calculation

Use the signal datum provided by the CVAFS system to create an initial calibration by applying either the average response factor or quadratic fit. Use the signal datum to determine the mercury concentration for each sample and QC sample utilizing the initial calibration.

Calculate LCS/ICV/CCV Recovery as follows:

$$\frac{\text{LCS/ICV/CCV concentration}}{\text{Concentration added}} \times 100 = \%\ \text{recovery}$$

Calculate %RPD as follows:

$$\frac{\text{Abs(Result 1 - Result 2)}}{\text{Avg. of results}} \times 100 = \%\text{RPD}$$

Calculate Spike Recovery as follows:

$$\frac{\text{Spiked sample concentration - Sample concentration}}{\text{Concentration added}} \times 100 = \%\ \text{recovery}$$

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TITLE: Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry

8.0 Corrective Actions

8.1 Data Qualification

DOC's, MDL's, and LOQ's should be redone until they meet acceptance criteria.

If the initial calibration curve is not within acceptance criteria, recalibrate or qualify (J) any positive sample values as estimated.

If the ICV does not meet acceptance criteria prepare a new curve and ICV or qualify (J) any positive sample values as estimated.

Samples analyzed with blanks (prep, initial, continuing) not meeting acceptance criteria may be reanalyzed or reported with qualification (B).

If the LCS or LCSD's do not meet percent recovery and/or percent relative difference (RPD) acceptance criteria they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (J).

If the spiked sample and spiked sample duplicate do not meet the percent relative difference (RPD) acceptance criteria the result may be reported with qualification (J) or reanalyzed.

If the spiked sample or spiked sample duplicate recovery is not within acceptance criteria samples may be reported with qualification (J) or reanalyzed. If the sample result is greater than 5 times the spike amount the percent recovery need not be calculated.

If a CCV does not meet acceptance criteria all associated samples must be reanalyzed or qualified (J) as estimated.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified:

1. Quality and expiration date of reagents
2. Cleanliness of containers, equipment, and labware
3. Integrity of standards
4. Condition of tubing
5. Air and argon flow

9.0 Health and Safety

Mercury is toxic.

Gloves and safety glasses must be worn when handling samples and chemicals. It is necessary to wear a lab coat to protect clothing.

Strong acids and bases should be added to containers and solutions under the hood. When all traces

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of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

Analysts should familiarize themselves with any chemical substances which are named along with their corresponding MSDS sheets.

Use precautions to prevent spills. Clean up all spills immediately and notify supervisor.

10.0 Responsibilities

It is the analyst's responsibility to read and follow this standard operating procedure, Successfully perform a demonstration of capability. Record all data as per section 7.0. Review all QC results for compliance with acceptance criteria.

It is the lab supervisor's responsibility to make sure standard operating procedures are up to date and that acceptance criteria have been set.

11.0 Waste Management and Pollution Prevention

Unused non-hazardous sample is neutralized to pH ~7 SU, and then may be disposed of as wastewater.

Waste disposal of prepared mercury samples, reagents, and standards are disposed of in designated dilute acids and metals waste containers.

Minimize reagent use. Prepare reagents in amounts that will be used before expiration. Dispose of samples and wastes in accordance with ESI policy, state, and federal regulation.

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TITLE: Extraction of Pentachlorophenol and other ABN Compounds in Aqueous Samples

Approved By:

QA Officer:_____ Date:_____

Technical Director:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	L. DeFrancesco	05/2005
1	Review and Update	M. Scott	11/2011
2	Update	M. Scott	02/2012
3	Review and Update	M. Scott	05/2013

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TITLE: Extraction of Pentachlorophenol and other ABN Compounds in Aqueous Samples

1.0 Purpose and Applicability

This standard operating procedure describes isolating organic compounds from aqueous samples. The SOP also describes concentration techniques suitable for preparing the extract for the appropriate determinative methods. This procedure is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organic compounds in preparation for a variety of chromatographic procedures.

A measured volume of sample, usually 1 liter, at a specified pH (see Table 1), is serially extracted with methylene chloride using a separatory funnel. The extract is dried, concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative method to be used (see Table 1 for appropriate exchange solvents).

2.0 Definitions

NA

3.0 Applicable Documents/References

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, US EPA SW 846, 3rd edition: Method 8270C

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, US EPA SW 846, 3rd edition: Method 3510C (Consult for interferences and pollution prevention)

Methods for Chemical Analysis of Water and Wastes, US EPA 600 - 4/79/020, Method 625

Agilent Technologies. Agilent 7683 Automatic Liquid Sampler. *Operation Guide.* 1997-2000.

Agilent Technologies. Agilent 6890 Series Gas Chromatograph. *Operating Manual.* 2000.

Agilent Technologies. Agilent 5973*Network* Mass Selective Detector. Hardware Manual. 1999.

Agilent Technologies. Environmental ChemStation. *G1701DA Version D.00.00.38 19-Nov 2001.* 1989-2001.

4.0 Materials and Apparatus

4.1 Reagents and Standards

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date.

Milli-Q water

Sodium hydroxide solution (10 N), NaOH: Dissolve 40g NaOH in Milli-Q water and dilute to 100 mL. Other concentrations of hydroxide solutions may be used to adjust sample pH, provided that the volume

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added does not appreciably change (e.g., <1%) the total sample volume.

Sodium sulfate (granular, anhydrous), Na_2SO_4: Purify by heating to 400°C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

Sulfuric acid solution (1:1 v/v), H_2SO_4: Slowly add 50 mL of H_2SO_4 to 50 mL of Milli-Q water. Other concentrations of acid solutions may be used to adjust sample pH, provided that the volume added does not appreciably change (e.g., <1%) the total sample volume.

Methylene chloride, CH_2Cl_2: boiling point 39°C, Pesticide grade.

Hexane, C_6H_{14}: boiling point 68.7°C, Pesticide grade.

2-Propanol, $CH_3CH(OH)CH_3$: boiling point 82.3°C, Pesticide grade.

Cyclohexane, C_6H_{12}: boiling point 80.7°C, Pesticide grade.

Acetonitrile, CH_3CN: boiling point 81.6°C, Pesticide grade.

Stock semi-volatile organic standards: In-house stock standard solutions may be prepared from neat stocks or purchased.

4.2 Equipment and Supplies

1 L separatory funnels, with polytetrafluoroethylene (PTFE) stopcock or screw-on cap.
Drying column
Kuderna-Danish (K-D) apparatus.
Concentrator tube - 10-mL, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.
Evaporation flask - 500-mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

Snyder column - Three-ball macro (Kontes K-503000-0121 or equivalent).
Snyder column - Two-ball micro (Kontes K-569001-0219 or equivalent).
Springs - 1/2 inch (Kontes K-662750 or equivalent).

NOTE: The following glassware is recommended for the purpose of solvent recovery during the concentration procedures requiring the use of Kuderna-Danish evaporative concentrators. Incorporation of this apparatus may be required by State or local municipality regulations that govern air emissions of volatile organics. EPA recommends the incorporation of this type of reclamation system as a method to implement an emissions reduction program. Solvent recovery is a means to conform with waste minimization and pollution prevention initiatives.

Solvent vapor recovery system (Kontes K-545000-1006 or K-547300-0000, Ace Glass 6614-30, or equivalent).
Boiling chips - Solvent-extracted, approximately 10/40 mesh (silicon carbide or equivalent).
Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

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Vials - 2-mL, glass with PTFE-lined screw-caps or crimp tops.
pH indicator paper - pH range including the desired extraction pH.
Erlenmeyer flask - 250-mL.
Syringe - 1-mL.
Graduated cylinder - 1-liter glass

5.0 Methods/Procedures

Samples should be collected in 1L glass jars with Teflon lined caps.
Samples received for semi-volatiles must have a pH in the range of 5 to 9 pH units.
If storage is required, samples are maintained at 4°C and may be held up to 7 days.

5.1 Extraction

Ascertain that all glassware is clean and dry. Thoroughly solvent rinse all glassware used in this procedure before proceeding with extractions.

Using a 1L graduated cylinder, measure 1L of sample. Alternatively, if the entire contents of the sample bottle are to be extracted, mark the level of sample on the outside of the bottle.If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1-L with Milli-Q water, or samples may be collected in smaller sample bottles and the whole sample used.

Pipette 1.0 mL of the surrogate spiking solution into each sample and mix well. (See Method 3500 and the determinative method to be used for details on the surrogate standard solution and matrix spiking solution).

For the sample in each batch (see Chapter One) selected for use as a matrix spike sample, add 1.0 mL of the matrix spiking standard.

If Method 3640, Gel-Permeation Cleanup, is to be employed, add twice the volume of the surrogate spiking solution and the matrix spiking standard, since half of the extract is not recovered from the GPC apparatus. (Alternatively, use 1.0 mL of the spiking solutions and concentrate the final extract to half the normal volume, e.g., 0.5 mL instead of 1.0 mL).

Check the pH of the sample with wide-range pH paper and adjust the pH, if necessary, to the pH indicated in Table 1, using 1:1 (v/v) sulfuric acid or 10 N sodium hydroxide. Lesser strengths of acid or base solution may be employed, provided that they do not result in a significant change (<1%) in the volume of sample extracted.

Quantitatively transfer the sample from the graduated cylinder (or sample bottle) to the separatory funnel. Use 60 mL of methylene chloride to rinse the cylinder (or bottle) and transfer this rinse solvent to the separatory funnel. If the sample was transferred directly from the sample bottle, refill the bottle to the mark made with water and then measure the volume of sample that was in the bottle.

Seal and shake the separatory funnel vigorously for 1 - 2 minutes with periodic venting to release excess pressure. Alternatively, pour the exchange solvent into the top of the Snyder column while the concentrator remains in the water bath.

NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken

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once. The separatory funnel should be vented into a hood to avoid exposure of the analyst to solvent vapors.

Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the solvent extract in an Erlenmeyer flask. If the emulsion cannot be broken optionally (recovery of < 80% of the methylene chloride, corrected for the water solubility of methylene chloride),the sample, solvent, and emulsion may be transferred into the extraction chamber of a continuous extractor and proceed as described in Method 3520, Continuous Liquid-Liquid Extraction.

Repeat the extraction two more times using fresh portions of solvent. Combine the three solvent extracts.

If further pH adjustment and extraction is required, adjust the pH of the aqueous phase to the desired pH indicated in Table 1. Serially extract three times with 60mL of methylene chloride. Collect and combine the extracts and label the combined extract appropriately.

If performing GC/MS analysis (Method 8270), the acid/neutral and base extracts may be combined prior to concentration. However, in some situations, separate concentration and analysis of the acid/neutral and base extracts may be preferable (e.g. if for regulatory purposes the presence or absence of specific acid/neutral or base compounds at low concentrations must be determined, separate extract analyses may be warranted).

Perform the concentration (if necessary) using the Kuderna-Danish Technique.

5.2a K-D technique (concentration)

Assemble a Kuderna-Danish (K-D) concentrator (Sec. 4.3) by attaching a 10mL concentrator tube to a 500mL evaporation flask.

Attach the solvent vapor recovery glassware (condenser and collection device) to the Snyder column of the K-D apparatus following manufacturer's instructions.

Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride and add it to the column to complete the quantitative transfer.

Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (15 - 20°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 - 20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

If a solvent exchange is required (as indicated in Table 1), momentarily remove the Snyder column, add

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50 mL of the exchange solvent, a new boiling chip, and reattach the Snyder column. Alternatively, pour the exchange solvent into the top of the Snyder column while the concentrator remains in the water bath. Concentrate the extract, raising the temperature of the water bath, if necessary, to maintain proper distillation.

Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1 - 2 mL of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the technique outlined below or adjusted to 10.0 mL with the solvent last used.

5.2b Glass vessel technique (concentration)

Dry the extract by passing it thru sodium sulfate (Na_2SO_4) into a wide base, narrow mouth glass vessel.

Next without boiling evaporate the extract at a moderate rate to less than 10mL in a warm water bath.

If further concentration is indicated in Table 1, either the micro-Snyder column technique or nitrogen blowdown technique is used to adjust the extract to the final volume required.

Micro-Snyder column technique

If further concentration is indicated in Table 1, add another clean boiling chip to the concentrator tube and attach a two-ball micro-Snyder column. Pre- wet the column by adding 0.5 mL of methylene chloride or exchange solvent to the top of the column. Place the K-D apparatus in a hot water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 5 - 10 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column, rinse the flask and its lower joints into the concentrator tube with 0.2 mL of methylene chloride or the exchange solvent, and adjust the final volume as indicated in Table 1, with solvent.

Nitrogen blowdown technique

Place the concentrator tube in a warm bath and without boiling evaporate the solvent to the final volume indicated in Table 1, using a gentle stream of clean, dry nitrogen.

CAUTION: New plastic tubing must not be used between the carbon trap and the sample, since it may introduce contaminants.

The internal wall of the tube must be rinsed several times with methylene chloride or appropriate solvent during the operation. During evaporation, the tube must be positioned to avoid water condensation (i.e., the solvent level should be below the level of the water bath). Under normal procedures, the extract must not be allowed to become dry.

CAUTION: When the volume of solvent is reduced below 1 mL, semi-volatile analytes may be lost.

The extract may now be analyzed for the target analytes using the appropriate determinative

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technique(s). If analysis of the extract will not be performed immediately, stopper the concentrator tube and store refrigerated. If the extract will be stored longer than 2 days it should be transferred to a vial with a PTFE-lined screw-cap or crimp top, and labeled appropriately.

6.0 Quality Control Requirements

The objective reporting limits can be found in Appendix A

6.1 QC Elements

Demonstration of capability (DOC), four replicates of a prepared quality control sample. Required with a change of instrument, method, or analyst.

Method detection limit (MDL) study, seven replicates (standard deviation * 3.14). Performed annually, with change in instrument, or method.

Limit of quantitation (LOQ), a single quality control sample, no more than 1-2x the reporting limit. Performed annually.

Analyze a blank, laboratory control, laboratory control sample duplicate, sample spike, and sample duplicate every analytical batch, every twenty samples, or as provided in the prep method.

NOTE: Sample matrix spikes and matrix spike duplicates are extracted either per client request or per project requirements. Every attempt will be made to obtain enough sample to perform a matrix spike with 10 % frequency. If a single sample needs to be extracted and there is not enough sample volume for a matrix spike, a synthetic sample, or LCS duplicate may be extracted and analyzed to provide 10% spike occurrence.

All QC elements and samples must be spiked with a surrogate standard.

QC elements may be added or removed to meet specific program or project requirements.

6.2 Acceptance Criteria

Demonstration of capability samples must meet LCS acceptance criteria.

The calculated MDL must be lower than the reporting limit.

The limit of quantitation standard acceptance criteria is 30-150%.

Acceptance criteria for all blanks is less than the reporting limit (see Appendix A) or less than 1/10 of sample value.

Acceptance criteria for LCS/LCSD recovery is 30%-150%, unless specified in method(See Appendix B). Acceptance criteria for LCSD percent relative range is less than or equal to 30%.

Acceptance criteria for relative percent difference between sample duplicates and matrix spike duplicates is less than or equal to 30%.

Acceptance criteria for spiked sample recovery is 30%-150%.

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Acceptance limits for surrogate standard recovery can be found in Appendix B.

Alternate acceptance criteria may be set for specific programs or projects.

6.3 Interferences

Refer to Method 3500.

The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510. Method 3510 is preferred over Method 3520 for the analysis of these classes of compounds. However, the recovery of phenols may be optimized by using Method 3520, and performing the initial extraction at the acid pH.

7.0 Calculations/Reporting

7.1 Record

Record the following:

1. Date
2. Initials of analyst
3. Sample ID, with dilution factor if applicable
4. Batch
5. Sample Code / Standard Code
6. Solvent lot ID
7. Surrogate ID and amount, with dilution factor if applicable
8. Spike ID and amount , with dilution factor if applicable
9. Initial volume
10. Final volume
11. pH
12. Method
13. Notes, if any

7.2 Calculations

Calibrate the system software as per the Agilent manuals referenced in section 3.0. Use the system software to calculate the semi-volatile values for QC elements and samples. Review the data to ensure it is correct and that all quality control requirements have been met. When needed manually integrate the values according to ESI SOP #1132 *Manual Chromatographic Peak Integration*.

Calculate LCS/SUR Recovery as follows:

$$\frac{\text{LCS/SUR concentration}}{\text{Concentration added}} \times 100 = \%\ \text{recovery}$$

Calculate %RPD as follows:

$$\frac{\text{Abs(Result 1 - Result 2)}}{\text{Avg. of results}} \times 100 = \%\text{RPD}$$

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Calculate Spike Recovery as follows:

$$\frac{\text{Spiked sample concentration - Sample concentration}}{\text{Concentration added}} \times 100 = \% \text{ recovery}$$

8.0 Corrective Actions

8.1 Data Qualification

DOC's, MDL's, and LOQ's should be redone until they meet acceptance criteria.

Samples analyzed with a prep blank not meeting acceptance criteria may be reanalyzed or reported with qualification (B).

If the LCS or LCSD's do not meet percent recovery and/or percent relative difference (RPD) acceptance criteria they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (J).

Samples analyzed in duplicate not meeting percent relative difference (RPD) acceptance criteria may be reported with qualification (J) or reanalyzed. If the sample result or sample duplicate result is less than 5 times the reporting limit, the percent relative difference need not be evaluated.

If the spiked sample recovery is not within acceptance criteria samples may be reported with qualification (J) or reanalyzed. If the sample result is greater than 5 times the spike amount the percent recovery need not be calculated.

If surrogate recovery is not within the acceptable range, the sample is re-extracted whenever possible. Sample matrix effect is proven if the results obtained from the re-extracted sample show similar results. When this occurs, both results are reported with an explanation. A problem with the first extraction is indicated when the results of the re-extracted sample are in the acceptable range. In this case, only the compliant results are reported. If no additional sample can be obtained, results are flagged in the report and a narrative is included qualifying the data.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified:

1. Quality and expiration date of reagents
2. Cleanliness of containers, equipment, and labware
3. Integrity of standards
4. GC/MS operation

9.0 Health and Safety

Methylene chloride and semi-volatile standards are extremely hazardous to everyone in the laboratory if handled improperly. It is important that all necessary safety precautions are followed.

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Gloves and safety glasses must be worn when handling samples, reagents, and standards. It is necessary to wear a lab coat to protect clothing. All solvent usage must occur in properly working fume hoods.

Analysts should familiarize themselves with any chemical substances which are named along with their corresponding MSDS sheets.

Strong acids and bases should be added to containers and solutions under the hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

10.0 Responsibilities

It is the analyst's responsibility to read and follow this standard operating procedure. Successfully perform a demonstration of capability. Record all data as per section 7.0. Review all QC results for compliance with acceptance criteria.

It is the lab supervisor's responsibility to make sure standard operating procedures are up to date and that acceptance criteria have been set.

11.0 Waste Management and Pollution Prevention

Unused non-hazardous sample may be disposed of as wastewater.

Solvents and extracted sample not evaporated in a hood are collected and disposed of as hazardous waste in designated solvent water waste containers.

Minimize solvent use. Prepare standards in amounts that will be used before expiration. Dispose of samples and wastes in accordance with ESI policy, state, and federal regulation.

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TITLE: Extraction of Pentachlorophenol and other ABN Compounds in Aqueous Samples

TABLE 1
SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Deter-minative method	Initial extraction pH	Secondary extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for cleanup (mL)	Final extract volume for analysis (mL)[a]
8041	≤2	none	2-propanol	hexane	1.0	1.0, 0.5[b]
8061	5-7	none	hexane	hexane	2.0	10.0
8070	as received	none	methanol	methylene chloride	2.0	10.0
8081	5-9	none	hexane	hexane	10.0	10.0
8082	5-9	none	hexane	hexane	10.0	10.0
8091	5-9	none	hexane	hexane	2.0	1.0
8100	as received	none	none	cyclohexane	2.0	1.0
8111	as received	none	hexane	hexane	2.0	10.0
8121	as received	none	hexane	hexane	2.0	1.0
8141	as received	none	hexane	hexane	10.0	10.0
8270[c,d]	<2	>11	none	-	-	1.0
8310	as received	none	acetonitrile	-	-	1.0
8321	as received	none	methanol	-	-	1.0
8325	7.0	none	methanol	-	-	1.0
8410	as received	none	methylene chloride	methylene chloride	10.0	0.0 (dry)

[a] For methods where the suggested final extract volume is 10.0 mL, the volume may be reduced to as low as 1.0 mL to achieve lower detection limits.
[b] Phenols may be analyzed, by Method 8041, using a 1.0 mL 2-propanol extract by GC/FID. Method 8041 also contains an optional derivatization

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procedure for phenols which results in a 0.5 mL hexane extract to be analyzed by GC/ECD.

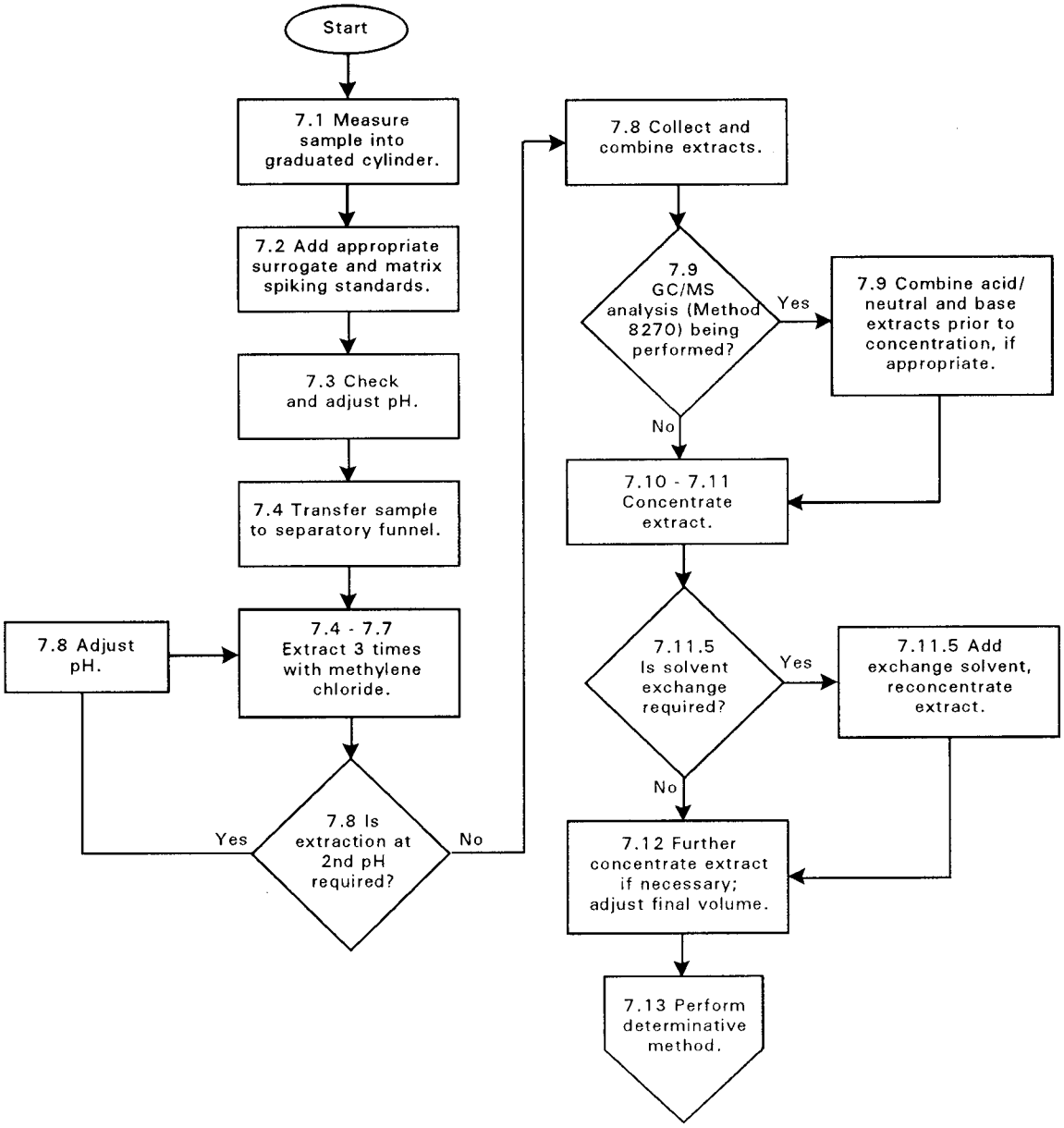
[c] The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available if required.

[d] Extraction pH sequence may be reversed to better separate acid and neutral waste components. Excessive pH adjustments may result in the loss of some analytes (see Sec. 3.2).

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TITLE: Extraction of Pentachlorophenol and other ABN Compounds in Aqueous Samples



APPENDIX A

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TITLE: Extraction of Pentachlorophenol and other ABN Compounds in Aqueous Samples

Parameter	RL(ug/L)	Parameter	RL(ug/L)	Parameter	RL(ug/L)
1,2,4-trichlorobenzene	5	4-chloroaniline	5	carbazole	5
1,2-dichlorobenzene	5	4-chlorophenyl-phenylether	5	chrysene	5
1,2-diphenylhydrazine (azo)	5	4-methylphenol (p-cresol)	5	di-n-butylphthalate	5
1,3-dichlorobenzene	5	4-nitroaniline	5	di-n-octylphthalate	5
1,4-dichlorobenzene	5	4-nitrophenol	10	dibenzo(a,h)anthracene	5
1-methylnaphthalene	5	N-nitroso-di-n-propylamine	5	dibenzofuran	5
2,4,5-trichlorophenol	5	N-nitrosodimethylamine	5	diethylphthalate	5
2,4,6-trichlorophenol	5	N-nitrosodiphenylamine	5	dimethylphthalate	5
2,4-dichlorophenol	5	acenaphthene	5	fluoranthene	5
2,4-dimethylphenol	5	acenaphthylene	5	fluorene	5
2,4-dinitrophenol	10	aniline	5	hexachloro-1,3-butadiene	5
2,4-dinitrotoluene	5	anthracene	5	hexachlorobenzene	5
2,6-dichlorophenol	5	benzidine	20	hexachlorocyclopentadiene	5
2,6-dinitrotoluene	5	benzo(a)anthracene	5	hexachloroethane	5
2-chloronaphthalene	5	benzo(a)pyrene	5	indeno(1,2,3-cd)pyrene	5
2-chlorophenol	5	benzo(b)fluoranthene	5	isophorone	5
2-methylnaphthalene	5	benzo(g,h,i)perylene	5	naphthalene	5
2-methylphenol	5	benzo(k)fluoranthene	5	nitrobenzene	5
2-nitroaniline	5	benzoic acid	50	pentachlorophenol	10
2-nitrophenol	5	benzyl alcohol	5	phenanthrene	5
3,3'-dichlorobenzidine	20	bis(2-chloroethoxy)methane	5	pyrene	5
3-nitroaniline	5	bis(2-chloroethyl)ether	5	pyridine	5
4,6-dinitro-2-methylphenol	20	bis(2-chloroisopropyl)ether	5		
4-bromophenyl-phenylether	5	bis(2-ethylhexyl)phthalate	5		
4-chloro-3-methylphenol	5	butylbenzylphthalate	5		

APPENDIX B

Parameter	LCS %	Parameter	LCS%	Parameter	LCS %
1,2,4-trichlorobenzene	44-142	4-chloroaniline	30-150	carbazole	30-150

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TITLE: Extraction of Pentachlorophenol and other ABN Compounds in Aqueous Samples

1,2-dichlorobenzene	32-129	4-chlorophenyl-phenylether	25-158	chrysene	17-168
1,2-diphenylhydrazine (azo)	30-150	4-methylphenol (p-cresol)	30-150	di-n-butylphthalate	1-118
1,3-dichlorobenzene	1-172	4-nitroaniline	30-150	di-n-octylphthalate	4-146
1,4-dichlorobenzene	20-124	4-nitrophenol	1-132	dibenzo(a,h)anthracene	1-227
1-methylnaphthalene	30-150	N-nitroso-di-n-propylamine	1-230	dibenzofuran	30-150
2,4,5-trichlorophenol	30-150	N-nitrosodimethylamine	30-150	diethylphthalate	1-114
2,4,6-trichlorophenol	37-144	N-nitrosodiphenylamine	30-150	dimethylphthalate	1-112
2,4-dichlorophenol	39-135	acenaphthene	47-145	fluoranthene	30-150
2,4-dimethylphenol	32-119	acenaphthylene	33-145	fluorene	59-121
2,4-dinitrophenol	1-191	aniline	30-150	hexachloro-1,3-butadiene	24-116
2,4-dinitrotoluene	39-139	anthracene	27-133	hexachlorobenzene	1-152
2,6-dichlorophenol	30-150	benzidine	30-150	hexachlorocyclopentadiene	30-150
2,6-dinitrotoluene	50-158	benzo(a)anthracene	33-143	hexachloroethane	40-113
2-chloronaphthalene	60-118	benzo(a)pyrene	17-163	indeno(1,2,3-cd)pyrene	1-171
2-chlorophenol	23-134	benzo(b)fluoranthene	24-159	isophorone	21-196
2-methylnaphthalene	30-150	benzo(g,h,i)perylene	1-219	naphthalene	21-133
2-methylphenol	30-150	benzo(k)fluoranthene	11-162	nitrobenzene	35-180
2-nitroaniline	30-150	benzoic acid	30-150	pentachlorophenol	14-176
2-nitrophenol	29-182	benzyl alcohol	30-150	phenanthrene	54-120
3,3'-dichlorobenzidine	1-262	bis(2-chloroethoxy)methane	33-184	pyrene	52-115
3-nitroaniline	30-150	bis(2-chloroethyl)ether	12-158	pyridine	30-150
4,6-dinitro-2-methylphenol	1-181	bis(2-chloroisopropyl)ether	36-166		
4-bromophenyl-phenylether	53-127	bis(2-ethylhexyl)phthalate	8-158		
4-chloro-3-methylphenol	22-147	butylbenzylphthalate	1-152		

APPENDIX B

Parameter	Surrogate %	Parameter	Surrogate %
2-fluorophenol	21-100	nitrobenzene-d5	35-114

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TITLE: Extraction of Pentachlorophenol and other ABN Compounds in Aqueous Samples

phenol-d5	10-102	2-fluorobiphenyl	43-116
2,4,6-tribromophenol	10-123	terphenyl-d14	33-141

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TITLE: Determination of Trace Metals by ICP-MS Analysis

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Draft Preparation	R. Foster	11/2007
1	Edits and Revisions	D. Shartrand	12/2007
2	Edit	M. Scott	05/2008
3	Review and update	M. Scott	12/2009
4	Reformat and Update	M. Scott	12/2011
5	Update based on audit response	M. Scott	03/2012
6	Update based on audit response	M. Scott	06/2012
7	Addition of substrate requirement for LCS and Blank	K. A. Simon	07/2012
8	Update of procedure to include interference check standard as part of analytical session	M. Scott	09/2014
9	Update to include ICSA/AB acceptance criteria, addition of post digestion spike and dilution test for 6020	J. Hobbs	06/2016

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1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the method for determining concentrations of trace metals in aqueous or extracted samples; calcium and magnesium concentrations are determined for the calculation of hardness values (See SOP QA-1309 "Computation of Hardness by Calculation Method").

Inductively Coupled Plasma (ICP-MS) instrumentation is used to determine the concentration of trace metals, Method 200.8 *Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry* Revision 5.4 EMMC Version. The applicable range for this method varies by element and is responsive to the requirements of the method and regulatory agencies.

Samples are preserved to a pH of <2.0 SU with HNO_3 and held at ambient temperature until analysis for up to 180 days. Samples may be digested prior to analysis (See SOP QA-1323 "Sample Preparation; Trace Metals Digestion"). The ICP makes multi-element determinations by measuring the electron multiplier ion intensities at characteristic atomic masses. The characteristic atomic masses are passed by a mass spectrometer sampling an argon plasma. Samples are aerosolized and applied to the plasma torch. Sample intensity is evaluated against both linear calibrations and quality control samples.

2.0 Definitions

Calibration (CAL) Standard: A solution prepared from the dilution of stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

Calibration Blank: The Initial Calibration Blank (ICB) and the Continuing Calibration Blank (CCB); A volume of reagent water acidified with the same acid matrix as the calibration standards. The calibration blank is a zero standard and is used to calibrate the ICP instrument and the analytical calibration curve.

Inference Check Solution (ICS): A solution prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of inferences and provide an adequate test of any corrections. The interference check solution A (ICSA) contains analytes that may cause interfence. The inference check solution AB (ICSAB) contains analytes that may cause interfence and target analytes. The ICSA and ICSAB solutions are run at the start of each method 6020 session and every 12 hours.

Instrument Calibration Verification: The Initial Calibration Verification (ICV) and the Continuing Calibration Verification (CCV); Instrument checks must be analyzed to verify instrument performance. The standard used must be obtained from an outside source different from the standard stock solutions and prepared in the same acid mixture as the calibration standards. The instrument calibration standard is prepared at the same concentration as the CAL standard.

Instrument Detection Limit (IDL): The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 7 replicate measurements of the calibration blank signal at the selected analytical masses.

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Internal Standard: Pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

Protocol Required Reporting Limit (PRRL) Solution: Laboratory reagent water fortified with analyte at the reporting limit. Required for some protocols.

Stock Standard Solution: A concentrated solution containing one or more method analytes prepared In the laboratory using assayed reference materials or purchased from a reputable commercial source.

Tuning Solution: A solution that is used to determine acceptable instrument performance prior to calibration and sample analyses.

See ESI's Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

Agilent Technologies, Inc. *Agilent 7500 series ICP-MS Hardware Manual.*

Agilent Technologies, Inc. *Agilent 7500 series ICP-MS Operator's Manual.*

Agilent Technologies, Inc. *Agilent 7800 series ICP-MS Hardware Manual.*

Agilent Technologies, Inc. *Agilent 7800 series ICP-MS Operator's Manual.*

US EPA. 1994. Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry Method 200.8.

Department of Defense (DoD), Department of Energy (DoE). 2013. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.0.

US EPA. 1996. *Inductively Coupled plasma Mass Spectrometry.* Method 6020.

SOP QA-1323 "Sample Preparation; Trace Metals Digestion"
SOP QA-1324 "Preparation of Glassware for Trace Metals Analysis"
SOP QA-1370 "Sample Preparation; Trace Metals Digestion of Tissue"

4.0 Materials and Apparatus

4.1 Reagents and Standards

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date. Solutions are stored at

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ambient temperature when not in use; solutions must be used in advance of their expiration dates.

Trace metals standards are prepared most frequently from commercially available standard solutions, in either single or multi-element concentrations. Standards are diluted using Milli-Q® water,1.5% HCl/6.5% HNO_3 solution, and volumetric glassware to achieve the desired concentrations. Standards are stored at ambient temperature away from sources of light and prepared daily with instrument use. Note that standard preparations may be altered to meet project specific criteria. Refer to Table 1 for standard volume preparations.

Argon gas, high purity (99.9998%)
Hydrogen gas, high purity (99.999%)
Helium gas, high purity (99.999%)
Interference A and AB Solutions, commercially prepared
Concentrated hydrochloric acid (~35%), ultra-high purity grade
Concentrated nitric acid (~70%), ultra-high purity grade
Milli-Q® water
Tuning solution, commercially prepared
Commercially available standards (single or multi-element standards), in both "high" and "low" concentrations, or ultra-high purity compounds

4.1.1 1% HCl/ 1% HNO_3 Solution

The solution is prepared daily by adding 36 mL of HNO_3 and 71 mL of HCl to 2 L of Milli-Q® water, then bringing to a final volume of 2.5 L.

4.1.2 ICV Intermediate Standard

Fill a 50 mL Class A volumetric flask with approximately 35 mL of Milli-Q® water. Add 5 mL of HNO_3 and 2.5 mL of HCl to the flask, spike with standards according to Table 1, then dilute to 50 mL with Milli-Q® water.

4.1.3 CCV Intermediate Standard

Fill a 100 mL Class A volumetric flask with approximately 70 mL of Milli-Q® water. Add 10 mL of HNO_3 and 5 mL of HCl to the flask, spike with standards according to Table 1, then dilute to 100 mL with Milli-Q® water.

4.1.4 CAL and CCV Standards

Dilute 5 mL of the CCV intermediate standard to 50 mL with 1% HNO_3 / 1% HCl and spike with 250 µL Internal Standard (final concentration is 50 ppb).

4.1.5 ICV Standard

Dilute 5 mL of the ICV intermediate standard to 50 mL with 1% HNO_3 / 1% HCl and spike with 250 µL Internal Standard (final concentration is 50 ppb).

4.1.6 ICB and CCB Standard

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Spike 50 mL of the 1% HNO_3 / 1% HCl with 250 µL of a multi-element internal standard. A 10 mL solution of the internal standard will yield an internal standard concentration of 50 ppb.

4.1.7 Reporting Limit (RL) Standard

Reporting Limit	Preparation
0.5	10 mL of 1% HNO_3 / 1% HCl final volume spiked with 0.1 µL of ICV Intermediate Standard
0.1	10 mL of 1% HNO_3 / 1% HCl final volume spiked with 0.1 µL of ICV Intermediate Standard
0.05	10 mL of 1% HNO_3 / 1% HCl final volume spiked with 0.01 µL of ICV Intermediate Standard

4.1.8 ICSA and ICSAB Solution

Dilute commercially available ICSA and ICSAB standard 1:10 with 1% HNO_3 / 1% HCl.

4.1.9 Post Digestion Spike (PDS)

Dilute 1.0 mL ICV Intermediate Standard to 10 mL final volume with 1% HNO_3 / 1% HCl. Sample to be spiked with PDS should be the same as the spike duplicate.

4.2 Equipment and Supplies

Adjustable volumetric pipettes
Agilent 7500 series inductively coupled plasma mass spectrometer with autosampler, data station -or-
Agilent 7800 series inductively coupled plasma mass spectrometer with autosampler, data station
Autosampler racks, capable of holding vials
Block digester
Class A volumetric glassware
Chemstation Version B.03.03 software -or-
MassHunter Version 4.2
Ice, prepared from high quality Milli-Q® water
Polyethylene or teflon autosampler vials
Pre-cleaned glass beads
Solid substrate
Teflon® chips

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5.0 Methods and Procedures

Samples should be collected in 250 to 500 mL plastic disposable bottles. Samples received for metals are preserved with HNO_3 to a pH of <2 SU and have a holding time of 180 days.

The manufacturer's operation manual for the ICP-MS instrumentation preparation and operating conditions should be consulted for equipment specifics.

5.1 System Start Up

5.1.1 Ensure that the cooling fan is on and check the outside of the fan's grate cover to ensure that the fan is running. Do not attempt to start the system up unless the cooling fan is running.

5.1.2 Turn on the water recirculator and start the plasma using system software. Monitor system meters that indicate problems with the pump, gas, etc.

5.1.3 Ensure that all tubing lines are properly connected. Check the tension on the peristaltic pump; sample should be flowing continuously into and out of the nebulizer. Observe tubes for flow.

5.1.4 Select target elements and set up instrument acquisition and quantitation parameters in the system software according to manufacturer's recommendations and based on the needs of the analytical session.

5.2 Instrument Pre-Calibration Routine

The following pre-calibration routine must be completed prior to calibrating the instrument until it can be documented with periodic performance data that the instrument meets the criteria listed below without daily tuning.

5.2.1 Initiate proper operating configuration of instrument and data system. Allow a period of about 30 minutes for the instrument to warm up, or until the spray chamber cooler temperature reaches 2±1°C. During this process, conduct mass calibration and resolution checks using the tuning solution.

Resolution at low mass is indicated by magnesium isotopes 24, 25, and 26. Resolution at high mass is indicated by lead isotopes 206, 207, and 208. For high quality performance, adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass for any of the tune elements.

5.2.2 Instrument stability must be demonstrated by running the tuning solution a minimum of five times with resulting relative standard deviations of absolute signals for all analytes of <5%.

5.3 Internal Standardization for Instrument Calibration

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Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. For full mass range scans, a minimum of three internal standards must be used. Procedures described in this method for general application detail the use of five internal standards: scandium, yttrium, indium, terbium and bismuth. These standards were used to generate the precision and recovery data attached to this method.

5.3.1 Internal standards must be present in all samples, standards and blanks at identical levels. This may be achieved by directly adding an aliquot of the internal standards to the CAL standard, blank or sample solution, or alternatively by mixing with the solution prior to nebulization using a second channel of the peristaltic pump and a mixing coil.

5.3.2 The concentration of the internal standard should be sufficiently high so that good precision is obtained in the measurement of the isotope used for data correction and in order to minimize the possibility of correction errors if the internal standard is naturally present in the sample. Depending on the sensitivity of the instrument, a concentration range of 20 to 200 µg/L of each internal standard is recommended. Internal standards should be added to blanks, samples and standards in a like manner so that dilution effects resulting from the addition may be disregarded.

5.4 Instrument Calibration

5.4.1 Prepare the CCV, CCB, ICV and ICB standards and place into the appropriate plastic bottles in the autosampler area.

5.4.2 Aspirate the tuning solution and verify that mass assignment and stability tuning criteria are met. Aspirate a blank sample to set instrument intercept and aspirate a medium level standard to set instrument slope.

5.4.3 Aspirate the ICV check solution and determine if the ICV meets criteria. Aspirate the ICB check solution and determine if the ICB meets criteria.

5.4.4 Run a RL standard or Protocol Required Reporting Limit (PRRL) Solution after the ICB and verify that results are within the program acceptance criteria.

5.4.5 Interfence check solutions (ICSA and ICSAB) must be run unless the instrument is being calibrated using matrix matching techniques that have been demonstrated to meet program criteria within the last year.

5.5 Blank Preparation

Laboratory blank samples will be prepared in the same manner as routine sediment or tissue samples with the exception that the blank includes a clean substrate. Acceptable substrates may include pre-cleaned glass beads (for a soil/sediment matrix) orTeflon® chips (for a tissue matrix). Ice, prepared from ≥18 µmhos/cm or Milli-Q® water may also be used as a solid substrate.

5.6 Laboratory Control Sample (LCS) Preparation

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LCS and Laboratory Control Sample Duplicate (LCSD) samples will be prepared in the same manner as routine sediment or tissue samples with the exception that the sediment or tissue is replaced with a clean substrate. Acceptable substrates may include: pre-cleaned glass beads for a soil/sediment matrix orTeflon® chips for tissue matrix. Ice, prepared from ≥18 µmhos/cm or Milli-Q® water may also be used as a solid substrate.

Alternately, a SRM (Standard Reference Material) may be used as a LCS/LCSD.

5.7 Sample Analysis

Samples for trace metals analysis are digested prior to their ICP analysis. See SOP QA-1323 "Sample Preparation; Trace Metals Digestion" or SOP QA-1370 "Sample Preparation; Trace Metals Digestion of Tissue" for instruction.

Place samples in the autosampler and verify correct entry in the ICP-MS software system sequence table, then initiate sample analysis.

5.8 Instrument Shut Down

The ICP-MS may be shut down manually through the instrument operation panel or automated through system sequence table programming. After the plasma is off the water circulator may be shut down. Unload tension from peristaltic pum p tubing. Do not turn off the exhaust fan.

6.0 Quality Control Requirements

Quality Control elements may be added, removed, or altered to meet specific program or project requirements. Alternate acceptance criteria may be set for specific programs or projects. For Department of Defense projects reference QSM 5.0, as appropriate.

Note that all quality control elements and samples must be spiked with an internal standard.

6.1 Demonstration of Capability (DOC)

An analyst must complete a DOC before working independently on this procedure and yearly thereafter. A DOC is also required with any changes to instrument, method, or analyst. To perform a DOC, four replicates of a prepared quality control sample must be prepared. DOC samples must meet LCS acceptance criteria. For more information on DOCs see SOP QA-1108 "Training".

6.2 Method Detection Limit (MDL)

The MDL is calculated using the appropriate student's t-parameter value multiplied by the standard deviation of 7 or more replicates of spike samples or standards. MDLs are performed annually and with changes in instrument or method. The calculated MDL must be lower than the reporting limit.

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The t-parameter value is from a one-tailed Student's t-Test at the 99% confidence interval using n-1 degrees of freedom, with "n" being the number of replicates.

The most recent MDL study can be found in the Inorganics Quality Control directory on the ESI network. For more information on MDLs see SOP QA-1111 "Determination of Method Detection and Reporting Limits".

6.3 Limit of Detection (LOD)

LOD determination will be conducted on a quarterly basis. For a single-analyte standard, the LOD is a single quality control sample that is approximately 2 to 3 times the MDL but no greater than the reporting limit. For a multi-analyte standard, the LOD is a single quality control sample that is approximately 1 to 4 times the MDL but no greater than the reporting limit.

The apparent signal to noise ratio at the LOD must be at least 3. For methods that do not produce a signal to noise, the LOD result must be at least 3 standard deviations greater than the mean method blank.

6.4 Limit of Quantitation (LOQ)

LOQ confirmation will be conducted on a quarterly basis. The LOQ is a single control sample no more than 1 to 2 times the reporting limit. The LOQ standard acceptance criteria is 70-130%. For more information on LODs and LOQs see SOP QA-1147 "Determination of LOD and LOQ Quality Control Samples".

6.5 Blank, LCS/LCSD, Matrix Spike (MS), Sample Duplicate, Relative Percent Difference (RPD)

6.5.1 Analyze a blank, LCS, and if desired a LCSD every analytical batch or 20 samples, whichever is more frequent. For solid samples, soils/sediment and tissues, the blank and LCS must include a non-contaminating substrate.

6.5.2 Acceptance criteria for all blanks is less that the reporting limit or less than 1/10th of sample value.

6.5.3 Analyze MS samples at a rate of 10%, or as otherwise specified by a client-specific scope of work.

6.5.4 When a PDS is analyzed, the acceptance recovery is 80 to 120%. If a dilution test is performed due to PDS failure, then a sample analyzed at a 1:5 dilution should be within ±10% of the original determination, if the analyte concentration was at least 10 times higher than the LOQ after dilution (method 6020 only).

6.5.5 Acceptance criteria for LCS/LCSD recovery is 85-115% and for LCSD RPD is less that or equal to 20%.

6.5.6 Acceptance criteria for MS recovery is 80-120%.

6.5.7 Acceptance criteria for RPD between sample duplicates is less than or equal to 20%.

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6.6 ICV, CCV, ICB and CCB

Note that the system calibration may be updated with the CCV and CCB, provided that they are followed by a ICV, ICB, reporting limit standard re-analysis and ICSA / ICSAB if needed.

6.6.1 Initial calibration must consist of a blank and one standard. ICV and ICB are performed following analysis of initial calibration standards. Acceptance criteria for the ICV is ±10%.

6.6.2 CCV and CCB are performed every ten samples and at the end of an analytical session. Acceptance criteria for the CCV is ±10% of references values.

6.6.3 The value of the ICB or CCB should be no higher than the 2.2 times the IDL or less that 1/10thof the reported values.

6.7 Linear Dynamic Range (LDR)

The LDR is the concentration range over which an analyte's relative response is within 10% of the initial calibration concentration. The linear dynamic range will be conducted on a quarterly basis or with change in instrument. It must be verified to a response of 90 to 110% recovery over the desired range of analysis.

6.8 ICSA and ICSAB Solutions

Acceptance criteria for the ICSA is that all non spiked analytes be lower than the LOD, unless there are verified impurities from the spike analytes. Acceptance criteria for ICSAB is 80 to 120% recovery. When necessary, interference check standards must be run at the beginning of the analytical run and/or every 12 hours, whichever is more frequent. Note that this acceptance criteria is for method 6020 only.

6.9 Reporting Limit (RL) Standard

The RL standard is performed immediately following ICB analysis. The acceptance criteria is ±40%.

6.10 Tuning Solution

The tuning solution must be performed at the beginning of each analytical session. The tune check must verify peak assignment within 1.0 amu, and an RPD of five replicate measurements verified to be < 5%.

6.11 Internal Standard Response

Acceptance criteria for the internal standard response (IS) is 60 to 125% recovery of the response for the preceding CCB.

6.12 The objective reporting limits vary from 0.0005 to 1.0 mg/L.

6.13 Interferences

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Samples submitted for boron or silica analysis should be prepared, stored and analyzed in plastic, PTFE or quartz vessels, from sample collection to the completion of sample analysis to prevent boron or silica from causing inference in the analysis. Additionally, sample dilution may minimize background intensities and inter-element corrections are set in the system software in compliance with EPA 200.8.

7.0 Calculations and Reporting

7.1 The following information should be recorded when performing a determination of trace metals by ICP-MS analysis:

- Date
- Analyst's initial
- Batch
- Sample ID / Standard ID, with dilution factor if applicable
- Amount / ID of standards
- Data file name
- Acquisition method
- Pipette / Scale IDs, where applicable
- Notes, if any

7.2 Calculations

Calibrate the system software as per the Agilent manuals referenced in section 3.0. Use the system software to calculate the metals values for quality control elements and samples. Review the data to ensure it is correct and that all quality control requirements have been met. Note that a project or program may require that sample results be at or below the daily single calibration point. Values higher than that single calibration point must be diluted and reanalyzed or qualified as not meeting protocol specific criteria.

7.2.1 Calculate LCS/IS/ICV/CCV Recovery as follows:

$$\frac{\text{LCS/IS/ICV/CCV Concentration}}{\text{Concentration added}} \times 100 = \%\text{Recovery}$$

7.2.2 Calculate %RPD as follows:

$$\frac{\text{Abs(Result 1 - Result 2)}}{\text{Average of results}} \times 100 = \%\text{RPD}$$

7.2.3 Calculate Spike Recovery as follows:

$$\frac{\text{Spiked Sample Concentration - Sample Concentration}}{\text{Concentration added}} \times 100 = \%\text{Recovery}$$

7.3 Reporting

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Results less than 10 ppb will be reported to 2 significant figures and results greater than 10 ppb will be reported to 3 significant figures, unless otherwise specified by the project plan.

8.0 Corrective Actions

8.1 Data Qualification

8.1.1 If DOCs, MDLs, LODs, LOQs, LDRs, and tune checks do not meet acceptability criteria, then repeat until acceptability criteria is met.

8.1.2 If internal standard response is outside limits, then qualify associated sample result as estimated (using the "J" qualifier).

8.1.3 If the ICV does not meet acceptance criteria, then prepare a new curve and ICV or qualify (using the "J" qualifier) any positive sample values as estimated.

8.1.4 If the ICSA or ICSAB does not meet acceptance criteria, then re-analyze the associated analytical sequence or qualify affected metals (using the "J" qualifier).

8.1.5 If samples analyzed with blanks (prep, initial, continuing) not meeting acceptance criteria, then they may be re-analyzed or reported with qualification (using the "B" qualifier).

8.1.6 If MS/MSD recoveries are unacceptable, then prepare and analyze a PDS from the same sample that the MS/MSD were prepared from. If the PDS does not meet acceptance criteria, then a dilution test should be performed at a 1:5 ratio (method 6020 only).

8.1.7 If the LCS or LCSDs do not meet percent recovery and/or RPD acceptance criteria, then they should be re-analyzed along with all associated QC and samples. Samples may be reported with qualification (using the "J" qualifier).

8.1.8 If samples analyzed in duplicate do not meeting RPD acceptance criteria, then they may be reported with qualification (using the "J" qualifier) or re-analyzed. If the sample result or sample duplicate result is less than 5 times the reporting limit, then the RPD need not be evaluated.

8.1.9 If the MS recovery or PDS recovery is not within acceptance criteria, then samples may be reported with qualification (using the "J" qualifier) or re-analyzed. If the sample result is greater than 5 times the spike amount, then the percent recovery need not be calculated.

8.1.10 If a dilution test does not meet acceptance criteria, then the results may be re-analyzed or reported with qualification (using the "J" qualifier).

8.1.11 If a CCV does not meet acceptance criteria, then all associated samples must be re-analyzed or qualified as estimated (using the "J" qualifier) .

8.2 Troubleshooting

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Detailed instrument troubleshooting procedures may be found in the manufacturer's manuals. In the event that calibrations or QC sample results are outside acceptance criteria, then the following contingency is followed in the order listed:

1. Prepare new QC samples.
2. Check to ensure the instrument is peaked properly.
3. Recalibrate per Section 6.6.
4. Check to tubing to ensure that all connections are correct and that sample is flowing throughout the system.
5. Check the tension on the pump: flow should be 0.1 to 0.4 mL/minute.
6. Check the autosampler probe and tubing.

9.0 Health and Safety

9.1 As with all samples, gloves and safety glasses should be worn when handling samples, reagents, and standards. It is necessary to wear a lab coat to protect clothing.

9.2 It is important that all necessary safety precautions are followed. Use precautions to prevent spills. If spillage does occur, then clean up all spills immediately and notify supervisor. Strong acids and bases should be added to containers and solutions under the fume hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

9.3 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding MSDS.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptance criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP, recording all data as described in Section 7.1, and reviewing all QC results for compliance with acceptance criteria.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities and DOC performance.

11.0 Waste Management and Pollution Prevention

Unused portions of samples are disposed of only after completion of sample analysis, submission of the final report and contracted sample retention times have been achieved. All conditions of disposal and all records and correspondence concerning the final disposition of the physical sample shall be

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recorded and retained. Records shall indicate the date of disposal, the nature of disposal and the name of the individual who performed the task.

Unused non-hazardous samples are neutralized to pH ~7 SU and then may be disposed of as wastewater. Waste disposal of prepared metals samples and standards are disposed of in designated dilute acids and metals waste container. Minimize reagent use and prepare reagents in amounts that will be used before expiration.

11.1 Chemically Preserved Samples

Chemically preserved samples should have a pH of <2 or >12 SU. The pH alone causes them to express a corrosively hazardous nature that must be neutralized prior to disposal. Once neutralized, samples are no longer viable for testing and are then considered waste. ESI's aqueous waste UV treatment "pit" system is used for discharge. If any samples are hazardous for reasons other than pH, then they must be disposed of according to their specific requirements based on hazard classification. The approximate volume of neutralized waste, date of disposal and initials of person performing the disposal are recorded in a logbook.

11.2 Hazardous Waste Samples

Hazarous waste samples must be handled by properly trained employees. Only the ESI hazardous waste coordinator may train ESI employees on handling hazardous waste. There are 3 main hazardous waste streams at ESI, "dilute metals acidic", "solvents" and "Lachat wastes". When a satellite container needs to be emptied, it is properly transported to the waste shed and added into the appropriately labeled container for that waste stream. Logbooks are kept as a record for volumes added to each drum of any specific waste stream and shipping manifests reflect when they are picked up for transport.

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Table 1. Standard Volume Preparations

Preparation of CAL Std/CCV Standards

Fill 100 mL Class A volumetric flask with ≈ 70mL Milli-Q water and add 10 mL HNO_3 and 5 mL HCl. Add the following to the flask. And then dilute to 100 mL with Milli-Q water. This solution is referred to as the "CCV Intermediate" standard.

100	µL	Cal Std #1	Ultra Scientific ICK-410A or equivalent
25	µL	Cal Std #3	
10	µL	1000 ppm Sb Std	
20	µL	1000 ppm B Std	
20	µL	1000 ppm Mo	
10	µL	1000 ppm Sn	
10	uL	1000ppm Sr	
500	uL	10ppm Hg	
100	µL	1000 ppm Au	

Preparation of ICV Standard

Fill 50 mL Class A volumetric flask with ≈ 35mL Milli-Q water and add 5 mL HNO_3 and 2.5 mL HCl. Add the following to the flask. And then dilute to 50 mL with Milli-Q water. This solution is referred to as the "ICV Intermediate" standard.

100	µL	Cal Std #1	Ultra Scientific ICK-410A or equivalent
25	µL	Cal Std #3	
10	µL	1000 ppm Sb Std	
10	µL	1000 ppm B Std	
10	µL	1000 ppm Mo	
10	uL	1000 ppm Sn	
5	uL	1000 ppm Sr	
125	µL	20 ppm Hg	
50	µL	1000 ppm Au	

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TITLE: Sample Preparation; Trace Metals Digestion of Tissue

Approved By:

QA Officer: _____ Date: _____

Technical Director: _____ Date: _____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	M. Scott	12/2007
1	Reformat and Update	M. Scott	03/2012
2	Addition of substrate requirement for LCS and Blank	K. A. Simon	07/2012
3	Update to digestion procedure	M. Scott	01/2015

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TITLE: Sample Preparation; Trace Metals Digestion of Tissue

1.0 Purpose and Applicability

This SOP describes the method of preliminary digestion to reduce interference by organic material and convert metals associated with particulates that can be determined by inductively-coupled plasma. This procedure uses the least rigorous digestion required to provide complete and consistent recovery compatible with the metal being analyzed.

2.0 Definitions

NA

3.0 Applicable Documents/References

APHA. 1995. Standard Methods for the Examination of Water and Wastewater, 19th Edition. Methods 3030D,3030E, and 3030F.

200.8 Determination of trace elements in waters and wastes by inductively coupled plasma - mass spectrometry 1994.

ESI SOP # 1324 and 1369.

4.0 Materials and Apparatus

4.1 Reagents/Standards

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date.

Milli-Q®

Ultra-Trace Metals Grade Nitric Acid (HNO_3), Concentrated and 50%

Ultra-Trace Metals Grade Hydrochloric Acid (HCl), 50%

Commercially Available Single- or Multi-Element Standards, in both “high” and “low” concentrations, or Ultra-High Purity Compounds

4.2 Equipment and Supplies:

See ESI SOP #1324 for proper cleaning techniques

50 mL digestion tubes
Adjustable Volumetric pipettes
0.2um Filtermate filters

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TITLE: Sample Preparation; Trace Metals Digestion of Tissue

Hot plate
Thermometer calibrated in 70° C - 100° C range
Analytical Balance
Non Contaminating Solid Substrate
Pre cleaned glass beads
Teflon® chips
Ice prepared from high quality Mill-Q® water

5.0 Methods/Procedures

5.1 Tissue Sample Preparation

Mix sample thoroughly and transfer a portion (usually 1g) to a tared tube. Samples have an expiration of 128 days. Record sample wet weight (WW).

Add 5 mL of concentrated HNO_3 and 5 mL of Milli-Q. Add spiking solutions appropriate to program or project-specific data quality objectives.

Place the digestion tube on a hot plate, or in a steam bath, covered with a watch-glass. Heat the sample for ½ hour then add 5 mL of HCl to the samples.

Heat the sample and gently reflux until the volume has gone down to about 4mL. Gentle boiling may take place, however rapid boiling must be avoided.

Cool and dilute to final volume with Milli-Q® water, mix well.

If suspended solids remain, the sample may be filtered using the Filtermate filters

Record the final volume. Final volume is based on the quantitation limit required by the client.

5.2 Blank Preparation

Laboratory blank samples will be prepared in the same manner as routine sediment or tissue samples with the exception that the blank includes a clean substrate. Acceptable substrates may include; pre-cleaned glass beads for a soil/sediment matrix orTeflon® chips for tissue matrix. Ice, prepared from 18 Meg Ohm, or better, Milli-Q® water, may also be used as a solid substrate.

5.3 LCS Preparation

Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) samples will be prepared in the same manner as routine sediment or tissue samples with the exception that the sediment or tissue is replaced with a clean substrate. Acceptable substrates may include; pre-cleaned glass beads for a soil/sediment matrix orTeflon® chips for tissue matrix. Ice, prepared from 18 Meg Ohm, or better, Milli-Q® water, may also be used as a solid substrate.

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6.0 Quality Control Requirements

The objective reporting limits vary from 0.1 to 1.0 ug/g.

6.1 QC Elements

Demonstration of capability (DOC), four replicates of a prepared quality control sample. Required with a change of instrument, method, or analyst.

Method detection limit (MDL) study, seven replicates (standard deviation * 3.14). Performed annually, with change in instrument, or method.

Limit of quantitation (LOQ), a single quality control sample, no more than 1-2x the reporting limit. Performed annually.

Analyze a blank, laboratory control, sample spike, sample spike duplicate, and if desired a laboratory control sample duplicate every analytical batch or twenty samples, whichever is more frequent.

QC elements may be added or removed to meet specific program or project requirements.

6.2 Acceptance Criteria

Demonstration of capability samples must meet LCS acceptance criteria.

The calculated MDL must be lower than the reporting limit.

The limit of quantitation standard acceptance criteria is 70-130%.

Acceptance criteria for all blanks is less than the reporting limit or less than 1/10 of sample value.

Acceptance criteria for LCS/LCSD recovery is 70%-130%. Acceptance criteria for LCSD percent relative range is less than or equal to 30%.

Acceptance criteria for relative percent difference between sample duplicates is less than or equal to 30%.

Acceptance criteria for spiked sample recovery is 70%-130% .

Alternate acceptance criteria may be set for specific programs or projects.

7.0 Calculations/Reporting

7.1 Record

Record the following:

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1. Date
2. Initials of analyst
3. Sample ID
4. Batch
5. Sample Code / Standard Code
6. Acid lot ID
7. Spike ID and amount , with dilution factor if applicable
8. Initial volume
9. Final volume
10. Method
11. Notes, if any

7.2 Calculations

See ESI SOP #1369 Determination of Trace Metals by ICP-MS Analysis.

8.0 Corrective Actions

8.1 Data Qualification

DOC's, MDL's, and LOQ's should be redone until they meet acceptance criteria.
Samples analyzed with a prep blank not meeting acceptance criteria may be reanalyzed or reported with qualification (B).

If the LCS or LCSD's do not meet percent recovery and/or percent relative difference (RPD) acceptance criteria they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (J).

Samples analyzed in duplicate not meeting percent relative difference (RPD) acceptance criteria may be reported with qualification (J) or reanalyzed. If the sample result or sample duplicate result is less than 5 times the reporting limit, the percent relative difference need not be evaluated.

If the spiked sample recovery is not within acceptance criteria samples may be reported with qualification (J) or reanalyzed. If the sample result is greater than 5 times the spike amount the percent recovery need not be calculated.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified:

1. Quality and expiration date of reagents
2. Cleanliness of containers, equipment, and labware
3. Integrity of standards
4. ICP-MS operation

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TITLE: Sample Preparation; Trace Metals Digestion of Tissue

9.0 Health and Safety

Gloves and safety glasses must be worn when handling samples and chemicals. It is necessary to wear a lab coat to protect clothing.

Strong acids and bases should be added to containers and solutions under the hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

Analysts should familiarize themselves with any chemical substances which are named along with their corresponding MSDS sheets.

Use precautions to prevent spills. Clean up all spills immediately and notify supervisor.

10.0 Responsibilities

It is the analyst's responsibility to read and follow this standard operating procedure. Successfully perform a demonstration of capability. Record all data as per section 7.0. Review all QC results for compliance with acceptance criteria.

It is the lab supervisor's responsibility to make sure standard operating procedures are up to date and that acceptance criteria have been set.

11.0 Waste Management and Pollution Prevention

Waste disposal of prepared metals samples and standards are disposed of in designated dilute acids and metals waste containers.

Minimize reagent use. Prepare reagents in amounts that will be used before expiration. Dispose of samples and wastes in accordance with ESI policy, state, and federal regulation.

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TITLE: Mercury in Sediment or Tissue by Cold Vapor Atomic Fluorescence Spectrometry.

Approved By:

QA Officer:_____ Date:_____

Technical Director:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	M. Scott	12/2007
1	Revision	M. Scott	05/2008
2	Reformat and Update	M. Scott	03/2012
3	Update based on audit response	M. Scott	06/2012
4	Addition of substrate requirement for LCS and Blank	K. A. Simon	07/2012
5	Update to sample preparation procedure	M. Scott	01/2015

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TITLE: Mercury in Sediment or Tissue by Cold Vapor Atomic Fluorescence Spectrometry.

1.0 Purpose and Applicability

This method is for the determination of mercury (Hg) in sediment or tissue by cold vapor atomic fluorescence spectrometry.

The samples are processed by digestion in Nitric acid followed by a Hydroxylamine solution just prior to analysis. The mercury levels are determined by running the samples through a PSA mercury fluorescence system and then evaluated against calibration and quality control samples. This method is used with a range of 10ng/L to 150ng/L.

2.0 Definitions

NA

3.0 Applicable Documents/References

Method 245.7 Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry. Revision 2.0 February 2005. EPA-821-R-05-001.

4.0 Materials and Apparatus

4.1 Reagents/Standards

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date.

Milli Q
Air - low in particulate and gaseous mercury
Argon Gas - high purity (99.998%)

Hydrochloric Acid Solution - add concentrated HCl to Milli-Q in a 1:1 ratio. Prepare as needed.

Sulfuric Acid Solution - add 100mL of concentrated sulfuric acid to Milli-Q for a final volume of 4L. Prepare as needed.

Hydroxylamine Solution - Dissolve 2.4g of Hydroxylamine Hydrochloride in 20.0 mL of Milli Q. Prepare weekly.

Stannous Chloride Solution - Dissolve 20.0g of stannous chloride into 2L of the Sulfuric acid solution or dissolve into 2 L of Milli-Q with 20.0 mL of concentrated HCl. Prepare before each analytical session.

Bromate/Bromide Solution - Dissolve .167g of Potassium Bromate and 0.714g of Potassium Bromide in 30.0 mL of Milli-Q. Prepare as needed.

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Mercury Standard, 1.0 mg/L - Add about 50mL of Milli-Q, 0.5mL of the Bromate/Bromide solution, and 0.25mL of the HCl solution to a 100mL volumetric flask. Add .100mL of the stock mercury standard and dilute to 100mL, transfer to a glass bottle. Stable until expiration date given for stock mercury standard.

Check Mercury Standard, 1.0 mg/L - Add about 50 mL of Milli-Q, 0.5mL of the Bromate/Bromide solution, and 0.25mL of the HCl solution to a 100 mL volumetric flask. Add 0.100 mL of the secondary mercury standard and dilute to 100 mL, transfer to a glass bottle. Stable until expiration date given for stock mercury standard.

Working Mercury Standard, 2000 ng/L - Dilute 0.2mL of the Mercury standard to 100 mL in a volumetric flask with Milli Q. This solution should be made new with each use.

ICV Mercury Standard - Dilute .2mL of the Check Mercury standard to 100mL in a volumetric flask with Milli Q. This solution should be made new with each use.

4.2 Equipment and Supplies:

CVAFS System: PSA Merlin
Software: Avalon P.S. Analytical Version 2.28
40mL glass vials
Adjustable pipettes
Volumetric Flasks
Graduated cylinders
Analytical balance, capable of weighing to 0.0001 g (1 mg)
Stir Bar, magnetic stir plate, and weigh boats
Computer & printer
Vial rack
Solid Substrate
- Pre cleaned glass beads
- Teflon® chips
- Ice prepared from high quality Mill-Q® water

5.0 Methods/Procedures

Sediment and tissue samples should be collected in a glass jar. Sediment samples should be stored at 4°C and tissue samples should remain frozen until they are to be analyzed. Samples have a holding time of 128 days.

5.1 Sample Preparation of Sediment or Tissue

Mix sample thoroughly and transfer a portion (usually 1g) to a tared tube. Samples have an expiration of 128 days. Record sample wet weight (WW).

Add 5 mL of concentrated HNO_3 and 5 mL of Milli-Q. Add spiking solutions appropriate to program or project-specific data quality objectives.

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Place the digestion tube on a hot plate, or in a steam bath, covered with a watch-glass. Heat the sample for ½ hour then add 5 mL of HCl to the samples.

Heat the sample and gently reflux until the volume has gone down to about 4mL. Gentle boiling may take place, however rapid boiling must be avoided.

Cool and dilute to final volume with Milli-Q® water, mix well.

If suspended solids remain, the sample may be filtered using the Filtermate filters

Record the final volume.

5.2 Preparation of Digest for Analysis

Samples are prepared by first treating empty 40mL vials with 2.25mL of HCl Solution and 0.45mL of the Bromate/Bromide solution (a dark yellow color should result). Then 20mL of each sample digest (digest may be pre-diluted to a final volume of 20mL) is transferred into a treated 40mL vial. The sample should be shaken and allow to digest for at least 30 minutes. If the yellow color does not remain more Bromate/Bromide should be added until a permanent yellow color is obtained.

After the samples are allowed to digest the caps are removed and 25uL of the Hydroxylamine solution is added to each vial, recapped swirled occasionally over a 5 minute period. The addition of the hydroxylamine should cause the yellow color to disappear. The hydroxylamine should be added at a time the coincides with the start of sample analysis.

5.3 Calibration Preparation

The curve, ICV, ICB, CCV, and CCB are prepared at the same time and in the same manner as the samples. The curve points are 10, 25,50,100,150ng/L. The ICV and CCV are prepared at 25ng/L. The CCB and ICB consist of 20mL of Milli-Q added to the pretreated vials. The following amounts of the Working Mercury Standard are brought to a final volume of 20mL in pill cups before being added to the pretreated 40mL vials.

Concentration (ng/L)	Volume added (mL)	Std. Source
10	.100	Working Hg Std.
25	.250	Working Hg Std.
50	.500	Working Hg Std.
100	1.00	Working Hg Std.
150	1.50	Working Hg Std.
CCV25	.250	Working Hg Std.
ICV25	.250	ICV Hg Std.

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The curve, ICV, ICB, CCV, and CCB are also allowed to digest for at least 30 minutes and then have 25uL of hydroxylamine solution added to them just prior to analysis.

5.4 Blank Preparation

Laboratory blank samples will be prepared in the same manner as routine sediment or tissue samples with the exception that the blank includes a clean substrate. Acceptable substrates may include; pre-cleaned glass beads for a soil/sediment matrix orTeflon® chips for tissue matrix. Ice, prepared from 18 Meg Ohm, or better, Milli-Q® water, may also be used as a solid substrate.

5.5 LCS Preparation

Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) samples will be prepared in the same manner as routine sediment or tissue samples with the exception that the sediment or tissue is replaced with a clean substrate. Acceptable substrates may include; pre-cleaned glass beads for a soil/sediment matrix orTeflon® chips for tissue matrix. Ice, prepared from 18 Meg Ohm, or better, Milli-Q® water, may also be used as a solid substrate.

5.6 Instrument Operation

Turn on the CVAFS system, air, and argon. Set the Argon to 400 cc/mn, the air to 2.5 L/mn, and the flow valve to .8 L/mn. Allow the instrument to warm up for 20 minutes with Milli-Q running through the reagent and sample lines. When ready to begin analysis place the reagent line in the Stannous Chloride solution and the sample line into the Sulfuric Acid solution and allow to flow through.

Analyze an instrument blank to verify no contamination is present, then run the curve, ICV, ICB. If the curve and initial calibration standards are acceptable proceed with batch analysis with CCV's and CCB's being run every 10 samples. The sample line is to be in the sample when the program is during its "rise" and "analysis" cycles and in the sulfuric acid solution at all other times.

When ready to shut down allow water to flush through the lines for at least 10 minutes. Then shut off the argon, air, and CVAFS system.

6.0 Quality Control Requirements

The objective reporting limit is 0.02 ug/g. The most recent MDL study can be found in the metals quality control directory.

6.1 QC Elements

Demonstration of capability (DOC), four replicates of a prepared quality control sample. Required with a change of instrument, method, or analyst.

Method detection limit (MDL) study, seven replicates (standard deviation * 3.14). Performed annually, with change in instrument, or method.

Limit of quantitation (LOQ), a single quality control sample, no more than 1-2x the reporting limit.

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Performed annually.

Analyze a blank, laboratory control, sample spike, sample spike duplicate, and if desired a laboratory control sample duplicate every analytical batch or twenty samples, whichever is more frequent.

Initial calibration consisting of a blank and five standards.

Initial calibration verification (ICV) and initial calibration blank (ICB) are performed following analysis of initial calibration standards.

Continuing calibration verification (CCV) and continuing calibration blank (CCB) are performed every ten samples and at the end of an analytical session.

QC elements may be added or removed to meet specific program or project requirements.

6.2 Acceptance Criteria

Demonstration of capability samples must meet LCS acceptance criteria.

The calculated MDL must be lower than the reporting limit.

The limit of quantitation standard acceptance criteria is 50-150%.

Acceptance criteria for initial calibration is a relative standard deviation for response factors less than 15% or a correlation coefficient of greater than 0.995.

Acceptance criteria for all blanks is < 0.02 ug/g or less than 1/10 of sample value.

Acceptance criteria for the initial calibration verification standard (ICV) must be ±15% of the target value.

Acceptance criteria for the continuing calibration verification standard (CCV) is must be within 75%-125% of the target value.

Recovery of the LCS/LCSD pair must be within 75%-125% of the target value. Additionally, the relative percent difference (RPD) between the LCS and LCSD must not exceed 25%.

Acceptance criteria for relative percent difference between sample spike and sample spike duplicates is less than or equal to 25%.

Recovery of the spiking compound must be within 75%-125% of the target value.

Alternate acceptance criteria may be set for specific programs or projects.

6.3 Interferences

Gold, silver, and iodine are known interferences. The bromate/bromide digestion reagent overcomes many chloride, sulfide, and molecular absorption interferences.

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7.0 Calculations/Reporting

7.1 Record

Record the following:

1. Date
2. Analysts initial
3. Batch
4. Sample ID / Standard ID, with dilution factor if applicable
5. Sample Code / Standard Code
6. Signal
7. Amount/ID of reagent
8. Amount/ID of standard
9. Sample volume
10. Notes, if any

7.2 Calculation

Use the signal datum provided by the CVAFS system to create an initial calibration by applying either the average response factor or quadratic fit. Use the signal datum to determine the mercury concentration for each sample and QC sample utilizing the initial calibration.

Calculate LCS/ICV/CCV Recovery as follows:

$$\frac{\text{LCS/ICV/CCV concentration}}{\text{Concentration added}} \times 100 = \%\ \text{recovery}$$

Calculate %RPD as follows:

$$\frac{\text{Abs(Result 1 - Result 2)}}{\text{Avg. of results}} \times 100 = \%\text{RPD}$$

Calculate Spike Recovery as follows:

$$\frac{\text{Spiked sample concentration - Sample concentration}}{\text{Concentration added}} \times 100 = \%\ \text{recovery}$$

8.0 Corrective Actions

8.1 Data Qualification

DOC's, MDL's, and LOQ's should be redone until they meet acceptance criteria.

If the initial calibration curve is not within acceptance criteria, recalibrate or qualify (J) any positive sample values as estimated.

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If the ICV does not meet acceptance criteria prepare a new curve and ICV or qualify (J) any positive sample values as estimated.

Samples analyzed with blanks (prep, initial, continuing) not meeting acceptance criteria may be reanalyzed or reported with qualification (B).

If the LCS or LCSD's do not meet percent recovery and/or percent relative difference (RPD) acceptance criteria they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (J).

If the spiked sample and spiked sample duplicate do not meet the percent relative difference (RPD) acceptance criteria the result may be reported with qualification (J) or reanalyzed.

If the spiked sample or spiked sample duplicate recovery is not within acceptance criteria samples may be reported with qualification (J) or reanalyzed. If the sample result is greater than 5 times the spike amount the percent recovery need not be calculated.

If a CCV does not meet acceptance criteria all associated samples must be reanalyzed or qualified (J) as estimated.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified:

1. Quality and expiration date of reagents
2. Cleanliness of containers, equipment, and labware
3. Integrity of standards
4. Condition of tubing
5. Air and argon flow

9.0 Health and Safety

Mercury is toxic.

Gloves and safety glasses must be worn when handling samples and chemicals. It is necessary to wear a lab coat to protect clothing.

Strong acids and bases should be added to containers and solutions under the hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

Analysts should familiarize themselves with any chemical substances which are named along with their corresponding MSDS sheets.

Use precautions to prevent spills. Clean up all spills immediately and notify supervisor.

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10.0 Responsibilities

It is the analyst's responsibility to read and follow this standard operating procedure. Successfully perform a demonstration of capability. Record all data as per section 7.0. Review all QC results for compliance with acceptance criteria.

It is the lab supervisor's responsibility to make sure standard operating procedures are up to date and that acceptance criteria have been set.

11.0 Waste Management and Pollution Prevention

Waste disposal of prepared mercury samples, reagents, and standards are disposed of in designated dilute acids and metals waste containers.

Minimize reagent use. Prepare reagents in amounts that will be used before expiration. Dispose of samples and wastes in accordance with ESI policy, state, and federal regulation.

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Title: Tissue Extraction for Organic Compounds

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	K. Simon	06/2009
1	Format Modification and Update	K. Simon	02/2010
2	Review and Update	M. Scott	08/2010
3	Renamed and Updated	M. Scott	01/2013
4	Update based on annual review	M. Scott	04/2014
5	Update to procedure	M. Scott	03/2015
6	Update to procedure based on DoD audit	E. Penta	06/2016

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Title: Tissue Extraction for Organic Compounds

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the procedure for extracting tissue samples for organic compounds using elements of US EPA method 3570. The procedure for florisil column cleanup uses US EPA method 3620 and the procedure for silica gel column cleanup uses US EPA method 3630. The procedure for sulfur cleanup uses US EPA method 3660B and the procedure for sulfuric acid cleanup uses US EPA method 3665A.

Samples are spiked with an appropriate surrogate solution and extracted with solvent in glass. The solvent extracts are collected and adjusted to the appropriate final volume, typically 0.2 to 2.0 mL, by ebullation on a warming bath, such as Mayer N-Evap, or using a K-D apparatus. The extract is stored in glass and refrigerated until GC or GC/MS analysis. Samples may require additional clean-up.

Cleanup options may include but are not limited to:

Pesticides (PEST): Florisil column chromatographic cleanup (see section 5.2).
Polynuclear Aromatic Hydrocarbons (PAH): Silica gel column chromatographic cleanup (see section 5.3).
PCB Congeners (CGR): Treatment with concentrated sulfuric acid (see section 5.4).

2.0 Definitions

See ESI's Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Method 3510C (SW-846). (This method should be consulted for interferences and pollution prevention.)

US EPA. 2002. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Method 3570. (SW-846).

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Methods 3620C and 3630C (SW-846).

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Methods 3660B (SW-846).

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Methods 3665A (SW-846).

US EPA. 1983. *Methods for Chemical Analysis of Water and Wastes.* Method 608.

US EPA. 1988. *Statement of Work for Organic Analysis, Multi-Media Concentration.* Pesticide D-11.

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US EPA. 1985. *Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry.* Method 680.

Agilent Technologies. 1999. *Hardware Manual.* 5973 Network Mass Selective Detector.

Agilent Technologies. 1997-2000. *Operation Guide.* Agilent 7683 Automatic Liquid Sampler.

Agilent Technologies. 2000. *Operating Manual.* Agilent 6890 Series Gas Chromatograph.

Department of Defense (DoD), Department of Energy (DoE). 2013. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.0.

Agilent Technologies. 1989-2001. *G1701DA Version D.00.00.38 19-Nov 2001.* Environmental ChemStation.

4.0 Materials and Apparatus

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date. Note that standards may also be prepared according to project specific analyte list.

4.1 Reagents and Standards

Acetone, pesticide grade
Cyclohexane, pesticide grade
Florisil, activated at 675°C for 4 hours
Hexane, pesticide grade
Methylene Chloride, pesticide grade
Silica Gel, use as received or activate at 130°C for 16 hours
Sodium Sulfate, heated at 400°C for 4 hours to remove phthalates and other interfering organic substances.
Pesticide Surrogate and Spike standards, provided by Supelco or other acceptable vendors. See Appendix A Part 1 for standard preparation instructions.
Congener Surrogate and Spike standards, provided by Ultra or other acceptable vendors. See Appendix A Part 2 for standard preparation instructions.
PAH Surrogate and Spike standards, provided by Environmental Consulting and Supplies or other acceptable vendors. See Appendix A Part 3 for standard preparation instructions.

4.2 Equipment and Supplies

40 mL Glass screw top vial
50 mL Round bottom glass vials
2 mL Glass extract collection vials, with Teflon lined lids
Analytical balance, capable of weighing to 0.01 g
Evaporation containers (e.g. centrifuge test tubes)
Fume hood

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Gas-tight volumetric syringes
Glass chromatography columns
Glass funnels
Kuderna-Danish apparatus
Micro-Snyder column
Mayer N-Evap Analytical Evaporator
Mortar and pestle
Silanized glass wool
Water/Steam bath, for K-D solvent evaporation
Zymark TurboVap Evaporator
Kimwipe

5.0 Methods and Procedures

Samples should be stored in glass jars with Teflon lined caps. Tissue samples must be frozen until extracted. It is recommended that extractions be performed as soon as possible, but should be done within 180 days.

5.1 Extraction Procedure

Ensure all glassware is clean and dry and thoroughly solvent rinse all glassware, mortar and pestle with the appropriate solvent before proceeding with extractions.

5.1.1 Measure and add approximately 2 to 10 g (depending on project requirements) of homogenized sample into mortar. Use sodium sulfate to grind mixture until sample turns to a fine, dry, free-flowing powder. Start with approximately 1 g of sodium sulfate and add more as needed. Transfer the sample from the mortar into a labeled 40 mL vial with a Teflon lined cap.

Note that this step deviates from the order of procedures in Method 3570. Method 3570 requires the addition of sodium sulfate to the extraction vessel before the sample and does not require the sodium sulfate and sample to be mixed in a mortar prior to being transferred into the extraction vessel.

5.1.2 Unless otherwise specified, 5 g sodium sulfate will serve as the laboratory blank, Laboratory Control Sample (LCS) and the Laboratory Control Sample Duplicate (LCSD).

5.1.3 Using a gas-tight volumetric syringe, carefully add the appropriate amount of surrogate solution to each sample, including the blank and LCS/LCSD. Add the appropriate amount of spiking solution to the LCS/LCSD (and to the Matrix Spike (MS) and Matrix Spike Duplicate (MSD) samples when available). See Appendix A for the appropriate spike and spike volumes used for PAH, CGR, and Pesticide extractions. Other spikes and spike volumes may be used to meet project specific requirements.

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Note that the amount of surrogate and spike compounds added to the soil deviates from Method 3570 specification of 50 µg and not all standards are made in methylene chloride. See Appendix A for standard concentrations and recipes.

5.1.4 Add approximately 10 to 15 mL or more of solvent (see Appendix A for appropriate extraction solvent) to all sample vials and shake each vial for 2 minutes. If required, samples may be rotated end over end for at least 4 hours for the first extraction. Let settle for 10 minutes and if necessary, vials may be centrifuged for 15 minutes at 500 rpm.

Note that the volume and type of solvent used for extraction deviates from Method 3570 specification of 12 mL of methylene chloride.

5.1.5 Decant extract into a glass vial for concentration. If necessary, extract can be filtered through a glass funnel containing sodium sulfate with glass wool plug or filter paper into a glass vessel. Sodium sulfate must be pre-rinsed and wetted with appropriate solvent prior to use. Once sample has been filtered, rinse sodium sulfate with appropriate solvent immediately, ensuring the sodium sulfate does not go dry. Before recapping vial, ensure the vial lip and threads are free from debris and wipe with a Kimwipe as necessary.

5.1.6. Repeat section 5.1.4 and 5.1.5 twice more. Glass vessel should be capped while not actively being added to. After final extraction, rinse sodium sulfate with 2-3mL of extraction solvent to ensure complete transfer.

5.1.7. Combine all three extracts and clean up as necessary (see Sections 5.2 through 5.5). Adjust to the appropriate final volume (typically 0.2 mL to 2.0 mL) by ebullation on a warming bath, such as the Mayer N-Evap or K-D apparatus.

If K-D is used, add Teflon boiling stick or other appropriate material to the concentration vessel and attach micro-Snyder column Pre-wet Snyder column with 0.5 mL of extraction solvent. Place in a water bath maintained at a constant temperature, approximately 60-65°C. Temperature and position of K-D apparatus should be adjusted so the solvent boils evenly, the Snyder column balls chatter and the columns do not flood. Concentrate extract until about 1 mL is remaining.

5.1.8 Let the final extract cool (and drain if K-D was used) then transfer to a screw cap Teflon lined septum glass vial for analysis. Rinse the evaporation container with solvent for complete transfer, and prepare the exact final volume with a gas tight syringe. See Appendix A for appropriate final volumes. If the sample residue prevents concentrating to the desired volume, then adjust the final volume upward with solvent and record to the nearest 0.01 mL.

5.1.9 Extract condition and preparation data are verified by the laboratory supervisor and then transferred to the organics extract refrigerator maintained at 4±2°C until time of analysis.

5.2 Florisil Column Cleanup

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Florisil columns may be useful in cleaning up pesticide extracts. Following the Florisil column cleanup, proceed to finalize the extract volume and prepare for analysis (see steps 5.1.7 through 5.1.9).

5.2.1 Prepare a Florisil column for each extract using a 10 mL glass chromatography column fitted with silanized glass wool and a sodium sulfate plug at the bottom. Fill the column with 6 to 7 cc of Florisil and tap the column gently to settle the powder. Insert a sodium sulfate plug into the top of the column.

5.2.2 Secure the column on a pipette stand in a fume hood. Pre-clean the column with approximately 25 mL of $MeCl_2$ and discard to waste. Pre-elute the column with approximately 25 mL of hexane and allow to drain into a waste container to be discarded. Keep the column wet with solvent above the sodium sulfate.

5.2.3 Transfer all or a known portion of the sample extract to the top of the Florisil column using a gas tight syringe. Record the amount of sample (mL) taken for the cleanup. Add sufficient hexane to the sample vial to rinse it, then transfer rinse to the column. Elute the column with 50 mL of $MeCl_2$. Collect the eluate in a clean, labeled, solvent rinsed evaporation container. Extracts must be exchanged into hexane before the extract is prepared for analysis.

5.3 Silica Gel Column Cleanup

Silica gel columns may be useful for PAH extracts. Following the silica gel column cleanup, proceed to finalize the extract volume and prepare for analysis (see steps 5.1.7 through 5.1.9).

5.3.1 Prepare a silica gel column for each extract using a 10 mL glass chromatography column fitted with silanized glass wool and a sodium sulfate plug at the bottom. Fill the column with 6 to 7 cc of silica gel and tap the column gently to settle the powder. Insert a sodium sulfate plug into the top of the column.

5.3.2 Secure the column on a pipette stand in a solvent delivery system in a fume hood. Pre- clean the column with approximately 25 mL of $MeCl_2$ and discard to waste. Pre-eulte the column with 25 mL of pentane into a waste container to be discarded. Keep the column wet with solvent above the sodium sulfate.

5.3.3 Transfer all or a known portion of the sample extract to the top of the silica gel column using a gas tight syringe. Extracts must be exchanged into hexane before being transferred to the column. Record the amount of sample (mL) taken for cleanup. Add sufficient hexane to the sample vial to rinse, then transfer the rinse to the column. Elute the column with 50 mL of 1+1 $MeCl_2$/pentane. Collect the eluate in a clean, labeled, solvent rinsed evaporation container.

5.4 Sulfuric Acid Cleanup

Sulfuric acid is used to clean up CGR extracts. Any column cleanup should occur before the acid cleanup. Extracts must be exchanged into hexane before adding any acid. Add approximately 0.5 mL of concentrated sulfuric acid to the extract, cap and shake. Allow the

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extract to settle for at least one hour, where the extract will separate into two distinct layers. Carefully pull off the top layer (the organic layer) using a gas tight syringe. Transfer to clean vial for analysis. Be very careful not to get any acid in the extract for analysis. Record the extract volume recovered.

5.5 Copper Cleanup

Copper is used to remove sulfur from sample extracts. The copper must be very reactive prior to use. All oxides must be removed so the copper is shiny and bright. If the copper does not look this way, then it needs to be reactivated by rinsing with dilute HNO_3 acid, Milli-Q® water, and then acetone. Approximately 2 g of cleaned copper is added to the sample extract and agitated for a minimum of one minute. Copper should be added to extract until there is no sulfur left to react with the copper (copper will stop turning black once all sulfur has been removed). Once this has been achieved, the extract should be removed from the copper and transferred to a clean vial for analysis.

6.0 Quality Control Requirements

Quality control elements may be added, removed, or altered to meet specific program or project requirements. Alternate acceptance criteria may be set for specific programs or projects, and reporting limits are specific to each parameter and client protocols. For DoD-specific projects see the QSM 5.0, as appropriate.

Note that all QC elements and samples must be spiked with a surrogate standard.

6.1 Demonstrations of Capability (DOC)

An analyst must complete a DOC before working independently on this procedure and yearly thereafter. A DOC is also required with any changes to instrument, method, or analyst. To perform a DOC, four replicates of a prepared quality control sample must be prepared. DOC samples must meet LCS acceptance criteria. For more information on DOCs see SOP QA-1108 “Training”.

6.2 Method Detection Limit (MDL)

The MDL is calculated using the appropriate student’s t-parameter value multiplied by the standard deviation of 7 or more replicates of spiked samples or standards. MDLs are performed annually and with changes in instrument or method. The calculated MDL must be lower than the reporting limit.

The t-parameter value is from a one-tailed Student’s t-Test at the 99% confidence interval using n-1 degrees of freedom, with “n” being the number of replicates.

The most recent MDL study can be found in the inorganics quality control directory on the ESI network. For more information on MDLs see SOP QA-1111 “Determination of Method Detection and Reporting Limits”.

6.3 Limit of Detection (LOD)

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LOD determination will be conducted on a quarterly basis. For a single-analyte standard, the LOD is a single quality control sample that is approximately 2 to 3 times the MDL but no greater than the reporting limit. For a multi-analyte standard, the LOD is a single quality control sample that is approximately 1 to 4 times the MDL but no greater than the reporting limit.

The apparent signal to noise ratio at the LOD must be at least 3 or for methods that do not produce a signal to noise the LOD result must be at least 3 standard deviations greater than the mean method blank.

6.4 Limit of Quantitation (LOQ)

LOQ confirmation will be conducted on a quarterly basis. The LOQ is a single quality control sample no more than 1 to 2 times the reporting limit. The LOQ standard acceptance criteria is 30-150%. For more information on LODs and LOQs see SOP QA-1147 “Determination of LOD and LOQ Quality Control Samples”.

6.5 Blank, LCS/LCSD, MS/MSD, Sample Duplicate, Relative Percent Difference (RPD)

6.5.1 A blank, LCS/LCSD, MS/MSD and sample duplicate will be analyzed every batch or every 20 samples, whichever is more frequent.

6.5.2 Acceptance criteria for all blanks is less than the reporting limit or less than 1/10th of sample value.

6.5.3 Acceptance criteria for LCS/LCSD recovery is 30-150%. Acceptance criteria for LCSD RPD is less than or equal to 30%.

6.5.4 Acceptance criteria for MS/MSD recovery is 30-150%.

6.5.5 Acceptance criteria for RPD between sample duplicates is ≤30%.

6.5.6 Advisory limits for surrogate standard recovery is 30-150%.

6.6 Interferences

Solvents, standards, and labware may be contaminated with artifacts that would affect results. Phthalate esters can pose a contamination issue with all analytes not just phthalate esters themselves.

7.0 Calculations and Reporting

7.1 The following information should be recorded when performing a tissue extraction for organic compounds:

- Date
- Analyst’s initial
- Batch

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- Sample ID, with dilution factor if applicable
- Sample code / standard code
- Solvent lot ID
- Surrogate ID and amount, with dilution factor (if applicable)
- Spike ID and amount, with dilution factor (if applicable)
- Initial weight of wet sample
- Final volume
- Volume taken for cleanup (if applicable)
- Volume of cleaned sample
- Method
- Balance identification number
- Filter paper lot number (if applicable)
- Silica gel, florisil, and glass wool lot numbers (if applicable)
- Notes, if any

7.2 Calculations

Calibrate the system software as per the Agilent manuals referenced in Section 3.0. When reasonable, use the system software to calculate the values for QC elements and samples. Review the data to ensure it is correct and that all quality control requirements have been met. When needed, manually integrate the values according to ESI SOP QA-1132 “Manual Chromatographic Peak Integration”.

7.2.1 Calculate LCS/Surrogate Recovery as follows:

$$\frac{\text{LCS/SUR Concentration}}{\text{Concentration added}} \times 100 = \%\ \text{Recovery}$$

7.2.2 Calculate %RPD as follows:

$$\frac{\text{Abs(Result 1 - Result 2)}}{\text{Average of results}} \times 100 = \%\text{RPD}$$

7.2.3 Calculate Spike Recovery as follows:

$$\frac{\text{Spiked Sample Concentration - Sample Concentration}}{\text{Concentration added}} \times 100 = \%\ \text{Recovery}$$

8.0Corrective Actions

8.1 Data Qualification

8.1.1 If DOCs, MDLs, LODs, or LOQs do not meet acceptability criteria, then repeat until acceptability criteria is met.

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8.1.2 If the LCS or LCSD does not meet percent recovery and/or RPD acceptance criteria, then they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (using the "J" qualifier).

8.1.3 If samples analyzed with blanks are not meeting acceptance criteria, then they may be reanalyzed or reported with qualification (using the "B" qualifier).

8.1.4 If samples analyzed in duplicate do not meet RPD acceptance criteria, then they may be reanalyzed or reported with qualification (using the "J" qualifier). If the sample result or sample duplicate result is less than 5 times the reporting limit, then the RPD does not need to be evaluated.

8.1.5 If the MS recovery is not within acceptance criteria, then samples may be reanalyzed or reported with qualification (using the "J" qualifier). If the sample result is greater than 5 times the spike amount, then the percent recovery need not be calculated.

8.1.6 If the surrogate recovery is not within the acceptable range, then the sample is re-extracted whenever possible. Sample matrix effect will be proven if the results obtained from the re-extracted sample show similar results as the first recovery. When this occurs, both results are reported with an explanation. If the results of the re-extracted sample are in the acceptable range, then there is a problem with the first extraction and only the compliant results are reported. If no additional sample can be obtained, then results are flagged in the report and a narrative is included qualifying the data.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified.

- Quality and expiration date of reagents
- Cleanliness of containers, equipment, and labware
- Integrity of standards
- GC and/or GC/MS operation

9.0 Health and Safety

9.1 As with all samples, gloves and safety glasses should be worn when handling samples, reagents, and standards. It is necessary to wear a lab coat to protect clothing. All solvent usage must occur in properly working fume hoods.

9.2 Methylene chloride, pentane, and cyclohexane are extremely hazardous to everyone in the laboratory if handled improperly. It is important that all necessary safety precautions are followed.

9.3 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding MSDS.

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10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptance criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP, recording all data as described in Section 7.1, and reviewing all QC results for compliance with acceptance criteria.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities and DOC performance.

11.0 Waste Management and Pollution Prevention

Unused non-hazardous tissue samples may be disposed of as general trash. Solvents not evaporated in a hood are collected and disposed of as hazardous waste in designated solvent water waste containers. Extracted tissue, once dried should not be hazardous but may be disposed of as hazardous waste. Extracts and standard residues are deemed hazardous and are disposed of as such. Minimize solvent use. Prepare standards in amounts that will be used before expiration.

11.1 Solid Materials

Waste storage, transfer and disposal will be in compliance with NH DES and US EPA waste disposal regulations. Non-hazardous solid materials, sediments and soils will be disposed of through an appropriate waste disposal site. All non-hazardous solid waste will be drained of as much free liquid as possible. The remaining sample is removed from its original container and placed with other non-hazardous waste samples in a 55 gallon metal drum. Once full, these containers will then be transferred to an approved waste disposal facility. This transfer is recorded on a waste manifest and they are retained for a minimum of 3 years.

11.2 Hazardous Waste Samples

Hazardous waste samples must be handled by properly trained employees. Only the ESI hazardous waste coordinator may train ESI employees on handling hazardous waste. There are 3 main hazardous waste streams at ESI, "dilute metals acidic", "solvents" and "Lachat wastes". When a satellite container needs to be emptied, it is properly transported to the waste shed and added into the appropriately labeled container for that waste stream. Logbooks are kept as a record for volumes added to each drum of any specific waste stream and shipping manifests reflect when they are picked up for transport.

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Title: Tissue Extraction for Organic Compounds

Appendix A

Pesticides			Congener			PAH		
Initial Volume: 4 g			Initial Volume: 8 g			Initial Volume: 8 g		
Final Volume: 0.4 mL			Final Volume: 0.8 mL			Final Volume: 0.8 mL		
Solvent: Hexane			Solvent: $MeCl_2$			Solvent: $MeCl_2$		
Surrogate - 50 µL			Surrogate - 80 µL			Surrogate - 80 µL		
Tetrachloro-m-xylene	0.5	µg/mL	198	2	µg/mL	o-terphenyl	10	µg/mL
Decachlorobiphenyl	0.5	µg/mL				2-fluorobiphenyl	10	µg/mL
			Spike - 40 µL					
Pesticide A Mix - 125 µL			8	4	µg/mL	Spike - 80 µL		
TCMX (SS)	0.08	µg/mL	18	4	µg/mL	naphthalene	10	µg/mL
alpha-BHC	0.08	µg/mL	28	4	µg/mL	acenaphthylene	10	µg/mL
gamma-BHC (Lindane)	0.08	µg/mL	44	4	µg/mL	acenaphthene	10	µg/mL
heptachlor	0.08	µg/mL	52	4	µg/mL	fluorene	10	µg/mL
endosulfan I	0.08	µg/mL	66	4	µg/mL	phenanthrene	10	µg/mL
dieldrin	0.16	µg/mL	77	4	µg/mL	anthracene	10	µg/mL
endrin	0.16	µg/mL	101	4	µg/mL	fluoranthene	10	µg/mL
4,4'-DDD	0.16	µg/mL	105	4	µg/mL	pyrene	10	µg/mL
4,4'-DDT	0.16	µg/mL	118	4	µg/mL	benzo[a]anthracene	10	µg/mL
methoxychlor	0.8	µg/mL	126	4	µg/mL	chrysene	10	µg/mL
209 (SS)	0.16	µg/mL	128	4	µg/mL	benzo[b]fluoranthene	10	µg/mL
			138	4	µg/mL	benzo[k]fluoranthene	10	µg/mL
Pesticide B Mix - 125 µL			153	4	µg/mL	benzo[a]pyrene	10	µg/mL
TCMX (SS)	0.08	µg/mL	170	4	µg/mL	indeno[1,2,3-cd]pyrene	10	µg/mL
beta-BHC	0.08	µg/mL	180	4	µg/mL	dibenz[a,h]anthracene	10	µg/mL
delta-BHC	0.08	µg/mL	187	4	µg/mL	benzo[g,h,i]perylene	10	µg/mL
aldrin	0.08	µg/mL	195	4	µg/mL			
heptachlor epoxide	0.08	µg/mL	206	4	µg/mL			
gamma-chlordane	0.08	µg/mL	209	4	µg/mL			
alpha-chlordane	0.08	µg/mL	49	4	µg/mL			
4,4'-DDE	0.16	µg/mL	183	4	µg/mL			
endosulfan II	0.16	µg/mL	87	4	µg/mL			
endrin aldehyde	0.16	µg/mL	184	4	µg/mL			
endosulfan sulfate	0.16	µg/mL						
endrin ketone	0.16	µg/mL						
209 (SS)	0.16	µg/mL						
Pesticide C Mix - 125 µL								
oxychlordane	0.08	µg/mL						
chlorpyrifos	0.08	µg/mL						
trans-nonachlor	0.08	µg/mL						
cis-nonachlor	0.08	µg/mL						
hexachlorobenzene	0.08	µg/mL						

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Part 1 (See above for compounds in each standard)

Pesticide Surrogate Mix - 0.5 µg/mL
Stock Standard: Supelco Cat# 861284 200 µg/mL
Dilute 0.075 mL stock standard to final volume 30 mL with hexane

Pesticide Mix A - 80/160 ng/mL
Stock Standard: Supelco Cat# 47977 0.5-5.0 µg/mL
Dilute 1.0 mL stock standard to final volume 6.25 mL with hexane

Pesticide Mix B - 80/160 ng/mL
Stock Standard: Supelco Cat# 47977 0.5-1.0 µg/mL
Dilute 1.0 mL stock standard to final volume 6.25 mL with hexane

Pesticide Mix C - 80 ng/mL
Stock Standard: Individual component (see list in Appendix A) at 100 µg/mL from Crescent Chemical Company
Dilute 0.2 mL each stock standard to final volume 2.0 mL with hexane

Part 2 (See above for compounds in each standard)

Congener Surrogate - 2 µg/mL
Stock Standard: Ultra Cat# RPC-075AS 100 µg/mL
Dilute 0.6 mL stock standard to final volume 30 mL with 50:50 hexane:acetone

Congener Spike - 4 µg/mL
Stock Standards: Ultra Scientific Cat# RPC-EPA-2-1, RPC-030AS, RPC-073AS, RPC-099AS, RPC-075AS and RPC-168AS 100 µg/mL
Dilute 0.15 mL of each stock standard to final volume 3.75 mL with hexane

Part 3 (See above for compounds in each standard)

PAH Surrogates - 10 µg/mL
Stock Standards: 2-Fluorobiphenyl Chemservice Cat #N-10359-16 Neat
O-terphenyl Chemservice Cat# S-12693X4-1ML 1000 µg/mL
Intermediate Stock Standards: 2-Fluorobiphenyl - 50 mg stock standard to final volume 25 mLwith MeCl2 2000 µg/mL
O-terphenyl 1.0 mL stock standard to final volume 10 mL with acetone 200 µg/mL
Second Intermediate Stock Standard: 2-Fluorobiphenyl - Dilute 1 mL intermediate standard to final volume 10 mL with acetone 200 µg/mL
2-Fluorobiphenyl: Dilute 2.0 mL second intermediate standard to final volume 40 mL with acetone 10 µg/mL
O-terphenyl: Dilute intermediate standard to final volume 40 mL with acetone 10 µg/mL

PAH Spike Mix - 10 µg/mL
Stock Standard: Environmental Consulting and Supplies, Inc Cat# ECS-A-032, ECS-B-032, ECS-N-032 2000 µg/mL
Intermediate Stock Standard: Dilute 2 mL of stock standard to final volume 40 mL acetone 100 µg/mL
Dilute 1.0 mL of intermediate stock standard to final volume 10 mL acetone

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TITLE: Total Organic Carbon in Sediment by Electrothermal Combustion

Approved By:

QA Officer:_____ Date:_____

Technical Director:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	R.Foster	06/2008
1	Annual Review and Update	R.Foster	05/2009
2	Update and Review	M. Scott	01/2015

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TITLE: Total Organic Carbon in Sediment by Electrothermal Combustion

1.0 Purpose and Applicability

This method describes the determination of Total Organic Carbon in sediment and solid samples.

Organic carbon is oxidized to carbon dioxide electrothermally in a stream of oxygen using a Phoenix 8000 with a Model 183 boat accessory. Sediment samples are weighed into platinum or quartz boats and introduced manually into the electrothermal furnace of the Model 183. The CO_2 produced is then measured directly by a non-dispersive infrared absorbance detector or by trapping the CO2 in an alkaline scrubber solution with subsequent analysis of inorganic carbon by non-dispersive infrared absorption. The instrument software is used to integrate the resulting absorbance.

The applicable range is .1 - 5 % carbon.

2.0 Definitions

TOC - Total Organic Carbon

3.0 Applicable Documents/References

Phoenix 8000 User Manual

APHA. 1998. *Standard Methods for the Examination of Water and Wastewater*, 20th Edition. Method 5310 C.

SW 846 3rd Edition. Method 9060.

4.0 Materials and Apparatus

4.1 Reagents/Standards

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date.

Reagent Grade Water (Milli-Q®)

Milli-Q® - acidified to a pH <2 with H_2SO_4

5.0 N Sodium Hydroxide

HCl, concentrated trace metals grade

Potassium Persulfate Reagent, 10% :
Dissolve 25.0 grams of reagent grade (98%) Sodium Persulfate ($Na_2S_2O_8$) in 213 mL of Milli-Q® water. Add 9 mL of concentrated phosphoric acid and mix well. Fill reservoir bottle for instrument and let sit overnight before use. Expiration is 10 days.

Phosphoric Acid, 21% :
Add 37mL reagent grade (85%) phosphoric acid to 188mL with Milli-Q® water (final volume 225mL). Fill reservoir bottle for instrument. 1 month expiration.

Potassium hydrogen phthalate ($KC_8H_5O_4$) Organic Carbon Standard, 40,000 ug/mL:
0.85g of KHP ($KC_8H_5O_4$) and 50uL of phosphoric acid to a final volume of 10mL with Milli-Q.

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1 month expiration.

Inorganic Carbon Standard 1000 ug/mL:
0.8825g Na_2CO_3 and 1 pellet of NaOH to a final volume of 100mL with Milli-Q. Also prepare a second 1000 ug/mL standard with a second source for use in the initial calibration verification. 1 month expiration.

Working Calibration Standards, 20 ug/mL, 15, 10, 5, and .4ug/mL Carbon

Concentration (mg/L)	mL of 1000mg/L standard	Final volume with Milli Q
20	0.40	100mL
15	0.30	100mL
10	0.20	100mL
5.0	0.10	100mL
0.40	2.0 (20.0 mg/L standard)	100mL

4.2 Equipment and Supplies:

Conditioning oven, electric, gravity convection, capable of maintaining a stable temperature of up to 200˚C
Combustion furnace, electric, capable of combusting glassware at 400˚C for at lease 4 hours
Phoenix 8000 Total Organic Carbon Analyzer, IR detector and 36 position autosampler rack with Model 183 boat accessory
combustion boats (platinum or quartz)
Analytical balance, capable of weighing to 0.5 mg
Calibrated weights, certified
Glass or stainless measuring scoop
Mortar and pestle
Forceps
Granular copper, 20-30 mesh
Oxygen, 99% pure
Graduated cylinders
Adjustable pipettes
Glass vials
Crucibles
Glass impingers, 50 mL, graduated

5.0 Methods/Procedures

Samples should be collected in glass containers with teflon of foil lined caps. Samples may be air dried and should be maintained at 4°C in the dark. Analysis should be performed within 120 days from sampling.

5.1 Instrument Start Up

As per Phoenix 8000 instrument user manual.

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The combustion oven is turned on and allowed to come up to temperature.
The oxygen tank is opened to allow around 30 psi to flow through the oven.

5.2 Preparation of samples for analysis

To a pre-weighed crucible add a 20-30g portion of sample. Record the weight of sample and cucible. Dry at 103°C and record the weight of crucible and dried sample. Subtract the weight of the crucible from the weight of dry sample and crucuble to get the weight of dried sample. Acidify the dried sample in the crucible by moistening with small amounts of concentrated HCl. Add acid until no reaction is observed. Dry sample again and record the weight of treated sample and crucible. Sub tract the crucible weight to get the weight of acid-treated dried sample.

Grind the sample into a fine powder and store in a glass vial.

Tare a pre-ignited sample boat. Weigh 100 mg of acid-treated dried sample into the boat and record the weight to the nearest tenth of a milligram.

Load the boat into the furnace sample introduction port, close and seal the port. Pre-purge to waste with oxygen for 1 to 3 minutes. Re-direct the oxygen stream to the analyzer for direct analysis or to the impinger with scrubber solution for off-line sample preparation. Advance the boat into the furnace and combust for 1-5 minutes. Withdraw the sample and allow to cool before removing it from the apparatus.

If sample is collected in scrubber solution measure 100 mL of acidified Mili-Q into graduated cylinder and add enough 5N NaOH to bring the pH above 10. The 100mL is then portioned into two graduated impingers arranged in series.

Blanks, laboratory control samples, standard reference materials and spiked samples are combusted in the furnace and analyzed in the same manner as samples.

Analyze combusted samples directly with the Phoenix 8000 in IC mode. Combine impinger solutions and analyze with the Phoenix 8000 in IC mode diluting as needed to remain within the calibration range.

6.0 Quality Control Requirements

The objective reporting limit is 0.1%. The most recent MDL study can be found in the inorganics quality control directory.

6.1 QC Elements

Demonstration of capability (DOC), four replicates of a prepared quality control sample. Required with a change of instrument, method, or analyst.

Method detection limit (MDL) study, calculated from seven replicate 0.1% carbon laboratory control samples (standard deviation * 3.14). Performed annually, with change in instrument, or method.

Analyze a blank, laboratory control, sample spike, sample duplicate, and optionally a laboratory control sample duplicate every analytical batch or twenty samples, whichever is more frequent. Analyze a standard reference material (SRM) and standard reference material duplicate (SRMD) as required by project-specific quality assurance plans.

Initial calibration consisting of a blank and five inorganic carbon standards.

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Initial calibration verification (ICV) and initial calibration blank (ICB) areanalyzed following analysis of initial calibration standards.

Continuing calibration verification (CCV) and continuing calibration blank (CCB) are analyzed after every ten samples and at the end of an analytical session.

QC elements may be added or removed to meet specific program or project requirements.

6.1 Acceptance Criteria

Demonstration of capability samples must meet LCS acceptance criteria.

The calculated MDL must be lower than the reporting limit.

Acceptance criteria for initial calibration is a relative standard deviation for response factors less than 15% or a correlation coefficient of greater than 0.995.

Acceptance criteria for all blanks is < 0.1% (1 mg/L at the instrument) or less than 1/10 of sample value.

Acceptance criteria for LCS recovery is 70%-130%. Acceptance criteria for LCSRep1/LCSRep2 percent relative range is less than or equal to 30%.

Acceptance criteria for SRM recovery is 70%-130% or that set by the supplier. Acceptance criteria for SRMRep1/SRMRep2 percent relative range is less than or equal to 30%.

Each sample is analyzed in duplicate. Each value is reported (Total organic carbon Replicate 1 and Total organic carbon Replicate 2). No method-specific acceptance criteria are set for sample replicate relative range due of the limitation of the method to small aliquot size and the wide range of sample homogeneity encountered.

Acceptance criteria for MS recovery is 60%-140%. Acceptance criteria for MSRep1/MSRep2 percent relative range is less than or equal to 30%. The limit is advisory. Sample values are qualified if the limit is not met but no corrective action is necessary.

Acceptance criteria for the initial calibration verification standard (ICV) is ± 15%.

Acceptance criteria for the continuing calibration verification standard (CCV) is ± 25%.

Alternate acceptance criteria may be set for specific programs or projects.

6.3 Interferences

The possible interferences are site specific and should be discussed with the technical director prior to the initiation of sample analysis.

7.0 Calculations/Reporting

7.1 Record

Record the following:

1. Date

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2. Analysts initial
3. Batch
4. Sample ID / Standard ID, with dilution factor if applicable
5. Sample weights
5. ID of reagents
6. Amount/ID of standard
7. Data file name
8. Notes, if any

7.2 Calculation

Calibrate the system software as per the Phoenix 8000 User Manual. (see section 6.0). Use the inorganic carbon values from the instrument to calculate the % carbon for QC elements and samples. Review the data to ensure it is correct and that all quality control requirements have been met.

% Carbon = (mg Carbon / mg sample) * 100

$$\text{mg Carbon} = \frac{\text{instrument value (mg/L)} * \text{FV (mL)} * \text{dilution}}{1000\ (\text{mL/L})}$$

mg sample = acid sample ratio * mg sample

$$\text{acid sample ratio} = \frac{\text{dried sample wt - crucible wt}}{\text{treated dried sample wt - crucible wt}}$$

Calculate LCS/ICV/CCV Recovery as follows:

$$\frac{\text{LCS/ICV/CCV concentration}}{\text{Concentration added}} \times 100 = \%\ \text{recovery}$$

Calculate %RPD as follows:

$$\frac{\text{Abs(Result 1 - Result 2)}}{\text{Avg. of results}} \times 100 = \%\text{RPD}$$

Calculate Spike Recovery as follows:

$$\frac{\text{Spike P concentration - Sample P concentration}}{\text{Concentration added to Spike}} \times 100$$

8.0 Corrective Actions

8.1 Data Qualification

DOC's and MDL's should be redone until they meet acceptance criteria.

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If the initial calibration curve is not within acceptance criteria, recalibrate or qualify (J) any positive sample values as estimated.

If the ICV does not meet acceptance criteria prepare a new ICV or new curve and ICV or qualify (J) any positive sample values as estimated.

Samples analyzed with blanks (prep, initial, continuing) not meeting acceptance criteria may be reanalyzed or reported with qualification (B).

If the LCS or LCS replicates do not meet percent recovery and/or percent relative difference (RPD) acceptance criteria they should be reanalyzed along with associated QC and samples or samples must be reported with qualification (J).

If the spiked sample recovery or spiked sample replicates are not within acceptance criteria samples may be reported with qualification (J) or reanalyzed. If the sample result is greater than 5 times the spike amount the percent recovery need not be evaluated.

If a CCV does not meet acceptance criteria all associated samples must be reanalyzed or qualified (J) as estimated.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified:

1. Quality and expiration date of reagents
2. Cleanliness of containers, equipment, and labware
3. Integrity of standards
4. Combustion oven operation
5. Phoenix 8000 operation
   See section 7 of Phoenix 8000 User Manual

9.0 Health and Safety

The combustion unit may be hot and can cause burns.

Gloves and safety glasses should be worn when handling samples and chemicals. It is advisable to wear a lab coat to protect clothing.

Strong acids and bases should be added to containers and solutions under the hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

Analysts should familiarize themselves with any chemical substances which are named along with their corresponding MSDS sheets.

10.0 Responsibilities

It is the analyst's responsibility to read and follow this standard operating procedure. Successfully perform a demonstration of capability. Record all data as per section 7.0. Review all QC results for compliance with acceptance criteria.

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It is the lab supervisor's responsibility to make sure standard operating procedures are up to date and that acceptance criteria have been set.

11.0 Waste Management and Pollution Prevention

Unused non-hazardous sediment sample may be disposed of as general trash.

TOC analyzer waste, reagents, and standards are disposed of in designated dilute acids and metals waste containers.

Minimize reagent use. Prepare reagents in amounts that will be used before expiration. Dispose of samples, reagents, and wastes in accordance with ESI policy and state and federal regulation.

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TITLE: Analysis of Selected PCB Congeners by GC/MS - Region II Testing

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	SOP Preparation	A. MacKinnon	01/2018

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TITLE: Analysis of Selected PCB Congeners by GC/MS - Region II Testing

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the gas chromatographic/mass spectrometric method used to analyze extractable PCB Congeners in water, soil, and tissue matrices by either EPA method 8270C or EPA method 680. This protocol is designed for use with samples from US EPA Region II and the New York District ACOE. Specific analytes include:

PCB 8	2,4'-dichlorobiphenyl
PCB 18	2,2',5-trichlorobiphenyl
PCB 28	2,4,4'-trichlorobiphenyl
PCB 44	2,2',3,5'-tetrachlorobiphenyl
PCB 49	2,2',4,5'-tetrachlorobiphenyl
PCB 52	2,2',5,5'-tetrachlorobiphenyl
PCB 66	2,3',4,4'-tetrachlorobiphenyl
PCB 87	2,2',3,4,5'-pentachlorobiphenyl
PCB 101	2,2',4,5,5'-pentachlorobiphenyl
PCB 105	2,3,3',4,4'-pentachlorobiphenyl
PCB 118	2,3',4,4',5-pentachlorobiphenyl
PCB 128	2,2',3,3',4,4'-hexachlorobiphenyl
PCB 138	2,2',3,4,4',5'-hexachlorobiphenyl
PCB 153	2,2',4,4',5,5'-hexachlorobiphenyl
PCB 170	2,2',3,3',4,4',5-heptachlorobiphenyl
PCB 180	2,2',3,4,4',5,5'-heptachlorobiphenyl
PCB 183	2,2',3,4,4',5',6-heptachlorobiphenyl
PCB 184	2,2',3,4,4',6,6'-heptachlorobiphenyl
PCB 187	2,2',3,4',5,5',6-heptachlorobiphenyl
PCB 195	2,2',3,3',4,4',5,6-octachlorobiphenyl
PCB 206	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl
PCB 209	2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl

Detection limits of less than 1.0 ng/g in sediment, 0.4 ng/g in tissue, and 0.002 µg/L in water are typical. Instrumental procedures and their requirements are a modification of those described in referenced EPA methods. One modification is in tuning the mass spectrometer for maximum sensitivity in the mass to charge region of 400 to 500 amu. Mass assignments and characteristic ion cluster ratios may be verified with decafluorotriphenylphosphine, but the ratios of 69,125, and 442 to mass 198 are set to accommodate the desired increase of this mid-mass sensitivity. Performance of this method is limited to analysts experienced in GC/MS operation and in the interpretation of gas chromatograms and mass spectra.

The chromatography columns called for in this method do not resolve each of these listed target analyte from all other of the 209 PCB congeners with the same level of chlorination, but literature review and in-house method validation shows that most coelutions are with homologs not found in the aroclors at significant levels. Contributions to target values from unresolved compounds that may occur are to PCB 49 from 47, PCB 87 from 115, PCB 118 from 149, PCB 138 from 163 and 164, and to PCB 156 from 171.

2.0 Definitions

See ESI's Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Method 8270C (SW-846).
Agilent Technologies. 1997-2000. *Operation Guide.* Agilent 7683 Automatic Liquid Sampler.

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US EPA. 1985. *Test Methods for Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectroscopy*. Method 680 (SW-846).

US EPA. 1999. *Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS*. Method 1668. Revision A.

NOAA. 1993. *Sampling and Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Project*. Analytical Procedures to Quantify Organic Contaminants (Volume IV). NOAA Technical Memorandum NOS ORCA 71.

Department of Defense (DoD), Department of Energy (DoE). 2017. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.1.

4.0 Materials and Apparatus

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date. Standard composition and concentration may vary based on project-specific requirements.

4.1 Reagents and Standards

4.1.1 Solvents, pesticide grade

4.1.2 10-50 ng/µL Decaflurotriphenylphosphine (DFTPP)

Vial is stored tightly capped and in the freezer between uses. Standard is injected to tine the mass spectrophotometer and is used to check daily prior to running a batch.

4.1.3 PCB Congener Surrogate Standard

Standard in methylene chloride a minimum of 2 µg/mL CGR 198 and 2 µg/mL CGR 103 (optional)

4.1.4 PCB Congener Spike Standard

Standard in hexane containing 2.0 µg/mL of target the analytes:

PCB 8	2,4'-dichlorobiphenyl
PCB 18	2,2',5-trichlorobiphenyl
PCB 28	2,4,4'-trichlorobiphenyl
PCB 44	2,2',3,5'-tetrachlorobiphenyl
PCB 49	2,2',4,5'-tetrachlorobiphenyl
PCB 52	2,2',5,5'-tetrachlorobiphenyl
PCB 66	2,3',4,4'-tetrachlorobiphenyl
PCB 87	2,2',3,4,5'-pentachlorobiphenyl
PCB 101	2,2',4,5,5'-pentachlorobiphenyl
PCB 105	2,3,3',4,4'-pentachlorobiphenyl
PCB 118	2,3',4,4',5-pentachlorobiphenyl

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PCB 128	2,2',3,3',4,4'-hexachlorobiphenyl
PCB 138	2,2',3,4,4',5'-hexachlorobiphenyl
PCB 153	2,2',4,4',5,5'-hexachlorobiphenyl
PCB 170	2,2',3,3',4,4',5-heptachlorobiphenyl
PCB 180	2,2',3,4,4',5,5'-heptachlorobiphenyl
PCB 183	2,2',3,4,4',5',6-heptachlorobiphenyl
PCB 184	2,2',3,4,4',6,6'-heptachlorobiphenyl
PCB 187	2,2',3,4',5,5',6-heptachlorobiphenyl
PCB 195	2,2',3,3',4,4',5,6-octachlorobiphenyl
PCB 206	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl
PCB 209	2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl

4.1.5 PCB Congener Internal Standard

Standard containing a minimum of: 10 µg/mL phenanthrene-d10 and 10 µg/mL chrysene-d12

4.1.6 Standard reference material NIST 1493

4.2 Equipment and Supplies

Agilent 6890/5973 GC/MS
Agilent HP7683 autosampler, or equivalent
Electronic data capturing software and data analysis software
Column: Restek RT-X5 (30 to 60-m x 0.25-mm internal diameter fused silica capillary column with a 0.25 µm film thickness bonded phase), or equivalent DB-5 like column
Gas scrubbers for removal of moisture/impurities in the GC carrier gas
Gas tight syringes
Sample vials with screw caps

5.0 Methods and Procedures

5.1 Method Summary

Samples are extracted with a pesticide-grade solvent and the extracts are concentrated and carried through a series of cleanup techniques. The extract is then analyzed by injection into a gas chromatographic (GC) system where PCBs are detected by a mass spectrometer. Gas chromatographic/mass spectrometer (GC/MS) conditions, calibration procedures, and GC/MS data handling procedures for quantitating PCB congener concentrations are described below.

Sample extract components are separated with capillary column gas chromatography (GC) and identified and measured with low resolution, electron ionization mass spectrometry (MS) operated in the selected ion monitoring mode (SIM). An interfaced data system is used to control data acquisition and to store, retrieve, and manipulate mass spectral data. A properly configured and tuned Agilent 6890 GC/5973 MS is suitable perform this analysis. SIM mode is the acronym for Selected Ion Monitoring, where the MS is programmed to monitor and record responses from only those ions needed for quantitation, confirmation of analyte identity, and for verification of absence of some interferences.

One or two surrogate compounds are added to each sample before sample preparation; these compounds are PCB 198 and optionally PCB 103. Two internal standards, chrysene-d12 and phenanthrene-d10, are added to each sample extract before GC/MS analysis and are used to calibrate MS response. Each analyte concentration value is based on the integrated ion abundance of one characteristic ion. The concentration is measured for each PCB target and surrogate congener. Total PCB concentration in each sample extract may be estimated by summing the congener concentrations and multiplying that sum by 2. Each

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selected PCB congeners is included in calibration standards, and is assigned to one of the two internal standards, chrysene-d12 and phenanthrene-d10.

5.2 Mass Spectrometer Tuning and GC Column Performance Check

The MS is initially tuned to meet the manufacturer's specifications using PFTBA. Mass fragmentation criteria have been set for the tuning compound decafluorotriphenyl-phosphine (DFTPP; see Appendix 2). The mass spectrometer is then hardware tuned to meet the these DFTPP criteria. The tune must be verified before any analyses are made, and every 12 hours of analysis time. The tune is checked by injecting 1 µL of 10-50 ng/µL DFTPP. Full scan spectra are obtained using tune parameters that will be used for calibration and sample analysis. The resulting DFTPP spectrum is checked against the criteria in Appendix 2 using the system software .

A report is generated that includes the criteria table, mass spectrum of passing scan, tabulation of ion fragment abundances normalized to m/e 198, and the abundance of m/e 198 (see section 1 for modification). Scans under the peak may be averaged, and background scans subtracted in evaluating the tune. A text file of the passing tune criteria is saved. Inspect the chromatogram for peak tailing.

5.2 Establish Instrument MS Conditions

For SIM Calibration for PCB determinations, set the MS to acquire SIM response for the ion sets below over the appropriate retention time windows. Approximate relative retention times of calibration congeners and approximate relative retention time windows for PCB isomer groups are:

Isomer Group	Approximate CGR RRT Range	CGR Start	CGR End	Conf. Ion	Int. Ion	+70 Ion	+35 Ion
Monochlorobiphenyls	0.50 -0.72	- -	8	188	190	256	222
Dichlorobiphenyls	0.60 -0.85	8	52	222	224	292	256
Trichlorobiphenyls	0.70 -0.90	18	52	256	258	326	290
Tetrachlorobiphenyls	0.75 -0.10	52	77	290	292	360	326
Pentachlorobiphenyls	0.75 -0.93	101	126	324	326	394	360
Hexachlorobiphenyls	0.90 -1.07	153	128	358	360	430	394
Heptachlorobiphenyls	0.90 -1.13	184	170	392	394	464	430
Octachlorobiphenyls	0.95 -1.25	198	195	430	428	498	464
Nonachlorobiphenyls	1.05 -1.25	195	209	464	466		498
Decachlorobiphenyl	1.05 -1.25	195	209	498	500		

Retention time relative to chrysene-d12 with a 30 m · 0.25 mm ID RTX-5 fused silica capillary.

5.3 Calibration

Initial multipoint calibration is performed to determine the instrument response factors over the concentration range to be used. When the compound response is linear over the calibration range (RSD of response factors within limit) the average response factor may be used to calculate target analyte concentrations. Each day that samples are to be analyzed, a continuing calibration verification (CCV) standard is analyzed. The CCV standard response is evaluated for percent recovery to initial calibration response. If acceptance criteria are met, the initial calibration response may be used for quantitation, otherwise a new initial calibration is obtained or data are qualified.

Analyze a minimum of five analytical standards that will represent the concentration range of the samples. Calibration range is typically from 2 ng/mL to 200 ng/mL.

The concentration of the low standard should be 2 to 3 times the detection limit and is the

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reporting limit. The high concentration standard is typically 200 ng/mL but could be as much as ten times higher without loss of linearity.

For each level of calibration, calculate the response factor for each analyte of interest present in the calibration standard using the software supplied with the GC/MS data system. For each analyte, calculate the average response factor (RF) and the RF percent relative standard deviation (RSD). RSD acceptance criteria for each target is less than 20% or a program or project specific limit.

An initial calibration verification (ICV) standard from a second source is analyzed. Acceptance criteria for percent recovery is 20% for each target analyte or as specified in a project or program-specific QA plan.

5.4 Internal Standards

Quantitation for this method is done using the internal standard method. Five microliters of 40 ng/µL internal standard are added to each 200 mL of calibration standard or sample extract. The ratio of target response to internal standard response (the response factor, RF) is calculated for each target compound at each calibration level according to the equation below:

$$RF = \frac{(\text{area target})\,(\text{IS concentration})}{(\text{area IS})(\text{target concentration})}$$

For each target analyte the response factors for all levels in the calibration are averaged. The average response factor is used to calculate its concentration in sample extracts according to the equation below:

$$\text{Target Concentration} = \frac{(\text{area target in sample})(\text{IS concentration})}{(\text{area IS in sample})(\text{avg RF of target})}$$

The internal standard contains phenanthrene-d10 and chrysene-d12 at 40 µg/mL.

5.5 Continuing Calibration

Instrument calibration is checked each day prior to sample analysis by the injection of a calibration standard and a calibration blank. With the following procedures, verify initial calibration every 10 samples, or mid batch for batches of less than 20.

Analyze a 1 µL aliquot of the 10-50 ng/ml DFTPP solution and verify acceptable MS calibration and performance. Inject an aliquot of the Medium CAL standard and analyze with the same conditions used during initial calibration.

Determine that neither the area measured for m/z 240 for chrysene-d12 nor that for m/z 188 for phenanthrene-d10 has decreased by more than 20% from the area measured in the most recent previous analysis of a calibration solution or by more than 20% from that measured at initial calibration.

Response Factor Recovery - the measured RF for each analyte and surrogate compound must be within ±20% of the initial calibration. If not, repeat the calibration verification, recalibrate or qualify associated positive sample results.

SIM Analyte Retention Time Reproducibility - the target analyte retention times should be with ±10 seconds of the previous calibration verification, but analyst experience must be used.

5.6 Sample Handling

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Aqueous samples should be collected in 1 L glass jars with Teflon lined caps. Remove known free residual chlorine with ascorbic acid. Adjust pH to 5 - 9 SU if needed. Store samples at 4±2°C. Holding time for aqueous samples is 7 days, for solids it is 14 days (1 year if frozen), and for tissues it is 1 year if frozen. Sample preparation areas and equipment must be scrupulously precleaned so as not to introduce contamination. Precleaning may include soap and water, solvent rinse, or ignition in a muffle furnace. Samples may be composited at client request.

Sample extracts are stored in 2 mL screw top vials with Teflon septa labeled with lab ID, fraction, final volume, and IS ID. Extracts are refrigerated when not under analysis. All extracts must be analyzed within 40 days. Extracts are disposed of after approximately 2 months or according to special contract. The extracts are considered hazardous waste and are disposed of accordingly.

5.7 Sample Preparation

Detailed preparation procedure for extraction is described in the appropriate preparatory SOP which details how a sample is spiked with surrogate standards, extracted, cleaned up, and adjusted to final volume. The extracts are placed in 2 m glass screw top vials. A blank is extracted with every batch of samples. A spiked sample or laboratory control sample is extracted with each batch samples or by client request. Moisture content is determined for solids.

5.8 Instrument Analysis

Acquire GC/MS response for each sample and QC sample using the same instrument conditions used for initial calibration. Instrument run information is recorded including date, acquisition method name, sample file number, sample ID, internal STD ID, analysts initials and notes.

An example daily analytical sequence follows:

a) Tune evaluation standard (DFTPP 10-50 ng/µL)
b) Five-point calibration sequence with ICV standard or CCV standard
c) ICB
d) Sample extracts (prep blanks, LCSs, samples, and spikes)
e) 12-hour DFTPP tune evaluation standard
f) CCV standard run every 10 samples or the middle of a batch of fewer than 20 samples
g) CCB run every 10 samples or the middle of a batch of fewer than 20 samples
h) Sample extracts

5.9 Data Analysis

Using SIM GC/MS an analyst competent in the interpretation of mass spectra identifies each analyte in a sample by comparing the ratio of the target ion and the confirmation ion with that of the authentic analyte. Guidance is provided in Table 1. Criteria that must be satisfied to verify the identifications of target compounds includes elution of the sample analyte within GC retention time window (typically 0.05 minutes or less) and correspondence of the sample analyte ion ratios and those of calibration standards

For each target analyte, the corresponding confirmation ion or ions listed must be present and the relative intensities must agree to within ±20 percent. This criteria may be modified for trace levels and is left to the project manager's/analyst's discretion.

A quantifiable analyte peak should exhibit a signal to noise ratio of approximately 2:1 or greater unless specified otherwise in the project protocol. Target PCB congener compounds are quantified by the internal standard method, using the average response factor from the initial calibration unless otherwise specified in the project protocols. The primary and

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confirmatory characteristic ion for each target analyte listed in Table 1 is used for quantitation.

For target congeners with Cl3-Cl7 levels of chlorination, examine each target's ion current profile for any intense coeluting peak which contains two additional chlorines. If present, an optional correction can be made. Obtain and record the area for the appropriate ion for the target analyte. Use the information in Table 2 to correct the measured area of that target congener. For example, if a Cl7-PCB elutes in the PCB 115 window as indicated by a large m/z 394 peak, the Cl7-PCB will contribute to the areas for m/z 326 and 324. Obtain and record the area for m/z 394 (the quant mass for the Cl7-PCB). Multiply this area by the correction factor in Table 4 and subtract from the total m/z 326 area. Alternatively, obtain and record the area for m/z 322 in the coeluting peak (a characteristic m/z of Cl7 not present in Cl5). Multiply this area by the correction factor in Table 3 and subtract from the total m/z 326 area.

For Cl2-Cl8 isomer groups, examine ICPs for intense peaks that would indicate a coeluting PCB containing one additional chlorine. This coelution causes interferences because of the natural abundance of 13C. (This interference will be small and can be neglected except when measuring the area of a small amount of PCB homologs coeluting with a large amount of other PCBs containing one higher level of chlorination.) To correct for this interference, obtain and record the area for the appropriate ion in Table 4 for the candidate PCB isomer group. Use the information in Table 3 to correct the measured abundance.

Use the data system software to generate an ion current profile ratio of the measured peak response of the confirmation ion(s) relative to the quantitation ion, and compare to the acceptable ratio. Verify the presence of M-70 ion where applicable. If acceptable ratios are not obtained, a coeluting or partially coeluting compound may be interfering. Examination of data may provide information that will allow application of additional background corrections to improve the ion ratio.

Manual integration is an important component of sample analysis and must be applied to ensure that the most accurate data are generated. Given the complex nature of environmental samples it is critical that a trained analyst reviews each individual analyte in each sample using a GC/MS data system, and manually optimizes the integration as needed. Each analyst is trained in proper peak integration (see SOP QA-1132 "Manual Chromatographic Peak Integration"). The data system baseline integration can be used to quantify samples. If, during the review of the chromatogram, the analyst notices improper integration by the data system, he/she will manually reintegrate the peak using proper integration techniques. Peaks that have been manually integrated are identified on the quantitation reports when an "m" appears adjacent to the area response.

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TITLE: Analysis of Selected PCB Congeners by GC/MS - Region II Testing

Table 1. Ion Ratios for Identification of PCB Congeners

Analyte	Quant. Ion (Q)	Confirm Ion (C)	%C/Q
Monchlorobiphenyls	188	190	30
Dichlorobiphenyls	222	224	65
Trichlorobiphenyls	256	258	95
Tetrachlorobipheny	292	290	75
Pentachlorobipheny	326	324	65
Hexachlorobiphenyl	360	362	80
Heptachlorobipheny	394	396	95
Octachlorobiphenly	430	428	90
Nonchlorobiphenyls	464	466	75
Decachlorobiphenyl	498	500	85

If coelution of a target analyte and a congener of higher chlorination occurs, interference ions listed in Tables 2-4 may be measured and, if present at levels that would affect quantitation ion areas, appropriate manual quantitation correction may be made and documented with the raw data.

Table 2. Correction for Interference from Congeners with Two Additional Chlorines

Candidate Isomer Group	Quant. Ion	Confirm. Ion	Ion to Determine Interference	% of Interf. Ion Area to be Subtracted from Quant. Ion Area	% of Interf. Ion Area to be Subtracted from Confirm. Ion Area
Trichlorobiphenyls	256	258	254	99%	33%
Tetrachlorobipheny	292	290	288	65%	131%
Pentachlorobipheny	326	324	322	108%	164%
Hexachlorobiphenyl	360	362	356	161%	71%
Heptachlorobipheny	394	396	390	225%	123%

Table 3. Alternate Correction for Interference from Congeners with Two Additional Chlorines

Candidate Isomer Group	Quant. Ion	Confirm. Ion	Ion to Determine Interference	% of Interf. Ion Area to be Subtracted from Quant. Ion Area	% of Interf. Ion Area to be Subtracted from Confirm. Ion Area
Trichlorobiphenyls	256	258	326	56%	%
Tetrachlorobipheny	292	290	360	38%	%
Pentachlorobipheny	326	324	394	50%	%
Hexachlorobiphenyl	360	362	430	47%	%
Heptachlorobipheny	394	396	464	44%	%

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Table 4. Correction for Interference from Congeners with One Additional Chlorine

Candidate Isomer Group	Quant. Ion	Ion Measured to Determine Interference	% of Meas. Ion Area to be Subtracted from Quant. Ion Area
Dichlorobiphenyls	222	221	13.5%
Trichlorobiphenyls	256	255	13.5%
Tetrachlorobiphenyls	292	289	17.4%
Pentachlorobiphenyls	326	323	22.0%
Hexachlorobiphenyls	360	357	26.5%
Heptachlorobiphenyls	394	391	30.9%
Octachlorobiphenyls	430	425	40.0%

6.0 Quality Control Requirements

Quality control elements may be added, removed, or altered to meet specific program or project requirements. Acceptance criteria shall be updated based on the associated control charts once sufficient data points have been collected (or atleast 20 data points) and yearly thereafter. Alternate acceptance criteria may be set for specific programs or projects. The objective reporting limits can be found in Appendix 1 and the most recent MDL study can be found in the Organics directory. For DoD-specific projects see the QSM 5.1, as appropriate.

All QC elements, samples, and calibration standards must be spiked with internal standards and all QC elements and samples must be spiked with a surrogate standard. MS is tuned with DFTPP; see Appendix 2 for DFTPP criteria.

6.1 Demonstrations of Capability (DOC)

An analyst must complete a DOC before working independently on this procedure and yearly thereafter. A DOC is also required with any changes to instrument, method, or analyst. To perform a DOC, four replicates of a prepared quality control sample must be prepared. DOC samples must meet LCS acceptance criteria. For more information on DOCs see SOP QA-1108 “Training”.

6.2 Method Detection Limit (MDL)

The MDL is calculated using the appropriate student’s t-parameter value multiplied by the standard deviation of 7 or more replicates of spiked samples or standards. MDLs are performed annually and with major changes in instrument or method. The calculated MDL must be lower than the reporting limit.

The t-parameter value is from a one-tailed Student’s t-Test at the 99% confidence interval using n-1 degrees of freedom, with “n” being the number of replicates.

The most recent MDL study can be found in the organics quality control directory on the ESI network. For more information on MDLs see SOP QA-1111 “Determination of Method Detection and Reporting Limits”.

6.3 Limit of Detection (LOD)

LOD determination will be conducted on a quarterly basis. For a single-analyte standard, the LOD is a single quality control sample that is approximately 2 to 3 times the MDL but no greater than the reporting limit. For a multi-analyte standard, the LOD is a single quality

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control sample that is approximately 1 to 4 times the MDL but no greater than the reporting limit.

The apparent signal to noise ratio at the LOD must be at least 3. For methods that do not produce a signal to noise, the LOD result must be at least 3 standard deviations greater than the mean method blank.

6.4 Limit of Quantitation (LOQ)

LOQ confirmation will be conducted on a quarterly basis. The LOQ is a single quality control sample no more than 1 to 2 times the reporting limit. The LOQ standard acceptance criteria is 30-150%.

For more information on LODs and LOQs see SOP QA-1147 “Determination of LOD and LOQ Quality Control Samples”.

6.5 Blank, LCS, Matrix Spike (MS) samples, Standard Reference Material (SRM), Relative Standard Deviation (RSD)

6.5.1 A blank, LCS, MS, MS duplicate (MSD), and MS triplicate (MST) will be analyzed every batch, every 20 samples, or as provided in the prep method. An SRM will be analyzed every batch, every 20 samples, or as provided in the prep method for aqueous and sediment matrices.

6.5.2 Acceptance criteria for all blanks is less than the reporting limit.

6.5.3 Acceptance criteria for LCS recovery is 50%-150%. The LCS will be spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix 1.

6.5.4 Acceptance criteria for spiked sample recovery is 50%-150%. The MS/MSD/MST samples will be spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix 1.

6.5.5 Acceptance criteria for RSD between matrix spike duplicates is $\leq$50%.

6.5.6 MS/MSD/MST samples are extracted either per client request or per project requirements. Every attempt will be made to obtain enough sample to perform a matrix spike with 5% frequency. If a single sample needs to be extracted and there is not enough sample volume for a matrix spike, a synthetic sample, or LCS duplicate may be extracted and analyzed to provide 5% spike occurrence. For aqueous analysis site water will be used to perform all MS/MSD/MST samples. For sediment analysis reference sediment will be used to perform all MS/MSD/MST samples. For tissue analysispretest tissue will be used to perform all MS/MSD/MST samples.

6.5.7 Acceptance criteria for surrogate standard recovery is 30-150%. All applicable surrogate standards must be within the acceptance limits to be considered acceptable.

6.5.8 Acceptance criteria for standard reference material recovery is 50-150%. The SRM is evaluated for analytes at >3x the reporting limit. Reporting limits can be found in Appendix 1.

6.6 Calibration, Calibration Verifications and Blanks

6.6.1 Initial calibration consists of a blank and a minimum of 5 standards. Initial Calibration Verification (ICV) and Initial Calibration Blank (ICB) are performed following analysis of initial calibration standards. Continuing Calibration Blank (CCB) and Continuing

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Calibration Verification (CCV) are performed every 10 samples, or mid batch for batches of less than 20.

6.6.2 Acceptance criteria for in initial calibration is a relative standard deviation (RSD) for response factors less than 20%.

6.6.3 Acceptance criteria for the ICV and CCV standards is a recovery of ±20%. Acceptance criteria for the ICB and CCB is less than the reporting limit.

6.7 Areas of internal standards are monitored to check system stability. Samples must have IS areas 50- 200% of those of the initial or continuing calibration standard. IS retention times must fall within 30 seconds (and should be within 10 seconds) of those in the initial or continuing calibration standard.

6.8 Refer to US EPA methods 680 and 8270C for a complete list of interferences

7.0 Calculations and Reporting

7.1 The following information should be recorded when performing an analysis of selected PCBs Congeners by GC/MS:

- Date
- Analyst's initial
- Batch
- Sample ID / Standard ID, with dilution factor if applicable
- Data file name
- Acquisition method
- Notes, if any

7.2 Calculations

7.2.1 For every sample that is run, a quantitation report is generated on the host computer. This report lists the retention times and areas for all peaks which match the standard library criteria. For GC/MS the criteria includes elution time within a narrow window, and a match with standard spectra. The concentration is also reported, based on response factors calculated from the daily standard. This concentration is the value for analytes in the extract itself; it does not account for dilutions or exact volume/mass of sample used to prepare the extract. Retention time of hits are compared to those of the standard, and questionable integrations and spectra are checked by the analyst. The chromatogram is inspected for unintegrated peaks. Large nontargets are identified by library searching.

The results to be listed on the final report are calculated using the system software generated analyte value, analyst provided instrument dilution factor, laboratory provided sample volume or weight, moisture content if needed and extract final volume(s) as follows:

$$\frac{(\text{Qrpt Value}) \times (\text{Inst Dilution}) \times (\text{Prep Final Vol}) \times (\text{Prep Dilution})}{(\text{Prep Amount}) \times (\text{Optional Dry Fraction})} = \text{Report Value}$$

Where:

Qrpt Value = µg/mL in the (diluted) extract (taken from GCMS quantitation report)
Inst Dilution = Dilution of final extract prior to IS addition and injection
Prep Final Vol = mL final volume of prepared extract (taken from prep sample table)
Prep Dilution = Dilution of initial extract, if any, during prep (taken from prep sample

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table)
Optional Dry Fraction = Solid sample dry fraction relative to condition as received (taken from table of separate dry weight analysis results)

The concentrations for solids are reported on a dry weight basis. A draft of the final report is printed in the laboratory for validation by the analyst. This is forwarded to the Lab Manager for final review and sign-off before generation of the finished report for the client.

7.2.2 Calculate the Standard Recovery as follows:

$$\frac{\text{Standard Concentration}}{\text{Concentration Added}} * 100 = \%\ \text{Recovery}$$

7.2.3 Use the following equation to calculate the %RSD:

$$\frac{\text{Std(Result 1 - Result 2)}}{\text{Avg(Result 1 - Result 2)}} * 100 = \%\text{RSD}$$

7.2.4 Calculate the Sample Spike Recovery as follows:

$$\frac{\text{Spike Concentration - Sample Concentration}}{\text{Concentration added to Spike}} * 100 = \%\text{Recovery}$$

8.0 Corrective Actions

All QC measures have associated acceptance windows. A failure of any of these measures may necessitate corrective action. The action may be to correct performance of a specific technique, or to repair a hardware defect. Affected samples should be re-analyzed or re-extracted first if necessary and then reanalyzed. Data that is unavoidably compromised and cannot be regenerated must be flagged. In such cases, the client is contacted promptly for more sample.

8.1 Data Qualification

8.1.1 If DOCs, MDLs, LODs, or LOQs do not meet acceptability criteria, then repeat until acceptability criteria is met.

8.1.2 If the DFTPP does not meet the criteria then the DFTPP must be reanalyzed or the hardware must be re-tuned.

8.1.3 If the initial calibration curve is not within acceptance criteria, then recalibrate or qualify any positive sample values as estimated (using the “J” qualifier).

8.1.4 If the ICV does not meet acceptance criteria prepare a new curve and ICV or qualify any positive sample values as estimated (using the “J” qualifier).

8.1.5 If samples are analyzed with blanks (prep, initial, continuing) not meeting acceptance criteria, then they should be reanalyzed or reported with qualification (using the “B” qualifier).

8.1.6 If the LCS samples do not meet percent recovery and/or RSD acceptance criteria, then they should be reanalyzed along with all associated QC and samples orreported with qualification (using the “J” qualifier).

8.1.7 If the MS recovery is not within acceptance criteria, then samples may be reanalyzed or reported with qualification (using the “J” qualifier). If the sample result is greater than 5 times the spike amount the percent recovery need not be calculated.

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8.1.8 If surrogate recovery is not within the acceptable range, the client is notified and the sample is re-extracted whenever possible. Sample matrix effect is proven if the results obtained from the re-extracted sample show similar results. When this occurs, both results are reported with an explanation. A problem with the first extraction is indicated when the results of the re-extracted sample are in the acceptable range. In this case, only the compliant results are reported. If no additional sample can be obtained, results are flagged in the report and a narrative is included qualifying the data.

8.1.9 If a CCV does not meet acceptance criteria, then all associated samples must be reanalyzed or qualified as estimated (using the "J" qualifier).

8.1.10 If the internal standards do not meet acceptance criteria, then all associated samples must be reanalyzed or qualified as estimated (using the "J" qualifier).

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified.

- Quality and expiration date of standards
- Cleanliness of containers, equipment, and labware
- Instrument operation

9.0 Health and Safety

9.1 Methylene chloride and PCB congener standards are extremely hazardous to everyone in the laboratory if handled improperly. It is important that all necessary safety precautions are followed. Gloves and safety glasses must be worn when handling samples, reagents, and standards. It is necessary to wear a lab coat to protect clothing. All solvent usage must occur in properly working fume hoods. Use precautions to prevent spills; if any spills do occur, clean up all spills immediately and notify supervisor.

9.2 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding Safety Data Sheets.

9.3 Strong acids and bases should be added to containers and solutions under the hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptance criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP, recording all data as described in Section 7.1, and reviewing all QC results for compliance with acceptance criteria.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities and DOC performance.

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11.0 Waste Management and Pollution Prevention

Unused non-hazardous sample may be disposed of in designated waste drums. Solvents not evaporated in a hood are collected and disposed of as hazardous waste in designated solvent water waste containers. Extracts and standard residues are deemed hazardous and are disposed of as such. Minimize solvent use and prepare standards in amounts that will be used before expiration.

11.1 Solid Materials

Waste storage, transfer and disposal will be in compliance with NH DES and US EPA waste disposal regulations. Non-hazardous solid materials, sediments and soils will be disposed of through an appropriate waste disposal site. All non-hazardous solid waste will be drained of as much free liquid as possible. The remaining sample is removed from its original container and placed with other non-hazardous waste samples in a 55 gallon metal drum. Once full, these containers will then be transferred to an approved waste disposal facility. This transfer is recorded on a waste manifest and they are retained for a minimum of 3 years.

11.2 Aqueous Samples

Water samples that have been solvent extracted are added to the solvent water waste stream. Any aqueous samples received unpreserved as extra volume that were not solvent extracted may be disposed of through ESI's aqueous waste UV treatment system located on the first floor. Access points for aqueous waste disposal are located throughout the laboratory, second floor's access "waste chute" is located in the dishwashing area. ESI's Federal and State NPDES Permit allows waste disposed of in this manner to be discharged to the Taylor River.

Any samples found to be toxic may require further testing or treatment in order to determine proper disposal technique to assure compliance with all local, state and federal regulations.

11.3 Hazardous Waste Samples

Hazardous waste samples must be handled by properly trained employees. Only the ESI hazardous waste coordinator may train ESI employees on handling hazardous waste. There are 3 main hazardous waste streams at ESI, "dilute metals acidic", "solvents" and "Lachat wastes". When a satellite container needs to be emptied, it is properly transported to the waste shed and added into the appropriately labeled container for that waste stream. The volume and type of waste is recorded in the appropriate log book. Shipping manifests reflect when the waste drums are picked up for transport.

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TITLE: Analysis of Selected PCB Congeners by GC/MS - Region II Testing

Appendix 1. Analyte List and Target Reporting Limits

Analyte	Extract (ng/mL)	Tissue (ng/g)	Sediment (ng/g)	Water (µg/L)
PCB 8	2	0.4	1	0.002
PCB 18	2	0.4	1	0.002
PCB 28	2	0.4	1	0.002
PCB 44	2	0.4	1	0.002
PCB 49	2	0.4	1	0.002
PCB 52	2	0.4	1	0.002
PCB 66	2	0.4	1	0.002
PCB 87	2	0.4	1	0.002
PCB 101	2	0.4	1	0.002
PCB 105	2	0.4	1	0.002
PCB 118	2	0.4	1	0.002
PCB 128	2	0.4	1	0.002
PCB 138	2	0.4	1	0.002
PCB 153	2	0.4	1	0.002
PCB 170	2	0.4	1	0.002
PCB 180	2	0.4	1	0.002
PCB 183	2	0.4	1	0.002
PCB 184	2	0.4	1	0.002
PCB 187	2	0.4	1	0.002
PCB 195	2	0.4	1	0.002
PCB 206	2	0.4	1	0.002
PCB 209	2	0.4	1	0.002

Appendix 2.

DFTPP Key Masses and Abundance Criteria

Mass	m/z Abundance criteria
51	10-60 percent of Mass 198
68	Less than 2 percent of Mass 69
70	Less than 2 percent of Mass 69
127	20-80 percent of Mass 198
197	Less than 1 percent of Mass 198
198	Base peak, for percent relative abundance
199	5-9 percent of Mass 198
275	5-100 percent of Mass 198
365	Greater than 1 percent of Mass 198
441	Present but less than Mass 443
442	Greater than 40 percent of Mass 198
443	17-23 percent of Mass 442

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Tune Report Example:

```
DFTPP

Data File : C:\DATAPATH\PAH\DFTPP.D Vial: 1
Acq On : 31 Dec 2000 8:35 am Operator: JFK
Sample : DFTPP O4747L40 Inst : GCM5973N02
Misc : Multiplr: 1.00
MS Integration Params: rteint.p

Method : C:\680PAH1.M (RTE Integrator)
Title : PAH Calibration

Spectrum Information: Average of 15.153 to 15.177 min.
```

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	10	60	13.7	72442	PASS
68	69	0.00	2	0.0	0	PASS
69	198	0.00	100	17.0	90408	PASS
70	69	0.00	2	0.4	342	PASS
127	198	10	60	34.0	180524	PASS
197	198	0.00	1	0.0	0	PASS
198	198	100	100	100.0	530511	PASS
199	198	5	9	6.7	35359	PASS
275	198	5	50	33.0	175154	PASS
365	198	1	100	4.8	25422	PASS
441	443	0.01	100	72.6	176685	PASS
442	198	39	400	242.3	1285498	PASS
443	442	17	23	18.9	243491	PASS

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TITLE: Extraction of Pesticides and PCBs in Aqueous Samples - Region II Testing

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	SOP Preparation	A. MacKinnon	01/2018

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1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the procedure for the extraction of organochlorine pesticides and PCBs from aqueous samples such as surface, well and discharge waters based on SW-846 methods 3510 and 3610, and EPA method 608. This protocol is designed for use with samples from US EPA Region II and the New York District ACOE. Samples prepared by this method are analyzed according to SOP QA-1351 "Analysis of Pesticides and PCBs in Extracts of Soil, Tissue and Water" based on SW-846 methods 8081A and 8082, and EPA method 608.

Water samples are spiked with a surrogate solution and shaken with solvent in a separatory funnel to extract the organic compounds. The solvent layers are collected and concentrated using a KD apparatus. The extract is put into a vial and refrigerated until GC analysis. Samples requiring a clean-up procedure will go through clean-up for pesticides(extract is eluted through a florisil column)or for PCBs (1 mL of concentrated sulfuric acid is added to the extract). Other clean ups are listed in methods 8081A and 8082 of SW 846.

2.0 Definitions

See ESI's Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

US EPA. 1996. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods*. Third Edition. Method 3510C (SW-846). (Consult for interferences and pollution prevention.)

US EPA. 1996. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods*. Third Edition. Method 3620 (SW-846).

US EPA. 1983. *Methods for Chemical Analysis of Water and Wastes*. Appendix A: Method 608.

US EPA Contract Laboratory Program Statement of Work for Organic Analysis, Multi-media Concentration. Revision 2/88, Pesticide D-11.

Agilent Technologies. 1997-2000. *Operation Guide.* Agilent 7683 Automatic Liquid Sampler.

Agilent Technologies. 2000. *Operating Manual.* Agilent 6890 Series Gas Chromatograph.

Agilent Technologies. 1989-2001. *G1701DA Version D.00.00.38 19-Nov 2001.* Environmental ChemStation.

Department of Defense (DoD), Department of Energy (DoE). 2017. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.1.

SOP QA-1132 "Manual Chromatographic Peak Integration"
SOP QA-1351 "Analysis of Pesticides and PCBs in Extracts of Soil, Tissue and Water"

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4.0 Materials and Apparatus

4.1 Reagents and Standards

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date. Refer to Appendix D for standard preparation.

1:1 Sulfuric acid
Acetone, pesticide grade
Florisil, activated at 675°C for 4 hours
Hexane, pesticide grade
Methylene chloride ($MeCl_2$), pesticide grade
Milli-Q® water
Sodium Sulfate, heated at 400°C for 4 hours to remove phthalates and other interfering organic substances
PCB Spike Standard in acetone containing 10.0 µg/mL aroclor 1016 and 1260
Pesticide Spike mix A and mix B supplied by Supelco or other acceptable retailers
Pesticide standard mix C, project specific
2,4'-DDE standard supplied by Restek or other acceptable retailers
2,4'-DDD standard supplied by Restek or other acceptable retailers
2,4'-DDT standard supplied by Restek or other acceptable retailers
Pesticide Surrogate Standard in acetone containing 0.5 µg/mL Decachlorobiphenyl and tetrachloro-m-xylene, and when appropriate, dibutylchlorendate.
Standard reference material NIST 1493
Standard reference material NIST 1942

4.2 Equipment and Supplies

1 L Graduated cylinder
1 L Separatory funnels
2.0 mL glass sample vials
10 mL Graduated cylinder
250 mL Erlenmeyer flask
Aluminum foil
Disposable pasteur pipette
Filter paper, Whatman 40
Gas-tight volumetric syringes, 250 µL, 500 µL, 100 µL and 1000 µL
Glass funnels
Glass pointy centrifuge tubes
Glass test tubes with teflon lined caps
Glass wool
Kuderna-Danish (K-D) apparatus
pH Paper
Water/steam bath (for K-D solvent evaporation)

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5.0 Methods and Procedures

Samples should be collected in 1 L glass jars with Teflon lined caps. Samples received for pesticides/PCB analysis must have a pH between 5 - 9 SU. If storage is required, samples are maintained at 4±2°C and may be held for up to 7 days. Sediment samples may be held for 14 days if maintained at 4±2°C (or up to one year if frozen). Tissue samples may be held for up to one year if frozen.

5.1 Extraction Procedure

Ascertain that all glassware is clean and dry. Thoroughly solvent rinse all glassware used in this procedure before proceeding with extractions.

5.1.1 Assemble a 1 L separatory funnel with stopcock and stopper, glass funnel with filter paper, and an Erlenmeyer flask for each sample. Add approximately 10 g of sodium sulfate to each filter paper lined funnel and rinse with methylene chloride or hexane.

5.1.2 Measure and add approximately 1 L of sample to separatory funnel. If a graduated cylinder is used, rinse cylinder with Milli-Q® water and then with methylene chloride or hexane before proceeding to the next sample.

5.1.3 Preparatory Blank and Laboratory Control Sample

Unless otherwise specified, Milli-Q® water will serve as the prep blank and LCS. For each blank and LCS, measure and add 1 L of Milli-Q® water to a separatory funnel.

5.1.4 Matrix Spike (MS) Samples

If sufficient sample volume is provided, an MS, MS duplicate, and MS triplicate (MS/MSD/MST) should be extracted with each batch. The sample used as the MS/MSD/MST will be the representative site water. For sediment analysis reference sediment will be used to perform all MS/MSD/MST samples. For tissue analysis pretest tissue will be used to perform all MS/MSD/MST samples.

5.1.5 Using a gas-tight volumetric syringe, add 100 µL or appropriate amount of Pest/PCB surrogate solution to each sample, including the blank, LCS and MSs. Extreme accuracy should be used when measuring and adding the surrogate and spike solutions. Rinse syringe with the appropriate solvent before and after use.

5.1.6 If samples are being analyzed for pesticides only, then add 375 µL of mix A, 375 µL of mix B, and 375 µL of mix C pesticide spiking solution to the LCS and MS/MSD/MST. See Appendix C for concentrations and compounds in each mix listed above. All spiked samples are spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix E.

If samples are being tested for PCBs only, then add 125 µL of 10 µg/mL Aroclor 1016 and 125 µL of 10 µg/mL Aroclor 1260 to the LCSs and MS/MSD, when

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relevant. See Appendix C for concentrations and compounds in each mix listed above.

5.1.7 Use pH paper to check the sample pH; the pH should be neutral (between 5 - 9 SU). Adjust pH if necessary using 1:1 sulfuric acid or 10% sodium hydroxide.

5.1.8 Add 60 mL of methylene chloride to each separatory funnel. Alternatively, hexane may be used if methylene chloride will extract too many interferences and acceptance criteria can still be met.

5.1.9 Shake and vent each separatory funnel under the hood for 2 minutes. Allow the organic layer to separate from the water phase for approximately 10 minutes. Swirl the separatory funnel several times to promote separation of the methylene chloride or hexane layer.

5.1.10 If an emulsion layer is not present, then drain the organic layer through the sodium sulfate filled funnel into the appropriate 250 mL Erlenmeyer flask.

If an emulsion layer is present, then drain into a labeled centrifuge bottle and centrifuge for 5 minutes. Pipette the bottom layer into the appropriate 250 mL Erlenmeyer flask. Return water layer to separatory funnel. Gentle sonication will also break an emulsion.

5.1.11 Repeat extraction (steps 5.1.8 to 5.1.10) twice more, then cover each flask with foil and set aside. Dispose of waste water by pouring into a solvent water waste container.

5.1.12 Assemble a Kuderna-Danish (K-D) apparatus with concentrator tube for each sample. Alternatively, a 60 mL pointy glass centrifuge tube may be used. The flasks are gently heated to evaporate the solvent down to 10 mL in a fume hood. The extract is placed into a K-D apparatus or centrifuge tube for final concentration to 1 mL.

5.1.13 Rinse the Erlenmeyer flask with methylene chloride or hexane and decant rinses into the K-D or centrifuge tube.

Note that if methylene chloride was used to extract, then the sample must be exchanged into hexane according to the following procedure:

Concentrate the extract to 2-3 mL then add 2 mL of hexane for the solvent exchange. Mix the extract well and continue to concentrate to 1 mL, or appropriate final volume. Allow the flask and funnel to completely dry in the fume hood before removal then transfer the 1mL extract to a screw cap vial for analysis. If the sample is not able to be concentrated to 1 mL, then measure the volume and adjust with hexane if necessary.

5.2 Pesticides - Florisil Column Cleanup

For samples requiring both Pesticides and PCBs use only 0.5 mL of extract for each clean up.

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5.2.1 Prepare a Florisil column for each extract by pushing a glass wool plug to the tip of the pipette. Add appromimately 500 mg of sodium sulfate and then fill the pipette with florisil to the 10 mL mark and tap the column to settle the powder. Add another small sodium sulfate plug, about 500 mg, to the top of the column.

5.2.2 Secure the column on a pipette stand in a fume hood. Rinse the column with approximately 15 mL $MeCl_2$ and then pre-elute the column with approximately 25 mL of hexane. Allow to drain into a flammable waste container; discard the solvent.

5.2.3 Transfer the sample extract to the top of the Florisil column using a gas tight syringe. Add 1 mL of hexane to the sample vial to rinse it then transfer rinse to the column; rinse twice with hexane.

5.2.4 Elute the column with 50 mL of $MeCl_2$. After the initial hexane rinsing do not allow the column to go dry during the entire cleanup procedure. Collect the eluate in a clean, labeled, $MeCl_2$ rinsed, glass test tube. Following the Florisil column clean up, reduce the extract to approximately 1.0 mL and exchange to hexane. Bring the extract to a final volume of 1.0 mL. The extract is now ready for analysis.

5.2.5 Transfer the extract to a vial. Extracts are checked by the supervisor and then stored in the organics extract refrigerator at 4±2°C until time of analysis.

5.3 PCB - Sulfuric Acid Cleanup

For samples requiring both Pesticides and PCBs, use only 0.5 mL of extract for each clean up.

Add 0.5 mL of concentrated sulfuric acid to the extract, cap and shake. Allow the extract to settle for at least one hour. The extract will separate into 2 distinct layers. Carefully pull off the top layer (the organic layer) using a gas tight syringe. Transfer to clean vial for analysis. Use care during transfer; any acid present in the extract for analysis will destroy the column.

6.0 Quality Control Requirements

Quality control elements may be added, removed, or altered to meet specific program or project requirements. Alternate acceptance criteria may be set for specific programs or projects. The objective reporting limits can be found in Appendix B. For DoD-specific projects see the QSM 5.1, as appropriate.

6.1 Demonstrations of Capability (DOC)

An analyst must complete a DOC before working independently on this procedure and yearly thereafter. A DOC is also required with any changes to instrument, method, or analyst. To perform a DOC, four replicates of a prepared quality control sample must be prepared. DOC samples must meet LCS acceptance criteria. For more information on DOCs see SOP QA-1108 “Training”.

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6.2 Method Detection Limit (MDL)

The MDL is calculated using the appropriate student's t-parameter value multiplied by the standard deviation of 8 or more replicates of spiked samples or standards. MDLs are performed annually and with major changes in instrument or method. The calculated MDL must be lower than the reporting limit.

The t-parameter value is from a one-tailed Student's t-Test at the 99% confidence interval using n-1 degrees of freedom, with "n" being the number of replicates.

The most recent MDL study can be found in the organics directory on the ESI network. For more information on MDLs see SOP QA-1111 "Determination of Method Detection and Reporting Limits".

6.3 Limit of Detection (LOD)

LOD determination will be conducted on a quarterly basis. For a single-analyte standard, the LOD is a single quality control sample that is approximately 2 to 3 times the MDL but no greater than the reporting limit. For a multi-analyte standard, the LOD is a single quality control sample that is approximately 1 to 4 times the MDL but no greater than the reporting limit.

The apparent signal to noise ratio at the LOD must be at least 3. For methods that do not produce a signal to noise, the LOD result must be at least 3 standard deviations greater than the mean method blank.

6.4 Limit of Quantitation (LOQ)

LOQ confirmation will be conducted on a quarterly basis. The LOQ is a single quality control sample no more than 1 to 2 times the reporting limit. The LOQ standard acceptance criteria is 30-150%. For more information on LODs and LOQs see SOP QA-1147 "Determination of LOD and LOQ Quality Control Samples".

6.5 Blank, LCS, MS samples, Standard Reference Material (SRM), and Relative Standard Deviation (RSD)

6.5.1 A blank, LCS, MS/MSD/MST, and SRM will be analyzed every batch, every 20 samples, or as provided in the prep method.

6.5.2 Acceptance criteria for all blanks is less than the reporting limit (see Appendix E).

6.5.3 Acceptance criteria for LCS recovery is 50-150%, unless method limits are more stringent (See Appendix A). The LCS will be spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix E.

6.5.4 Acceptance criteria for spiked sample recovery is 50-150%. MS/MSD/MST will be spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix E.

6.5.5 MS/MSD/MST samples are extracted either per client request or per project

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requirements. Every attempt will be made to obtain enough sample to perform a matrix spike with 5% frequency. If a single sample needs to be extracted and there is not enough sample volume for a matrix spike, a synthetic sample, or LCS duplicate may be extracted and analyzed to provide 5% spike occurrence. For aqueous analysis site water will be used to perform all MS/MSD/MST samples. For sediment analysis reference sediment will be used to perform all MS/MSD/MST samples. For tissue analysispretest tissue will be used to perform all MS/MSD/MST samples.

6.5.6 Acceptance criteria for surrogate standard recovery is 30-150%. All applicable surrogate standards must be within the acceptance range to be considered acceptable.

6.6 All quality control elements and samples must be spiked with a surrogate standard. Acceptance criteria for surrogate standard recovery is 30-150%. All applicable surrogate standards must be within the acceptance range to be considered acceptable.

6.7 Acceptance criteria for the initial calibration check recovery is 80-120%. Acceptance criteria for the continuing calibration check recovery is 80-120%.

6.8 Acceptance criteria for SRM recovery is 50-150%. An SRM will be evaluated for analytes at >3x the reporting limit. Reporting limits can be found in Appendix E.

6.9 Refer to EPA Method 608 for a complete list of potential interferences.

7.0 Calculations and Reporting

7.1 The following information should be recorded when performing an analysis of pesticides and PCBs in aqueous samples:

- Date
- Analyst's initial
- Sample ID, with dilution factor if applicable
- Batch
- Sample Code / Standard Code
- Solvent lot ID
- Surrogate ID and amount, with dilution factor if applicable
- Spike ID and amount , with dilution factor if applicable
- Internal Standard ID and amount
- Initial volume
- Final volume
- pH, pH strip ID
- Method
- Notes, if any

7.2 Calculations

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Calibrate the system software as per the Agilent manuals referenced in Section 3.0. Use the system software to calculate the pesticide/PCB values for QC elements and samples. Review the data to ensure it is correct and that all quality control requirements have been met. When needed, manually integrate the values according to SOP QA-1132 "Manual Chromatographic Peak Integration".

7.2.1 Calculate LCS/Surrogate Recovery as follows:

$$\frac{\text{LCS/Surrogate Concentration}}{\text{Concentration Added}} \times 100 = \%\text{Recovery}$$

7.2.2 Calculate %RSD as follows:

$$\frac{\text{Std}(\text{Result 1 - Result 2})}{\text{Avg}(\text{Result 1 - Result 2})} \times 100 = \%\text{RSD}$$

7.2.3 Calculate Spike Recovery as follows:

$$\frac{\text{Spiked Sample Concentration - Sample Concentration}}{\text{Concentration Added}} \times 100 = \%\text{Recovery}$$

8.0 Corrective Actions

8.1 Data Qualification

8.1.1 If DOCs, MDLs, LODs, or LOQs do not meet acceptability criteria, then repeat until acceptability criteria is met.

8.1.2 If samples analyzed are associated with blanks not meeting acceptance criteria, then they may be reanalyzed or reported with qualification (using the "B" qualifier).

8.1.3 If LCS samples do not meet percent recovery and/or RSD acceptance criteria, then they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (using the "J" qualifier).

8.1.4 If MS samples do not meet RSD acceptance criteria, then they may be reanalyzed or reported with qualification (using the "J" qualifier). If the sample result is less than 5 times the reporting limit, then the RSD does not need to be evaluated.

8.1.5 If the MS recovery is not within acceptance criteria, then samples may be reanalyzed or reported with qualification (using the "J" qualifier). If the sample result is greater than 5 times the spike amount, then the percent recovery need not be calculated.

8.1.6 If the surrogate recovery is not within the acceptable range, then the client is notified and the sample is re-extracted whenever possible. Sample matrix effect will be proven if the results obtained from the re-extracted sample show similar results as the first recovery. When this occurs, both results are reported with an explanation. If the results of the re-extracted sample are in the acceptable range, then there is a

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problem with the first extraction and only the compliant results are reported. If no additional sample can be obtained, then results are flagged in the report and a narrative is included qualifying the data.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified.

- Quality and expiration date of standards
- Cleanliness of containers, equipment, and labware
- Instrument operation

9.0 Health and Safety

9.1 Methylene chloride, pesticide standards, and PCB standards are extremely hazardous to everyone in the laboratory if handled improperly. It is important that all necessary safety precautions are followed. Gloves and safety glasses must be worn when handling samples, reagents, and standards. It is necessary to wear a lab coat to protect clothing. All solvent usage must occur in properly working fume hoods. Use precautions to prevent spills; if any spills do occur, clean up all spills immediately and notify supervisor.

9.2 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding Safety Data Sheets.

9.3 Strong acids and bases should be added to containers and solutions under the hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptance criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP, recording all data as described in Section 7.1, and reviewing all QC results for compliance with acceptance criteria.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities and DOC performance.

11.0 Waste Management and Pollution Prevention

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Unused non-hazardous sample may be disposed of as wastewater. Solvents and extracted sample not evaporated in a hood are collected and disposed of as hazardous waste in designated solvent water waste containers. Minimize solvent use. Prepare standards in amounts that will be used before expiration.

11.1 Aqueous Samples

After testing, aqueous samples will be disposed of through ESI's aqueous waste UV treatment system. This includes all unused samples, diluted samples and dilution waters that are not preserved. Access points for aqueous waste disposal in compliance with state and federal regulations are located throughout the laboratory and include streams for acidified samples, alkaline samples, and unpreserved samples. Any samples found to be toxic may require further testing or treatment in order to determine proper disposal technique to assure compliance with all local, state and federal regulations.

11.2 Hazardous Waste Samples

Hazardous waste samples must be handled by properly trained employees. Only the ESI hazardous waste coordinator may train ESI employees on handling hazardous waste. There are 3 main hazardous waste streams at ESI, “dilute metals acidic”, “solvents” and “Lachat wastes”. When a satellite container needs to be emptied, it is properly transported to the waste shed and added into the appropriately labeled container for that waste stream. The volume and type of waste is recorded in the appropriate log book. Shipping manifests reflect when the waste drums are picked up for transport.

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Appendix A. Quality Control Acceptance Criteria - Method 608[1]

Table 3—QC Acceptance Criteria—Method 608

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s, %
Aldrin	2.0	0.42	1.08 - 2.24	42 - 122
α-BHC	2.0	0.48	0.98 - 2.44	37 - 134
β-BHC	2.0	0.64	0.78 - 2.60	17- 147
δ-BHC	2.0	0.72	1.01 - 2.37	19 - 140
γ-BHC	2.0	0.46	0.86 - 2.32	32 - 127
Chlordane	50	10.0	27.6 - 54.3	45 - 119
4,4'-DDD	10	2.8	4.8 - 12.6	31 - 141
4,4'-DDE	2.0	0.55	1.08 - 2.60	30 - 145
4,4'-DDT	10	3.6	4.6 - 13.7	25 - 160
Dieldrin	2.0	0.76	1.15 - 2.49	36 - 146
Endosulfan I	2.0	0.49	1.14 - 2.82	45 - 153
Endosulfan II	10	6.1	2.2 - 17.1	D - 202
Endosulfan Sulfate	10	2.7	3.8 - 13.2	26 - 144
Endrin	10	3.7	5.1 - 12.6	30 - 147
Heptachlor	2.0	0.40	0.86 - 2.00	34 - 111
Heptachlor epoxide	2.0	0.41	1.13 - 2.63	37 - 142
Toxaphene	50.0	12.7	27.8 - 55.6	41 - 126
PCB-1016	50	10.0	30.5 - 51.5	50 - 114
PCB-1221	50	24.4	22.1 - 75.2	15 - 178
PCB-1232	50	17.9	14.0 - 98.5	10 - 215
PCB-1242	50	12.2	24.8 - 69.6	39 - 150
PCB-1248	50	15.9	29.0 - 70.2	38 - 158
PCB-1254	50	13.8	22.2 - 57.9	29 - 131
PCB-1260	50	10.4	18.7 - 54.9	8 - 127

s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, P_s = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

[1] Original table from US EPA Appendix A: Method 608, *Organochlorine Pesticide and PCBs*.

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Appendix B. Objective Reporting Limits

Compound	µg/L	µg/Kg	Compound	µg/L	µg/Kg
Aroclor 1016	0.05	2.5	hexachlorobenzene	0.002	1
Aroclor 1242	0.05	2.5	alpha-BHC	0.002	1
Aroclor 1221	0.05	2.5	gamma-BHC (Lindane)	0.002	1
Aroclor 1232	0.05	2.5	beta-BHC	0.002	1
Aroclor 1248	0.05	2.5	delta-BHC	0.002	1
Aroclor 1254	0.05	2.5	heptachlor	0.01	1
Aroclor 1260	0.05	2.5	aldrin	0.4	1
			oxychlordane	0.002	1
			chlorpyrifos	0.002	1
			heptachlor epoxide	0.01	1
			gamma-chlordane	0.002	1
			trans-nonachlor	0.01	1
			alpha-chlordane	0.03	1
			endosulfan I	0.01	1
			4,4'-DDE	4	1
			dieldrin	0.2	1
			endrin	0.004	1
			cis-nonachlor	0.002	1
			4,4'-DDD	0.010	1
			endosulfan II	0.01	1
			toxaphene	0.1	1
			4,4'-DDT	0.04	1
			endrin aldehyde	0.004	1
			endosulfan sulfate	0.01	1
			methoxychlor	0.02	1
			endrin ketone	0.004	1
			2,4-DDE	0.01	1
			2,4-DDD	0.01	1
			2,4-DDT	0.01	1

Appendix C. Pesticide Standard Mixes

Pesticide Standard Mix A		Pesticide Standard Mix B		Pesticide Standard Mix C	
TCMX (SS)	0.08 µg/mL	TCMX (SS)	0.08 µg/mL	oxychlordane	0.08 µg/mL
alpha-BHC	0.08 µg/mL	beta-BHC	0.08 µg/mL	chlorpyrifos	0.08 µg/mL
gamma-BHC (Lindane)	0.08 µg/mL	delta-BHC	0.08 µg/mL	trans-nonachlor	0.08 µg/mL
heptachlor	0.08 µg/mL	aldrin	0.08 µg/mL	cis-nonachlor	0.08 µg/mL
endosulfan I	0.08 µg/mL	heptachlor epoxide	0.08 µg/mL	hexachlorobenzene	0.08 µg/mL
dieldrin	0.16 µg/mL	gamma-chlordane	0.08 µg/mL		
endrin	0.16 µg/mL	alpha-chlordane	0.08 µg/mL		
4,4'-DDD	0.16 µg/mL	4,4'-DDE	0.16 µg/mL		
4,4'-DDT	0.16 µg/mL	endosulfan II	0.16 µg/mL		
methoxychlor	0.8 µg/mL	endrin aldehyde	0.16 µg/mL		
209 (SS)	0.16 µg/mL	endosulfan sulfate	0.16 µg/mL		
		endrin ketone	0.16 µg/mL		
		209 (SS)	0.16 µg/mL		

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Additional Compound Mix	
2,4'-DDD	0.16 µg/mL
2,4'-DDE	0.16 µg/mL
2,4'-DDT	0.16 µg/mL

Appendix D. Standard Preparation Recipes

Pesticides			PCB Aroclor		
Initial Volume: 1000mL			Initial Volume: 1000mL		
Final Volume: 1.0 mL			Final Volume: 1.0 mL		
Solvent:Hexane			Solvent: Hexane		
Surrogate - 100 µL			Surrogate - 100 µL		
Tetrachloro-m-xylene	0.5	µg/mL	Tetrachloro-m-xylene	0.5	µg/mL
Decachlorobiphenyl	0.5	µg/mL	Decachlorobiphenyl	0.5	µg/mL
Pesticide A Mix - 375 µL			Spike - 125 µL		
TCMX (SS)	0.08	µg/mL	Aroclor 1016	10	µg/mL
alpha-BHC	0.08	µg/mL	Aroclor 1260	10	µg/mL
gamma-BHC (Lindane)	0.08	µg/mL			
heptachlor	0.08	µg/mL			
endosulfan I	0.08	µg/mL			
dieldrin	0.16	µg/mL			
endrin	0.16	µg/mL			
4,4'-DDD	0.16	µg/mL			
4,4'-DDT	0.16	µg/mL			
methoxychlor	0.8	µg/mL			
209 (SS)	0.16	µg/mL			
Pesticide B Mix - 375 µL					
TCMX (SS)	0.08	µg/mL			
beta-BHC	0.08	µg/mL			
delta-BHC	0.08	µg/mL			
aldrin	0.08	µg/mL			
heptachlor epoxide	0.08	µg/mL			
gamma-chlordane	0.08	µg/mL			
alpha-chlordane	0.08	µg/mL			
4,4'-DDE	0.16	µg/mL			
endosulfan II	0.16	µg/mL			
endrin aldehyde	0.16	µg/mL			
endosulfan sulfate	0.16	µg/mL			
endrin ketone	0.16	µg/mL			
209 (SS)	0.16	µg/mL			
Pesticide C Mix - 375 µL					
oxychlordane	0.08	µg/mL			
chlorpyrifos	0.08	µg/mL			
trans-nonachlor	0.08	µg/mL			
cis-nonachlor	0.08	µg/mL			
hexachlorobenzene	0.08	µg/mL			
Additional Compound Mix - 375 µL					

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2,4'-DDD	0.16	µg/mL
2,4'-DDE	0.16	µg/mL
2,4'-DDT	0.16	µg/mL

Part 1
(see above for compounds in each standard)

Pesticide Surrogate Mix - 0.5 µg/mL
- Stock Standard: Supelco, Cat# 861284 (200 µg/mL)
- Preparation: Dilute 0.075 mL stock standard to final volume 30 mL with hexane

Pesticide Mix A - 80/160 ng/mL
- Stock Standard: Supelco, Cat# 47977 (0.5-5.0 µg/mL)
- Preparation: Dilute 1.0 mL stock standard to final volume 6.25 mL with hexane

Pesticide Mix B - 80/160 ng/mL
- Stock Standard: Supelco, Cat# 47977 (0.5-1.0 µg/mL)
- Preparation: Dilute 1.0 mL stock standard to final volume 6.25 mL with hexane

Pesticide Mix C - 80 ng/mL
- Stock Standard: Individual component (see list in Appendix A), Crescent Chemical Company (100 µg/mL)
- Preparation: Dilute 0.2 mL each stock standard to final volume 2.0 mL with hexane

Additional Compound Mix - 160 ng/mL
- Stock Standard: Individual component (see list in Appendix A), Restek (1000 µg/mL)
- Preparation: Dilute 1.0 µL each stock standard to final volume 6.25 mL with hexane

Part 2
(see above for compounds in each standard)

Congener Surrogate - 2 µg/mL
- Stock Standard: Ultra Scientific, Cat# RPC-075AS (100 µg/mL)
- Preparation: Dilute 0.6 mL stock standard to final volume 30 mL with 50:50 hexane:acetone

Congener Spike - 4 µg/mL
- Stock Standard:
  - Ultra Scientific, Cat# RPC-EPA-2-1, RPC-030AS, RPC-073AS, RPC-099AS, RPC-075AS and RPC-168AS (100 µg/mL)
- Preparation: Dilute 0.15 mL of each stock standard to final volume 3.75 mL with hexane

Part 3
(see above for compounds in each standard)

PAH Surrogates - 10 µg/mL
- Stock Standards:
  - 2-Fluorobiphenyl: Chemservice, Cat #N-10359-16 (Neat)
  - O-terphenyl: Chemservice, Cat# S-12693X4-1ML (1000 µg/mL)
- Preparation - Intermediate Stock Standards:
  - 2-Fluorobiphenyl: 50 mg stock standard to final volume 25 mLwith $MeCl_2$ (2000 µg/mL)
  - O-terphenyl: 1.0 mL stock standard to final volume 10 mL with acetone (200 µg/mL)
- Preparation - Second Intermediate Stock Standards:
  - 2-Fluorobiphenyl: Dilute 1 mL intermediate standard to final volume 10 mL with acetone (200 µg/mL)
  - 2-Fluorobiphenyl: Dilute 2.0 mL second intermediate standard to final volume 40 mL with acetone (10 µg/mL)
  - O-terphenyl: Dilute intermediate standard to final volume 40 mL with acetone (10 µg/mL)

PAH Spike Mix - 10 µg/mL
- Stock Standard: Environmental Consulting and Supplies, Inc., Cat# ECS-A-032, ECS-B-032, ECS-N-032 (2000 µg/mL)
- Preparation - Intermediate Stock Standards:
  - Dilute 2 mL of stock standard to final volume 40 mL acetone (100 µg/mL)
  - Dilute 1.0 mL of intermediate stock standard to final volume 10 mL acetone

Part 4

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(see above for compounds in each standard)

SVOC Surrogate - 100-200 µg/mL
- Stock Standard: ECS, Cat# ECS-N-027 (1000-2000 µg/mL)
- Preparation: Dilute 2 mL stock standard to final volume 20 mL with acetone

SVOC Spike Mixes - 100-200 µg/mL
- Stock Standards:
  - Acid Mix: ECS, Cat #ECS-N-004 (2000 µg/mL)
  - Base/Neutral Mix: ECS, Cat# ECS-N-005 (1000 µg/mL)
  - PAH Mix: ECS, Cat# ECS-N-032 (2000 µg/mL)
- Preparations:
  - Acid Mix: Dilute 2.0 mL stock standard to 20 mL with acetone (200 µg/mL)
  - Base/Neutral Mix: Dilute 2.0 mL stock standard to final volume 20 mL with acetone (100 µg/mL)
  - PAH Mix: Dilute 2.0 mL stock standard to final volume 40 mL with acetone (100 µg/mL)

Part 5
(See above for compounds in each standard)

PCB Surrogate Mix - 0.5 µg/mL
- Stock Standard: Supelco, Cat# 861284 (200 µg/mL)
- Preparation: Dilute 0.075 mL stock standard to final volume 30 mL with hexane

PCB Aroclor Spike Mix - 10 µg/mL
- Stock Standards:
  - Aroclor 1016: Ultra Scientific, Cat#PP-282-1 100 µg/mL
- Aroclor 1260: Ultra Scientific, Cat#PP-362-1 100 µg/mL
- Preparations:
  - Aroclor 1016: Dilute 1.0 mL stock standard to final volume 10.0 mL with hexane
  - Aroclor 1260: Dilute 1.0 mL stock standard to final volume 10.0 mL with hexane

Appendix E. Analyte List and Target Reporting Limits

Analyte	Tissue (ng/g)	Sediment (ng/g)	Water (µg/L)
Aldrin	4.3	1	0.4
Dieldrin	1.5	1	0.20
a-Chlordane	2.7	1	0.030
Trans Nonachlor	2.7	1	0.010
Heptachlor	2.7	1	0.010
Heptachlor Epoxide	2.7	1	0.010
Endosulfan I	0.32	1	0.010
Endosulfan II	0.32	1	0.010
Endosulfan Sulfate	0.32	1	0.010
4,4-DDT	1	1	0.04
2,4-DDT	1	1	0.010

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4,4-DDD	1	1	0.010
2,4-DDD	1	1	0.010
4,4-DDE	1	1	4.0
2,4-DDE	1	1	0.010

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TITLE: Analysis of Pesticides and PCBs in Extracts of Soil, Tissue and Water - Region II Testing

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	SOP Preparation	A. MacKinnon	01/2018

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1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) details the procedure for the analysis of soil, tissue and water extracts for pesticides and polychlorinated biphenyls (PCBs) using US EPA method 608 or method 8081, as specified by the project. This protocol is designed for use with samples from US EPA Region II and the New York District ACOE. Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD). Once the GC run is complete, the information is integrated and the data is analyzed and calculated. Detection limits achievable by this method are listed in Appendix 1.

This procedure is applicable to the following compounds: aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, alpha-chlordane, 4,4'-DDT, 4,4-DDE, 4,4'-DDD, 2,4'-DDT, 2,4'-DDD, 2,4'-DDE, dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, heptachlor epoxide, PCB-1242, PCB-1254, PCB-1221, PCB-1232, PCB-1248, PCB-1260, PCB-1016, toxaphene, endrin ketone, oxychlordane, chlorpyrifos, hexachlorobenzene, cis-nonachlor, trans-nonachlor, and methoxychlor.

2.0 Definitions

See ESI's Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Revision 2. Method 8081B (SW-846).

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4.0 Materials and Apparatus

4.1 Reagents and Standards

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable logbook), and expiration date.

Evaluation mix, containing DDT and endrin for checking column breakdown
Hexane, pesticide quality or equivalent for diluting samples and standards
Individual standards of aroclors (1016, 1221, 1242, 1232, 1248,1254, 1260), toxaphene and chlordane supplied by Ultra Scientific or other commercial retailers
Pesticide standard mix A and mix B supplied by Supelco or other acceptable retailers
Pesticide standard mix C, project specific
Surrogate mix Tetrachloro-m-xylene and decachlorobiphenyl 200 µg/mL in acetone dibutylchlorendate
Standard reference material NIST 1492

4.2 Equipment and Supplies

Analytical balance for weighing standards and solids
Class A volumetric flasks as required
Confirmation column: RTx-1701 30M x 0.32mm id
HP 6890 with column, ECD and HP data system, or equivalent
HP vials for standards and samples
Refrigerator for storage of standards and extracts
Syringes, as required for dilutions
Suggested column for ECD: RTx-35 30M x 0.32mm id

5.0 Methods and Procedures

5.1 Method Summary

Extracts are set up on an autosampler for the Gas Chromatograph (GC). The method 8080 is loaded into the software. This has all of the temperature setting requirements preprogrammed to allow for an easy and accurate analysis. The information is logged into the computer software sample table and into the GC-ECD run log. Once the GC run is complete, the information is integrated and the data is analyzed and calculated.

5.2 Analytical Standards and Surrogate Preparation

Standards are stored at $\leq 4^{\circ}C$ and should be kept in the dark. They are stable for 6 to 12 months. Standard expiration date may be extended up to 6 months if it can be demonstrated that no breakdown has occurred. Record standards and surrogates preparation in the appropriate standards log. Surrogates are valid indefinitely as long as the GC laboratory surrogate is prepared within 6 months of use.

5.2.1 Pesticide Working Standards

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Prepare pesticide working standards by diluting the appropriate stock standards, which includes: the commercial pesticide standard mixes (A and B) and when needed, pesticide mix C. Dilutions are made in hexane to achieve the desired concentrations. See Appendix 1: Part A for detailed preparation instructions.

5.2.2 Multi-Component Pesticides Working Standards

If detected in a sample, it may be necessary to prepare a single or five point calibration curve for toxaphene and/or chlordane. Prepare the multi-component pesticides working standards by diluting chlordane or toxaphene stock standard to achieve the desired concentrations. See Appendix 1: Parts E, F, G and H for detailed preparation instructions.

5.2.3 PCB Working Standards

Prepare PCB working standards by diluting aroclors 1016 and 1260 stock into hexane to achieve the desired concentrations. See Appendix 1: Parts C and D for detailed preparation instructions.

5.2.4 Surrogate Preparation

Surrogate solution is prepared from a commercially obtained surrogate mix containing tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (209). The surrogate mix is added to all samples and QC samples. Note that the pesticide standard mixes A and B contain both TCMX and 209. Surrogate calibration may be performed using the pesticide mixes or the surrogate solution. Dibutylchlorendate may also be used as a surrogate compound where appropriate.

The most surrogate solution (TCMX and 209) is prepared at 0.5 µg/mL by diluting 75 µL of the 200 µg/mL stock standard to a final volume of 30 mL with hexane.

5.2.5 Standards applicable to this SOP can be found in Appendix 2.

5.3 Calibration Procedure - Pesticide and PCB Analysis

Column performance must be demonstrated by using an evaluation mix that allows the calculation of DDT and Endrin breakdown. The breakdown of either compound must be <15%. If this criteria cannot be achieved, then instrument maintenance must be performed. Calculating the breakdown of DDT and endrin is not required when analyzing PCBs.

Initial multi-point calibration is performed to determine the instrument response factors over the usable concentration range. When the compound response is linear over the calibration range (RSD of response factors within limit of <20%) the average response factor may be used to calculate target analyte concentrations.

Each day that samples are to be analyzed, a Continuing Calibration Verification (CCV) standard is analyzed. The CCV standard response is evaluated for percent (%) recovery relative to initial calibration response. If acceptance criteria are met, the initial calibration response may be used for quantitation, otherwise a new initial calibration is obtained or

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sample values are qualified.

Response for multi-component analytes such as technical chlordane and toxaphene is obtained from a single point calibration. When tentatively identified in a sample, a multipoint calibration bracketing the sample concentration should be used.

See the instrument logbook for the current column and condition for the instrument in use. Typical conditions for an ECD are:

Column: 30M x 0.32mm ID CLP2
Column flow: 10 mL/min He
Makeup flow: 60 mL/min Ar/Me
Run time: 29 min
Injector temp: 200
Detector temp: 300
Oven temp: 160°(0) -5°/min - 260°(9)
Injection size: 1-3 µL

5.4 Initial Calibration

5.4.1 Pesticides

A breakdown evaluation mix is run; see section 6.8 for acceptance limits. Once the evaluation mix passes, a five point calibration is performed by analyzing the working standards detailed in Appendix 1. See section 6.8 for acceptance criteria. A single point toxaphene standard, and when needed a single point technical chlordane, is also analyzed after the five point calibration.

5.4.2 PCBs

A five point calibration may be performed by analyzing the aroclor 1016/1260 calibration standards detailed in Appendix 1. A linear calibration model should be applied to the five point calibration. This is followed by a single point of each of the remaining aroclors. The linear response for aroclors 1016 and 1260 may be applied to the other aroclors. Alternatively, a five point calibration may be performed on other aroclors for specific project objectives.

Three to five peaks are chosen to quantitate each aroclor. One of the peaks selected must be unique to that aroclor and all peaks must be characteristic of it. The peaks selected should be no less than 25% of the height of the largest aroclor peak.

5.4.3 Multi-Component Pesticides

When needed, a five point calibration for toxaphene and or chlordane may be obtained by analyzing the working standards detailed in Appendix 1, Parts E and G. The average response factor will be applied. See section 6.8 for acceptance criteria. For quantitation of toxaphene and chlordane, 3-5 characteristic peaks and or the total area are used.

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5.5 Initial Calibration Blank (ICB) and Initial Calibration Verification (ICV)

5.5.1 Pesticides

Following a five point calibration, a calibration verification standard containing all compounds in the initial calibration from a source (vendor or lot number) different from that used to make the initial calibration is analyzed. This should be prepared at a concentration near the midpoint of the calibration curve. An instrument blank is analyzed following calibration verification standard. Refer to Appendix 1: Part B for detailed preparation instructions.

5.5.2 PCBs

Following a five point calibration, a calibration verification standard, containing the aroclors used in the 5 point initial calibration, near the midpoint of the curve is to be analyzed. This standard must be made from a source (vendor or lot number) different from that used to make the initial calibration. An instrument blank is analyzed run following calibration verification standard. Refer to Appendix 1: Part D for detailed preparation instructions.

5.5.3 Multi-Component Pesticides

Following the five point calibration, a calibration verification standard, containing chlordane or toxaphene, near the midpoint of the curve is to be analyzed. This standard must be made from a source (vendor or lot number) different from that used to make the initial calibration. An instrument blank is analyzed run following calibration verification standard. Refer to Appendix 1: Part F or H for detailed preparation instructions.

5.6 Sample Analysis

After initial calibration, up to ten samples are analyzed before calibration verification. If a target pesticide or PCB is identified above the calibration limit, the sample must be diluted and rerun so that the response is within the calibration range. Surrogate recoveries for water and solid samples outside 30-150% or interferences present which prevent quantitation require client notification, the re-extraction of the sample and the possible qualification of the sample result.

5.7 Continuing Calibrations

Every ten samples, or the middle of a batch of fewer than 20 samples, a mid-range continuing calibration standard is analyzed. Response for each pesticide of interest must have a recovery of plus or minus 20% of that for intial calibration. For PCBs, the average response for the 5 peaks for aroclors 1016 and 1260 in the continuing calibration must be within 20% of the initial calibration. Failure requires performing instrument or column maintenance that results in a passing continuing calibration verification or a new initial calibration.

6.0 Quality Control Requirements

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Quality control elements may be added, removed, or altered to meet specific program or project requirements. Acceptance criteria shall be updated based on the associated control charts once sufficient data points have been collected (or atleast 20 data points) and yearly thereafter. Alternate acceptance criteria may be set for specific programs or projects. The objective reporting limits can be found in Appendix B of SOP QA-1344. The most recent MDL study can be found in the pesticide directory. For DoD-specific projects see the QSM 5.1, as appropriate. All quality control elements and samples must be spiked with a surrogate standard.

6.1 Demonstrations of Capability (DOC)

An analyst must complete a DOC before working independently on this procedure and yearly thereafter. A DOC is also required with any changes to instrument, method, or analyst. To perform a DOC, four replicates of a prepared quality control sample must be prepared. DOC samples must meet LCS acceptance criteria. For more information on DOCs see SOP QA-1108 “Training”.

6.2 Method Detection Limit (MDL)

The MDL is calculated using the appropriate student’s t-parameter value multiplied by the standard deviation of 8 or more replicates of spiked samples or standards. MDLs are performed annually and with major changes in instrument or method. The calculated MDL must be lower than the reporting limit.

The t-parameter value is from a one-tailed Student’s t-Test at the 99% confidence interval using n-1 degrees of freedom, with “n” being the number of replicates.

The most recent MDL study can be found in the organics quality control directory on the ESI network. For more information on MDLs see SOP QA-1111 “Determination of Method Detection and Reporting Limits”.

6.3 Limit of Detection (LOD)

LOD determination will be conducted on a quarterly basis. For a single-analyte standard, the LOD is a single quality control sample that is approximately 2 to 3 times the MDL but no greater than the reporting limit. For a multi-analyte standard, the LOD is a single quality control sample that is approximately 1 to 4 times the MDL but no greater than the reporting limit.

The apparent signal to noise ratio at the LOD must be at least 3. For methods that do not produce a signal to noise, the LOD result must be at least 3 standard deviations greater than the mean method blank.

The LOD verification will be performed quarterly for pesticides and aroclors 1016 and 1260. LOD verification of the remaining aroclors (1221, 1232, 1242, 1248 and 1254) will be performed for DoD projects on a per batch basis. If a significant volume of work or a long term project is expected, LOD verification of the remaining aroclors may be performed on a quarterly basis.

6.4 Limit of Quantitation (LOQ)

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LOQ confirmation will be conducted on a quarterly basis. The LOQ is a single quality control sample no more than 1 to 2 times the reporting limit. The LOQ standard acceptance criteria is 30-150%.

The LOQ confirmation will be performed quarterly for pesticides and aroclors 1016 and 1260. LOQ confirmation of the remaining aroclors (1221, 1232, 1242, 1248 and 1254) will be performed for DoD projects on a per batch basis. If a significant volume of work or a long term project is expected, LOQ verification of the remaining aroclors may be performed on a quarterly basis.

For more information on LODs and LOQs see SOP QA-1147 “Determination of LOD and LOQ Quality Control Samples”.

6.5 Blank, LCS, Matrix Spike (MS) Sample, Relative Standard Deviation (RSD), Standard Reference Material (SRM)

6.5.1 A blank, LCS, MS, MS duplicate (MSD), and MS triplicate (MST) samples will be analyzed every batch, every 20 samples, or as provided in the prep method. An SRM will be analyzed every batch, every 20 samples, or as provided in the prep method for aqueous and sediment matrices.

6.5.2 Acceptance criteria for all blanks is less than the reporting limit (see Appendix 3).

6.5.3 Acceptance criteria for LCS recovery is 50-150%, unless method limits are more stringent (See SOP QA-1344, Appendix A). LCS are spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix 3.

6.5.4 Acceptance criteria for spiked sample recovery is 50-150%. MS/MSD/MST are spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix 3.

6.5.5 MS/MSD/MST samples are extracted either per client request or per project requirements. Every attempt will be made to obtain enough sample to perform a matrix spike with 5% frequency. If a single sample needs to be extracted and there is not enough sample volume for a matrix spike, a synthetic sample, or LCS duplicate may be extracted and analyzed to provide 5% spike occurrence. For aqueous analysis site water will be used to perform all MS/MSD/MST samples. For sediment analysis reference sediment will be used to perform all MS/MSD/MST samples. For tissue analysispretest tissue will be used to perform all MS/MSD/MST samples.

6.5.6 Acceptable criteria for SRM recovery is 50-150%. SRMs are evaluated for analytes at >3x the reporting limit. Reporting limits can be found in Appendix 3.

6.6 Calibration, Calibration Verifications and Blanks

6.6.1 Acceptance criteria for in initial calibration is a relative standard deviation (RSD) for response factors less than 20%.

6.6.2 Acceptance criteria for the ICV standard is a recovery of ±20%.

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6.6.3 Acceptance criteria for the CCV standard is a recovery of ±20%.

6.6.4 Acceptance criteria for the ICB and CCB is less than the reporting limit. Reporting limits can be found in Appendix 3.

6.7 Acceptance criteria for surrogate standard recovery is 30-150%. All applicable surrogate standards must be within the acceptance range to be considered acceptable. If the surrogate recoverys fall outside of the acceptable range the client must be notified and the sample must be re extracted.

6.8 There must be an evaluation of DDT and endrin for column breakdown for pesticide analysis at least every 12 hours. DDT and endrin breakdown must be <15% for either compound.

6.9 Refer to EPA Method 608 for a complete list of potential interferences.

7.0 Calculations and Reporting

7.1 The following information should be recorded when performing an analysis of pesticides and PCBs:

- Date
- Analyst's initial
- Batch
- Calibration ID and recipe
- Sample ID / Standard ID, with dilution factor if applicable
- Data file name
- Acquisition method
- Notes, if any

7.2 Identification of Pesticides and PCBs

7.2.1 Pesticides

A pesticide is considered a potential hit if the peak falls within the retention time window for the analyte on the column in use and the concentration is greater than half the detection limit.

Use confirmation techniques when doubt exists over the identification of a peak on the chromatogram. A potentially doubtful pesticide hit must be confirmed by analyzing a second column with a different phase or by GC/MS. The analysis may be performed qualitatively using a single point calibration. If a second column confirmation produces a result that has a percent difference of >40%, then the chromatograms must be reviewed for potential problems (co-eluting peaks, baseline issues, etc.). If both chromatograms are determined to be valid, then the higher result should be reported.

7.2.2 Multi-Component Pesticides

When analysis for toxaphene or chlordane is performed, the chromatogram is first

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inspected for the presence of the multi-component pesticides. If the analyst determines that the pattern for the multi-component pesticide is recognized, then 5 to 6 peaks are selected, or total area, and used for quantitation. Quantitation may be done from a single point standard or from a multi point calibration when required by the project plan. When a single point is utilized then the result will be qualified as being quantitated against a single standard.

7.2.3 PCBs

An aroclor is considered a potential hit if the peaks fall within the retention time window for the analyte on the column in use. Use confirmation techniques when doubt exists over the identification of an aroclor peak on the chromatogram. A potentially doubtful aroclor hit must be confirmed by analyzing a second column with a different phase or by GC/MS. The analysis may be performed qualitatively using a single point calibration. If a second column confirmation produces a result that has a percent difference of >40%, then the chromatograms must be reviewed for potential problems (co-eluting peaks, baseline issues, etc.). If both chromatograms are determined to be valid, then the higher result should be reported.

Absolute retention times may be determined following OLMO1.8 SOW page D-47-48 during initial calibration. Analysts' judgement should always be used in peak identification and rejecting false positives. When needed, the sample chromatogram should be compared to the individual aroclor standards to aid in pattern recognition and aroclor identification.

7.3 Sample Calculations

7.3.1 The quantitation of aroclors is dependent upon which type is present. Quantitation of aroclor 1016 and 1260 is accomplished by averaging the concentrations from 5 major peaks of the aroclor in the sample which were calculated through a 5-point calibrated method. For the remaining aroclors, the sample is quantitated against a 1-point calibrated method. If more than one aroclor is observed in a sample, a peak common to both analytes must not be used to quantitate either compound. The result is combined with extraction lab data to generate the final report. The final report is inspected by a lab analyst or lab supervisor for errors, additions (if any) are forwarded for final review by the lab director.

Round the result to 2 significant figures after all calculations are complete.

7.3.2 Calculate the response of an analyte as peak areas using the quadratic equation as follows:

$B2X2 + B1X + B0 = R$

Where:

X = Concentration of analyte (µg/mL)
R = Response of analyte as peak area

Verify R by entering the appropriate values for B2, B1, B0, which are obtained by

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listing the LAS initial calibration method, and for X, which is the concentration value from the chromatogram.

7.3.3 Calculate the concentration of an analyte in a sample using the single point equation as follows:

$$Y = R2 * RF$$

Where:

RF(Response Factor) = concentration of analyte in standard/R1
Y = Concentration of analyte (µg/mL) in sample
R2 = Response of analyte as peak area in sample
R1 = Response of analyte as peak area in standard

7.3.4 Calculate the concentration of liquids as follows:
Concentration (µg/L) =

$$\frac{\text{(Concentration in µg/mL)*(Intermediate Volume)*(Final Volume)*(Dilution Factor)}}{\text{(Volume Extracted in L)*(Aliquot)}}$$

7.3.5 Calculate the concentration of solids as follows:

Concentration (µg/g) =

$$\frac{\text{(Concentration in µg/mL)*(Intermediate Volume)*(Final Volume)*(Dilution Factor)}}{\text{(Weight extracted in g)*(\% Dry Weight)*(Aliquot)}}$$

7.3.6 Calculate the Standard Recovery as follows:

$$\frac{\text{Standard Concentration}}{\text{Concentration Added}} * 100 = \%\ \text{Recovery}$$

7.3.7 Use the following equation to calculate the %RSD:

$$\frac{\text{Std(Result 1 - Result 2)}}{\text{Avg(Result 1 - Result 2)}} * 100 = \%RSD$$

7.3.8 Calculate the Sample Spike Recovery as follows:

$$\frac{\text{Spike Concentration - Sample Concentration}}{\text{Concentration added to Spike}} * 100 = \%\text{Recovery}$$

8.0 Corrective Actions

8.1 Data Qualification

All QC measures have associated acceptance windows. A failure of any of these measures

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may necessitate corrective action. The action may be to correct performance of a specific technique, or to repair a hardware defect. Affected samples should be re-analyzed or re-extracted first if necessary and then re-analyzed. Data that is unavoidably compromised and cannot be regenerated must be flagged. In such cases, the client is contacted promptly for more sample.

8.1.1 If DOCs, MDLs, LODs, or LOQs do not meet acceptability criteria, then repeat until acceptability criteria is met.

8.1.2 If the DDT/endrin breakdown check does not meet the criteria, then the standard must be reanalyzed. Alternatively, system maintenance may be required.

8.1.3 If the LCS sample does not meet percent recovery acceptance criteria, then they should be re-analyzed along with all associated QC and samples. Samples may be reported with qualification (using the “J” qualifier).

8.1.4 If samples analyzed are associated with blanks not meeting acceptance criteria, then they may be reanalyzed or reported with qualification (using the “B” qualifier). If the analyte detected in the blank, is not found in the sample, no qualification is needed. If the analyte detected in the blank is found in the sample <10 times the blank value, must be qualified. If an analyte is detected in the blank at >5 times the reporting limit, stop analysis and determine source of contamination.

8.1.5 If MS samples analyzed in duplicate do not meet percent recovery acceptance criteria, then they may be reanalyzed or reported with qualification (using the “J” qualifier). If the sample result or sample duplicate result is less than 5 times the reporting limit, then the RSD does not need to be evaluated.

8.1.6 If the MS recovery is not within acceptance criteria, then samples may be reanalyzed or reported with qualification (using the “J” qualifier). If the sample result is greater than 5 times the spike amount, then the percent recovery need not be calculated.

8.1.7 If the initial calibration curve is not within acceptance criteria, then recalibrate or qualify (using the “J” qualifier) any positive sample values as estimated.

8.1.8 If the ICV does not meet acceptance criteria, then prepare a new curve and ICV or qualify (using the “J” qualifier) any positive sample values as estimated.

8.1.9 If a CCV does not meet acceptance criteria, then all associated samples must be reanalyzed or qualified (using the “J” qualifier) as estimated.

8.1.10 If the surrogate recovery is not within the acceptable range, then the client is notified and the sample is re-extracted whenever possible. Sample matrix effect will be proven if the results obtained from the re-extracted sample show similar results as the first recovery. When this occurs, both results are reported with an explanation. If the results of the re-extracted sample are in the acceptable range, then there is a problem with the first extraction and only the compliant results are reported. If no additional sample can be obtained, then results are flagged in the report and a narrative is included qualifying the data.

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8.1.11 If a result is reported and the difference between the results from each column was greater than 40%, then the data must be qualified to make the client aware of the issue.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified.

- Quality and expiration date of standards
- Cleanliness of containers, equipment, and labware
- Instrument operation

9.0 Health and Safety

9.1 Pesticide standards, and PCB standards are extremely hazardous to everyone in the laboratory if handled improperly. It is important that all necessary safety precautions are followed. Gloves and safety glasses must be worn when handling samples, reagents, and standards. It is necessary to wear a lab coat to protect clothing. All solvent usage must occur in properly working fume hoods. Use precautions to prevent spills; if any spills do occur, clean up all spills immediately and notify supervisor.

9.2 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding Safety Data Sheets.

9.3 Strong acids and bases should be added to containers and solutions under the hood. When all traces of fumes and odors have dissipated, containers and solutions may be returned to the laboratory.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptance criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP, recording all data as described in Section 7.1, and reviewing all QC results for compliance with acceptance criteria.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities and DOC performance.

11.0 Waste Management and Pollution Prevention

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Unused non-hazardous sample may be disposed of as wastewater. Solvents and extracted sample not evaporated in a hood are collected and disposed of as hazardous waste in designated solvent water waste containers. Minimize solvent use by preparing standards in amounts that will be used before expiration.

11.1 Solid Materials

Waste storage, transfer and disposal will be in compliance with NH DES and US EPA waste disposal regulations. Non-hazardous solid materials, sediments and soils will be disposed of through an appropriate waste disposal site. All non-hazardous solid waste will be drained of as much free liquid as possible. The remaining sample is removed from its original container and placed with other non-hazardous waste samples in a 55 gallon metal drum. Once full, these containers will then be transferred to an approved waste disposal facility. This transfer is recorded on a waste manifest and they are retained for a minimum of 3 years.

11.2 Aqueous Samples

Water samples that have been solvent extracted are added to the solvent water waste stream. Any aqueous samples received unpreserved as extra volume that were not solvent extracted may be disposed of through ESI's aqueous waste UV treatment system located on the first floor. Access points for aqueous waste disposal are located throughout the laboratory, second floor's access "waste chute" is located in the dishwashing area. ESI's Federal and State NPDES Permit allows waste disposed of in this manner to be discharged to the Taylor River.

Any samples found to be toxic may require further testing or treatment in order to determine proper disposal technique to assure compliance with all local, state and federal regulations.

11.3 Hazardous Waste Samples

Hazardous waste samples must be handled by properly trained employees. Only the ESI hazardous waste coordinator may train ESI employees on handling hazardous waste. There are 3 main hazardous waste streams at ESI, "dilute metals acidic", "solvents" and "Lachat wastes". When a satellite container needs to be emptied, it is properly transported to the waste shed and added into the appropriately labeled container for that waste stream. The volume and type of waste is recorded in the appropriate log book. Shipping manifests reflect when the waste drums are picked up for transport.

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TITLE: Analysis of Pesticides and PCBs in Extracts of Soil, Tissue and Water - Region II Testing

Appendix 1.

In each of the following appendix 1 parts, use the described stock standards to make the appropriate working standards.

(FV = Final Volume)

Part A. Pesticide Calibration Curve and Continuing Calibration Recipe

Stock Standards

DDT/Endrin	0.5 µg/mL
A Mix	80/160 ng/mL
B Mix	80/160 ng/mL
C Mix	80 ng/mL
Additonal Compound Mix	160 ng/mL
Toxaphene	10 µg/mL

Working Standards

		50 µL of DDT/Endrin	to	FV 500 µL Hexane	= 50 ng/mL DDT/Endrin
12.5 µL of A Mix	+	12.5 µL of B Mix	to	FV 500 µL Hexane	= 2 ng/mL AB
		50 µL Toxaphene	to	FV 500 µL Hexane	= 1 µg/mL Toxaphene
		12.5 µL of C Mix	to	FV 500 µL Hexane	= 2 ng/mL C
		125 µL of C Mix	to	FV 500 µL Hexane	= 20 ng/mL C
		250 µL of C Mix	to	FV 500 µL Hexane	= 40 ng/mL C
		375 µL of C Mix	to	FV 500 µL Hexane	= 60 ng/mL C
		125 µL of A Mix	+	375 µL of B Mix	= 20 ng/mL A, 60 ng/mL B
		250 µL of A Mix	+	250 µL of B Mix	= 40 ng/mL AB
		375 µL of A Mix	+	125 µL of B Mix	= 60 ng/mL A, 20 ng/mL B
				500 µL of A Mix	= 80 ng/mL A
				500 µL of B Mix	= 80 ng/mL B
				500 µL of C Mix	= 80 ng/mL C
		12.5 µL of Additional Compound Mix	to	FV 500 µL Hexane	= 2 ng/mL C
		125 µL of Additional Compound Mix	to	FV 500 µL Hexane	= 20 ng/mL C
		250 µL of Additional Compound Mix	to	FV 500 µL Hexane	= 40 ng/mL C
		375 µL of Additional Compound Mix	to	FV 500 µL Hexane	= 60 ng/mL C
				500 µL of Additional Compound Mix	= 80 ng/mL C
		250 µL of A Mix	+	250 µL of B Mix	= CCV 40 ng/mL AB
		250 µL of C Mix	+	250 µL of Hexane	= CCV 40 ng/mL C
		250 µL of Additional Compound Mix	+	250 µL of Hexane	= CCV 40 ng/mL Additional Compound
				1 mL Hexane	= CCB

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Part B. Pesticide Initial Calibration Verification

Stock Standards

AB Mix	80 ng/mL
C Mix	80 ng/mL
Additional Compound	80 ng/mL
TCMX/209 (SS)	0.5 µg/mL

Working Standards

250 µL of AB Mix	to	FV 500 µL Hexane	=	40 ng/mL AB
250 µL of C Mix	to	FV 500 µL Hexane	=	40 ng/mL C
250 µL of Additional Compound Mix	to	FV 500 µL Hexane	=	40 ng/mL Additional
50 µL SS	to	FV 500 µL Hexane	=	ICB

Part C. PCB Calibration Curve and Continuing Calibration Recipe

Stock Standards

Aroclor 1016	10 µg/mL
Aroclor 1260	10 µg/mL
TCMX/209 (SS)	0.5 µg/mL

Working Standards

10 µL Aroclor 1016 +	10 µL Aroclor 1260 + 50 µL of SS	to	FV 1.0 mL Hexane	=	0.1 µg/mL	
20 µL Aroclor 1016 +	20 µL Aroclor 1260 + 50 µL of SS	to	FV 1.0 mL Hexane	=	0.2 µg/mL	
50 µL Aroclor 1016 +	50 µL Aroclor 1260 + 50 µL of SS	to	FV 1.0 mL Hexane	=	0.5 µg/mL	
75 µL Aroclor 1016 +	75 µL Aroclor 1260 + 50 µL of SS	to	FV 1.0 mL Hexane	=	0.75 µg/mL	
100 µL Aroclor 1016 +	100 µL Aroclor 1260 + 50 µL of SS	to	FV 1.0 mL Hexane	=	1.0 µg/mL	
50 µL Aroclor 1016 +	50 µL Aroclor 1260 + 50 µL of SS	to	FV 1.0 mL Hexane	=	0.5 µg/mL CCV	
			1 mL Hexane	=	CCB	

Part D. PCB Initial Calibration Verification

Stock Standards

Aroclor 1016	10 µg/mL
Aroclor 1260	10 µg/mL
TCMX/209 (SS)	0.5 µg/mL

Working Standards

50 µL Aroclor 1016 +	50 µL Aroclor 1260	to	FV 1.0 mL Hexane	=	0.5 µg/mL ICV
	50 µL SS	to	FV 500 µL Hexane	=	ICB

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Part E. Toxaphene Calibration Curve and Continuing Calibration Recipe

Stock Standards

Toxaphene 10 µg/mL
TCMX/209 (SS) 0.5 µg/mL

Working Standards

5 µL of Toxaphene + 50 µL of SS to FV 500 µL Hexane = 0.1 µg/mL
12.5 µL of Toxaphene + 50 µL of SS to FV 500 µL Hexane = 0.25 µg/mL
25 µL of Toxaphene + 50 µL of SS to FV 500 µL Hexane = 0.5 µg/mL
50 µL of Toxaphene + 50 µL of SS to FV 500 µL Hexane = 1.0 µg/mL
250 µL of Toxaphene + 50 µL of SS to FV 500 µL Hexane = 5.0 µg/mL
25 µL of Toxaphene + 50 µL of SS to FV 500 µL Hexane = 0.5 µg/mL CCV
500 µL Hexane = CCB

Part F. Toxaphene Initial Calibration Verification

Stock Standards

Toxaphene 10 µg/mL
TCMX/209 (SS) 0.5 µg/mL

Working Standards

25 µL of Toxaphene + 50 µL of SS to FV 500 µL Hexane = 0.5 µg/mL ICV
500 µL Hexane = ICB

Part G. Chlordane Calibration Curve and Continuing Calibration Recipe

Stock Standards

Chlordane 10 µg/mL
TCMX/209 (SS) 0.5 µg/mL

Working Standards

10 µL of Chlordane + 50 µL of SS to FV 500 µL Hexane = 0.2 µg/mL
50 µL of Chlordane + 50 µL of SS to FV 500 µL Hexane = 1.0 µg/mL
125 µL of Chlordane + 50 µL of SS to FV 500 µL Hexane = 2.5 µg/mL
250 µL of Chlordane + 50 µL of SS to FV 500 µL Hexane = 5.0 µg/mL
500 µL of Chlordane + 50 µL of SS to FV 500 µL Hexane = 10.0 µg/mL
125 µL of Chlordane + 50 µL of SS to FV 500 µL Hexane = 2.5 µg/mL
500 µL Hexane = CCB

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Part H. Chlordane Initial Calibration Verification

Stock Standards

Chlordane 10 µg/mL
TCMX/209 (SS) 0.5 µg/mL

Working Standards

125 µL of Chlordane + 50 µL of SS to FV 500 µL Hexane = 2.5 µg/mL ICV
500 µL Hexane = ICB

Part I. PCB Calibration Curve and Continuing Calibration Recipe

Stock Standards

2,2',4,5'-Tetrachlorobiphenyl (BZ # 49) 10 µg/mL
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ # 183) 10 µg/mL
TCMX/209 (SS) 0.5 µg/mL

Working Standards

10 µL BZ # 49 + 10 µL BZ # 183 + 50 µL of SS to FV 1.0 mL Hexane = 0.1 µg/mL
20 µL BZ # 49 + 20 µL BZ # 183 + 50 µL of SS to FV 1.0 mL Hexane = 0.2 µg/mL
50 µL BZ # 49 + 50 µL BZ # 183 + 50 µL of SS to FV 1.0 mL Hexane = 0.5 µg/mL
75 µL BZ # 49 + 75 µL BZ # 183 + 50 µL of SS to FV 1.0 mL Hexane = 0.75 µg/mL
100 µL BZ # 49 + 100 µL BZ # 183 + 50 µL of SS to FV 1.0 mL Hexane = 1.0 µg/mL
50 µL BZ # 49 + 50 µL BZ # 183 + 50 µL of SS to FV 1.0 mL Hexane = 0.5 µg/mL CCV
1 mL Hexane = CCB

Part J. PCB Initial Calibration Verification

Stock Standards

2,2',4,5'-Tetrachlorobiphenyl (BZ # 49) 10 µg/mL
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ # 183) 10 µg/mL
TCMX/209 (SS) 0.5 µg/mL

Working Standards

50 µL BZ # 49 + 50 µL BZ # 183 to FV 1.0 mL Hexane = 0.5 µg/mL ICV
50 µL SS to FV 500 µL Hexane = ICB

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Appendix 2. Pesticide Standard Mixes

Pesticide Standard Mix A		Pesticide Standard Mix B		Pesticide Standard Mix C	
Tetrachlor-m-xylene	0.08 µg/mL	Tetrachloro-m-xylene	0.08 µg/mL	Hexachlorobenzene	0.08 µg/mL
alpha-BHC	0.08 µg/mL	beta-BHC	0.08 µg/mL	Oxychlordane	0.08 µg/mL
gamma-BHC	0.08 µg/mL	delta-BHC	0.08 µg/mL	Chloryripnos	0.08 µg/mL
Heptachlor	0.08 µg/mL	Aldrin	0.08 µg/mL	Trans-nonachlor	0.08 µg/mL
Endosulfan I	0.08 µg/mL	Heptachlor epoxide	0.08 µg/mL	Cis-nonachlor	0.08 µg/mL
Dieldran	0.16 µg/mL	gamma-Chlordane	0.08 µg/mL		
Endrin	0.16 µg/mL	alpha-Chlordane	0.08 µg/mL		
4,4'-DDD	0.16 µg/mL	4,4'-DDE	0.16 µg/mL		
4,4'-DDT	0.16 µg/mL	Endosulfan II	0.16 µg/mL		
Methoxychlor	0.80 µg/mL	Endrin Aldehyde	0.16 µg/mL		
Decachlorobephenyl	0.16 µg/mL	Endosulfan sulfate	0.16 µg/mL		
		Endrin ketone	0.16 µg/mL		
		Decachlorobephenyl	0.16 µg/mL		

Pesticide Standard Mix Additional	
2,4'-DDD	0.16 µg/mL
2,4'-DDE	0.16 µg/mL
2,4'-DDT	0.16 µg/mL

Appendix 3. Analyte List and Target Reporting Limits

Analyte	Tissue (ng/g)	Sediment (ng/g)	Water (µg/L)
Aldrin	4.3	1	0.4
Dieldrin	1.5	1	0.20
a-Chlordane	2.7	1	0.030
Trans Nonachlor	2.7	1	0.010
Heptachlor	2.7	1	0.010
Heptachlor Epoxide	2.7	1	0.010
Endosulfan I	0.32	1	0.010
Endosulfan II	0.32	1	0.010
Endosulfan Sulfate	0.32	1	0.010

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4,4-DDT	1	1	0.04
2,4-DDT	1	1	0.010
4,4-DDD	1	1	0.010
2,4-DDD	1	1	0.010
4,4-DDE	1	1	4.0
2,4-DDE	1	1	0.010

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Title: Tissue Extraction for Organic Compounds - Region II Testing

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	A. MacKinnon	01/2018

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1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the procedure for extracting tissue samples for organic compounds using elements of US EPA method 3570. The procedure for florisil column cleanup uses US EPA method 3620 and the procedure for silica gel column cleanup uses US EPA method 3630. The procedure for sulfur cleanup uses US EPA method 3660B and the procedure for sulfuric acid cleanup uses US EPA method 3665A. This protocol is designed for use with samples from US EPA Region II and the New York District ACOE.

Samples are spiked with an appropriate surrogate solution and extracted with solvent in glass. The solvent extracts are collected and adjusted to the appropriate final volume, typically 0.2 to 2.0 mL, by ebullition on a warming bath, such as Meyer N-Evap, or using a K-D apparatus. The extract is stored in glass and refrigerated until GC or GC/MS analysis. Samples may require additional clean-up.

Cleanup options may include but are not limited to:

Pesticides (PEST): Florisil column chromatographic cleanup (see section 5.2).
Polynuclear Aromatic Hydrocarbons (PAH): Silica gel column chromatographic cleanup (see section 5.3).
PCB Congeners (CGR): Treatment with concentrated sulfuric acid (see section 5.4).

2.0 Definitions

See ESI's Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Method 3510C (SW-846). (This method should be consulted for interferences and pollution prevention.)

US EPA. 2002. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Method 3570. (SW-846).

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Methods 3620C and 3630C (SW-846).

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Methods 3660B (SW-846).

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Methods 3665A (SW-846).

US EPA. 1983. *Methods for Chemical Analysis of Water and Wastes.* Method 608.

US EPA. 1988. *Statement of Work for Organic Analysis, Multi-Media Concentration.* Pesticide D-11.

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US EPA. 1985. *Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry.* Method 680.

Agilent Technologies. 1999. *Hardware Manual.* 5973 Network Mass Selective Detector.

Agilent Technologies. 1997-2000. *Operation Guide.* Agilent 7683 Automatic Liquid Sampler.

Agilent Technologies. 2000. *Operating Manual.* Agilent 6890 Series Gas Chromatograph.

Agilent Technologies. 1989-2001. *G1701DA Version D.00.00.38 19-Nov 2001.* Environmental ChemStation.

Department of Defense (DoD), Department of Energy (DoE). 2017. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.1.

4.0 Materials and Apparatus

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date. Note that standards may also be prepared according to project specific analyte list.

4.1 Reagents and Standards

Acetone, pesticide grade
Cyclohexane, pesticide grade
Florisil, activated at 675°C for 4 hours
Hexane, pesticide grade
Methylene chloride, pesticide grade
Silica Gel, use as received or activate at 130°C for 16 hours
Sodium sulfate, heated at 400°C for 4 hours to remove phthalates and other interfering organic substances.
Pesticide surrogate and spike standards, provided by Supelco or other acceptable vendors. See Appendix A: Part 1 for standard preparation instructions.
Congener surrogate and spike standards, provided by Ultra or other acceptable vendors. See Appendix A: Part 2 for standard preparation instructions.
PAH surrogate and spike standards, provided by Environmental Consulting and Supplies or other acceptable vendors. See Appendix A: Part 3 for standard preparation instructions.

4.2 Equipment and Supplies

40 mL Glass screw top vial
50 mL Round bottom glass vials
2 mL Glass extract collection vials, with Teflon lined lids
Analytical balance, capable of weighing to 0.01 g
Evaporation containers (e.g. centrifuge test tubes)
Fume hood
Gas-tight volumetric syringes

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Glass chromatography columns
Glass funnels
Kuderna-Danish apparatus
Micro-Snyder column
Meyer N-Evap Analytical Evaporator
Mortar and pestle
Silanized glass wool
Water/steam bath, for K-D solvent evaporation
Zymark TurboVap Evaporator
Kimwipes

5.0 Methods and Procedures

Samples should be stored in glass jars with Teflon lined caps. Tissue samples must be frozen until extracted. It is recommended that extractions be performed as soon as possible, but should be done within 360 days. Aqueous samples should be collected in 1 L glass jars with Teflon lined caps. Remove known free residual chlorine with ascorbic acid. Adjust pH to 5 - 9 SU if needed. Store samples at 4±2°C. Holding time for aqueous samples is 7 days, for solids it is 14 days (1 year if frozen).

5.1 Extraction Procedure

Ensure all glassware is clean and dry and thoroughly solvent rinse all glassware, mortar and pestle with the appropriate solvent before proceeding with extractions.

5.1.1 Measure and add approximately 4 g of homogenized sample into mortar. Use sodium sulfate to grind mixture until sample turns to a fine, dry, free-flowing powder. Start with approximately 1 g of sodium sulfate and add more as needed. Transfer the sample from the mortar into a labeled 40 mL vial with a Teflon lined cap.

Note that this step deviates from the order of procedures in method 3570. Method 3570 requires the addition of sodium sulfate to the extraction vessel before the sample and does not require the sodium sulfate and sample to be mixed in a mortar prior to being transferred into the extraction vessel.

5.1.2 Unless otherwise specified, 4 g sodium sulfate will serve as the laboratory blank and Laboratory Control Sample (LCS).

5.1.3 Using a gas-tight volumetric syringe, carefully add the appropriate amount of surrogate solution to each sample, including the blank and LCS. Add the appropriate amount of spiking solution to the LCS and to the Matrix Spike (MS), Matrix Spike Duplicate (MSD), and Matrix Spike Triplicate (MST) samples. Sample should be spiked at 3-5x the reporting limit. See Appendix B for the appropriate reporting limits. See Appendix A for standard concentrations and recipes.

Note that the sample used to perform the MS/MSD/MST will be the pretest tissue of the *M. nasuta* or *N. virens* for tissues and site water for waters and reference sediment for solids analysis.

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5.1.4 Add approximately 10 to 15 mL or more of solvent (see Appendix A for appropriate extraction solvent) to all sample vials and shake each vial for 2 minutes. If required, samples may be rotated end over end for at least 4 hours for the first extraction. Let settle for 10 minutes and if necessary, vials may be centrifuged for 15 minutes at 500 rpms.

Note that the volume and type of solvent used for extraction deviates from method 3570 specification of 12 mL of methylene chloride.

5.1.5 Decant extract into a glass vial for concentration. If necessary, extract can be filtered through a glass funnel containing sodium sulfate with glass wool plug or filter paper into a glass vessel. Sodium sulfate must be pre-rinsed and wetted with appropriate solvent prior to use. Once sample has been filtered, rinse sodium sulfate with appropriate solvent immediately, ensuring the sodium sulfate does not go dry. Before recapping vial, ensure the vial lip and threads are free from debris and wipe with a Kimwipe as necessary.

5.1.6. Repeat section 5.1.4 and 5.1.5 twice more. Glass vessel should be capped while not actively being added to. After final extraction, rinse sodium sulfate with 2 - 3 mL of extraction solvent to ensure complete transfer.

5.1.7. Combine all three extracts and clean up as necessary (see sections 5.2 through 5.5). Adjust to the appropriate final volume (typically 0.2 - 2.0 mL) by ebullition on a warming bath, such as the Meyer N-Evap or K-D apparatus.

If K-D is used, add Teflon boiling stick or other appropriate material to the concentration vessel and attach micro-Snyder column pre-wet Snyder column with 0.5 mL of extraction solvent. Place in a water bath maintained at a constant temperature, approximately 60-65°C. Temperature and position of K-D apparatus should be adjusted so the solvent boils evenly, the Snyder column balls chatter and the columns do not flood. Concentrate extract until about 1 mL is remaining.

5.1.8 Let the final extract cool (and drain if K-D was used) then transfer to a screw cap Teflon lined septum glass vial for analysis. Rinse the evaporation container with solvent for complete transfer, and prepare the exact final volume with a gas tight syringe. See Appendix A for appropriate final volumes. If the sample residue prevents concentrating to the desired volume, then adjust the final volume upward with solvent and record to the nearest 0.01 mL.

5.1.9 Extract condition and preparation data are verified by the laboratory supervisor and then transferred to the organics extract refrigerator maintained at 4±2°C until time of analysis.

5.2 Florisil Column Cleanup

Florisil columns may be useful in cleaning up pesticide extracts. Following the florisil column cleanup, proceed to finalize the extract volume and prepare for analysis (see steps 5.1.7 through 5.1.9).

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5.2.1 Prepare a florisil column for each extract using a 10 mL glass chromatography column fitted with silanized glass wool and a sodium sulfate plug at the bottom. Fill the column with 6 - 7 cc of Florisil and tap the column gently to settle the powder. Insert a sodium sulfate plug into the top of the column.

5.2.2 Secure the column on a pipette stand in a fume hood. Pre-clean the column with approximately 25 mL of $MeCl_2$ and discard to waste. Pre-elute the column with approximately 25 mL of hexane and allow to drain into a waste container to be discarded. Keep the column wet with solvent above the sodium sulfate.

5.2.3 Transfer all or a known portion of the sample extract to the top of the florisil column using a gas tight syringe. Record the amount of sample (mL) taken for the cleanup. Add sufficient hexane to the sample vial to rinse it, then transfer rinse to the column. Elute the column with 50 mL of $MeCl_2$. Collect the eluate in a clean, labeled, solvent rinsed evaporation container. Extracts must be exchanged into hexane before the extract is prepared for analysis.

5.3 Silica Gel Column Cleanup

Silica gel columns may be useful for PAH extracts. Following the silica gel column cleanup, proceed to finalize the extract volume and prepare for analysis (see steps 5.1.7 through 5.1.9).

5.3.1 Prepare a silica gel column for each extract using a 10 mL glass chromatography column fitted with silanized glass wool and a sodium sulfate plug at the bottom. Fill the column with 6 to 7 cc of silica gel and tap the column gently to settle the powder. Insert a sodium sulfate plug into the top of the column.

5.3.2 Secure the column on a pipette stand in a solvent delivery system in a fume hood. Pre- clean the column with approximately 25 mL of $MeCl_2$ and discard to waste. Pre-eulte the column with 25 mL of pentane into a waste container to be discarded. Keep the column wet with solvent above the sodium sulfate.

5.3.3 Transfer all or a known portion of the sample extract to the top of the silica gel column using a gas tight syringe. Extracts must be exchanged into hexane before being transferred to the column. Record the amount of sample (mL) taken for cleanup. Add sufficient hexane to the sample vial to rinse, then transfer the rinse to the column. Elute the column with 50 mL of 1+1 $MeCl_2$/pentane. Collect the eluate in a clean, labeled, solvent rinsed evaporation container.

5.4 Sulfuric Acid Cleanup

Sulfuric acid is used to clean up CGR extracts. Any column cleanup should occur before the acid cleanup. Extracts must be exchanged into hexane before adding any acid. Add approximately 0.5 mL of concentrated sulfuric acid to the extract, cap and shake. Allow the extract to settle for at least one hour, where the extract will separate into two distinct layers. Carefully pull off the top layer (the organic layer) using a gas tight syringe. Transfer to clean vial for analysis. Be very careful not to get any acid in the extract for analysis. Record the extract volume recovered.

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5.5 Copper Cleanup

Copper is used to remove sulfur from sample extracts. The copper must be very reactive prior to use. All oxides must be removed so the copper is shiny and bright. If the copper does not look this way, then it needs to be reactivated by rinsing with dilute HNO_3 acid, Milli-Q® water, and then acetone. Approximately 2 g of cleaned copper is added to the sample extract and agitated for a minimum of one minute. Copper should be added to extract until there is no sulfur left to react with the copper (copper will stop turning black once all sulfur has been removed). Once this has been achieved, the extract should be removed from the copper and transferred to a clean vial for analysis.

6.0 Quality Control Requirements

Quality control elements may be added, removed, or altered to meet specific program or project requirements. Alternate acceptance criteria may be set for specific programs or projects, and reporting limits are specific to each parameter and client protocols. For DoD-specific projects see the QSM 5.1, as appropriate.

Note that all QC elements and samples must be spiked with a surrogate standard.

6.1 Demonstrations of Capability (DOC)

An analyst must complete a DOC before working independently on this procedure and yearly thereafter. A DOC is also required with any changes to instrument, method, or analyst. To perform a DOC, four replicates of a prepared quality control sample must be prepared. DOC samples must meet LCS acceptance criteria. For more information on DOCs see SOP QA-1108 “Training”.

6.2 Method Detection Limit (MDL)

The MDL is calculated using the appropriate student’s t-parameter value multiplied by the standard deviation of 8 or more replicates of spiked samples or standards. MDLs are performed annually and with changes in instrument or method. The calculated MDL must be lower than the reporting limit.

The t-parameter value is from a one-tailed Student’s t-Test at the 99% confidence interval using n-1 degrees of freedom, with “n” being the number of replicates.

The most recent MDL study can be found in the inorganics quality control directory on the ESI network. For more information on MDLs see SOP QA-1111 “Determination of Method Detection and Reporting Limits”.

6.3 Limit of Detection (LOD)

LOD determination will be conducted on a quarterly basis. For a single-analyte standard, the LOD is a single quality control sample that is approximately 2 to 3 times the MDL but no greater than the reporting limit. For a multi-analyte standard, the LOD is a single quality control sample that is approximately 1 to 4 times the MDL but no greater than the reporting limit.

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The apparent signal to noise ratio at the LOD must be at least 3 or for methods that do not produce a signal to noise the LOD result must be at least 3 standard deviations greater than the mean method blank.

6.4 Limit of Quantitation (LOQ)

LOQ confirmation will be conducted on a quarterly basis. The LOQ is a single quality control sample no more than 1 to 2 times the reporting limit. The LOQ standard acceptance criteria is 30-150%. For more information on LODs and LOQs see SOP QA-1147 “Determination of LOD and LOQ Quality Control Samples”.

6.5 Blank, LCS, MS, Relative Standard Deviation (RSD)

6.5.1 A blank and LCS will be analyzed every batch or every 20 samples, whichever is more frequent. MS/MSD/MST samples will be analyzed every batch of 20 samples or at 5% frequency.

6.5.2 Acceptance criteria for all blanks is less than the reporting limit.Reporting limits can be found in Appendix B.

6.5.3 Acceptance criteria for LCS recovery is 50-150%. LCS samples are spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix B.

6.5.4 Acceptance criteria for MS/MSD/MST recovery is 50-150%. Acceptance criteria for MSD RSD is less than or equal to 50%. MS/MSD/MST are spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix B. For aqueous analysis site water will be used to perform all MS/MSD/MST samples. For sediment analysis reference sediment will be used to perform all MS/MSD/MST samples. For tissue analysispretest tissue will be used to perform all MS/MSD/MST samples.

6.5.5 Acceptance criteria for surrogate standard recovery is 30-150%. All applicable surrogate standards must be within the acceptance range to be considered acceptable.If the surrogate recoverys fall outside of the acceptable range the client must be notified and the sample must be re extracted.

6.5.6 Acceptance criteria for initial calibration check standard recovery is 80-120%.

6.5.7 Acceptance criteria for continuing calibration check recovery is 80-120%.

6.6 Interferences

Solvents, standards, and labware may be contaminated with artifacts that would affect results. Phthalate esters can pose a contamination issue with all analytes not just phthalate esters themselves.

7.0 Calculations and Reporting

7.1 The following information should be recorded when performing a tissue extraction for organic compounds:

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- Date
- Analyst's initial
- Batch
- Sample ID, with dilution factor if applicable
- Sample code / standard code
- Solvent lot ID
- Surrogate ID and amount, with dilution factor (if applicable)
- Spike ID and amount, with dilution factor (if applicable)
- Internal Standard ID and amount
- Initial weight of wet sample
- Final volume
- Volume taken for cleanup (if applicable)
- Volume of cleaned sample
- Method
- Balance identification number
- Filter paper lot number (if applicable)
- Silica gel, florisil, and glass wool lot numbers (if applicable)
- Notes, if any

7.2 Calculations

Calibrate the system software as per the Agilent manuals referenced in Section 3.0. When reasonable, use the system software to calculate the values for QC elements and samples. Review the data to ensure it is correct and that all quality control requirements have been met. When needed, manually integrate the values according to ESI SOP QA-1132 "Manual Chromatographic Peak Integration".

7.2.1 Calculate LCS/Surrogate Recovery as follows:

$$\frac{\text{LCS/SUR Concentration}}{\text{Concentration added}} \times 100 = \%\ \text{Recovery}$$

7.2.2 Calculate %RSD as follows:

$$\frac{\text{Std}(\text{Result 1 - Result 2})}{\text{Avg}(\text{Result 1 - Result 2})} \times 100 = \%\text{RSD}$$

7.2.3 Calculate Spike Recovery as follows:

$$\frac{\text{Spiked Sample Concentration - Sample Concentration}}{\text{Concentration Added}} \times 100 = \%\ \text{Recovery}$$

8.0Corrective Actions

8.1 Data Qualification

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8.1.1 If DOCs, MDLs, LODs, or LOQs do not meet acceptability criteria, then repeat until acceptability criteria is met.

8.1.2 If the LCS sample does not meet percent recovery acceptance criteria, then they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (using the "J" qualifier).

8.1.3 If samples analyzed with blanks are not meeting acceptance criteria, then they may be reanalyzed or reported with qualification (using the "B" qualifier).

8.1.4 If MS samples do not meet percent recovery acceptance criteria, then they may be reanalyzed or reported with qualification (using the "J" qualifier). If the sample result or sample duplicate result is less than 5 times the reporting limit, then the RSD does not need to be evaluated.

8.1.5 If the MS recovery is not within acceptance criteria, then samples may be reanalyzed or reported with qualification (using the "J" qualifier). If the sample result is greater than 5 times the spike amount, then the percent recovery need not be calculated.

8.1.6 If the surrogate recovery is not within the acceptable range, then the client is notified and the sample is re-extracted whenever possible. Sample matrix effect will be proven if the results obtained from the re-extracted sample show similar results as the first recovery. When this occurs, both results are reported with an explanation. If the results of the re-extracted sample are in the acceptable range, then there is a problem with the first extraction and only the compliant results are reported. If no additional sample can be obtained, then results are flagged in the report and a narrative is included qualifying the data.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified.

- Quality and expiration date of reagents
- Cleanliness of containers, equipment, and labware
- Integrity of standards
- GC and/or GC/MS operation

9.0 Health and Safety

9.1 As with all samples, gloves and safety glasses should be worn when handling samples, reagents, and standards. It is necessary to wear a lab coat to protect clothing. All solvent usage must occur in properly working fume hoods.

9.2 Methylene chloride, pentane, and cyclohexane are extremely hazardous to everyone in the laboratory if handled improperly. It is important that all necessary safety precautions are followed.

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9.3 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding MSDS.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptance criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP, recording all data as described in Section 7.1, and reviewing all QC results for compliance with acceptance criteria.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities and DOC performance.

11.0 Waste Management and Pollution Prevention

Unused non-hazardous tissue samples may be disposed of as general trash. Solvents not evaporated in a hood are collected and disposed of as hazardous waste in designated solvent water waste containers. Extracted tissue, once dried should not be hazardous but may be disposed of as hazardous waste. Extracts and standard residues are deemed hazardous and are disposed of as such. Minimize solvent use. Prepare standards in amounts that will be used before expiration.

11.1 Solid Materials

Waste storage, transfer and disposal will be in compliance with NH DES and US EPA waste disposal regulations. Non-hazardous solid materials, sediments and soils will be disposed of through an appropriate waste disposal site. All non-hazardous solid waste will be drained of as much free liquid as possible. The remaining sample is removed from its original container and placed with other non-hazardous waste samples in a 55 gallon metal drum. Once full, these containers will then be transferred to an approved waste disposal facility. This transfer is recorded on a waste manifest and they are retained for a minimum of 3 years.

11.2 Hazardous Waste Samples

Hazardous waste samples must be handled by properly trained employees. Only the ESI hazardous waste coordinator may train ESI employees on handling hazardous waste. There are 3 main hazardous waste streams at ESI, "dilute metals acidic", "solvents" and "Lachat wastes". When a satellite container needs to be emptied, it is properly transported to the waste shed and added into the appropriately labeled container for that waste stream. Logbooks are kept as a record for volumes added to each drum of any specific waste stream and shipping manifests reflect when they are picked up for transport.

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Appendix A

Pesticides			Congener			PAH		
Initial Volume: 4 g			Initial Volume: 4 g			Initial Volume: 4 g		
Final Volume: 0.4 mL			Final Volume: 0.4 mL			Final Volume: 0.4 mL		
Solvent: Hexane			Solvent: $MeCl_2$			Solvent: $MeCl_2$		
Surrogate - 50 µL			Surrogate - 40 µL			Surrogate - 40 µL		
Tetrachloro-m-xylene	0.5	µg/mL	198	2	µg/mL	o-terphenyl	10	µg/mL
Decachlorobiphenyl	0.5	µg/mL				2-fluorobiphenyl	10	µg/mL
			Spike - 20 µL					
Pesticide A Mix - 80 µL			8	4	µg/mL	Spike - 100 µL		
TCMX (SS)	0.08	µg/mL	18	4	µg/mL	naphthalene	10	µg/mL
alpha-BHC	0.08	µg/mL	28	4	µg/mL	acenaphthylene	10	µg/mL
gamma-BHC (Lindane)	0.08	µg/mL	44	4	µg/mL	acenaphthene	10	µg/mL
heptachlor	0.08	µg/mL	52	4	µg/mL	fluorene	10	µg/mL
endosulfan I	0.08	µg/mL	66	4	µg/mL	phenanthrene	10	µg/mL
dieldrin	0.16	µg/mL	77	4	µg/mL	anthracene	10	µg/mL
endrin	0.16	µg/mL	101	4	µg/mL	fluoranthene	10	µg/mL
4,4'-DDD	0.16	µg/mL	105	4	µg/mL	pyrene	10	µg/mL
4,4'-DDT	0.16	µg/mL	118	4	µg/mL	benzo[a]anthracene	10	µg/mL
methoxychlor	0.8	µg/mL	126	4	µg/mL	chrysene	10	µg/mL
209 (SS)	0.16	µg/mL	128	4	µg/mL	benzo[b]fluoranthene	10	µg/mL
			138	4	µg/mL	benzo[k]fluoranthene	10	µg/mL
Pesticide B Mix - 80 µL			153	4	µg/mL	benzo[a]pyrene	10	µg/mL
TCMX (SS)	0.08	µg/mL	170	4	µg/mL	indeno[1,2,3-cd]pyrene	10	µg/mL
beta-BHC	0.08	µg/mL	180	4	µg/mL	dibenz[a,h]anthracene	10	µg/mL
delta-BHC	0.08	µg/mL	187	4	µg/mL	benzo[g,h,i]perylene	10	µg/mL
aldrin	0.08	µg/mL	195	4	µg/mL			
heptachlor epoxide	0.08	µg/mL	206	4	µg/mL			
gamma-chlordane	0.08	µg/mL	209	4	µg/mL			
alpha-chlordane	0.08	µg/mL	49	4	µg/mL			
4,4'-DDE	0.16	µg/mL	183	4	µg/mL			
endosulfan II	0.16	µg/mL	87	4	µg/mL			
endrin aldehyde	0.16	µg/mL	184	4	µg/mL			
endosulfan sulfate	0.16	µg/mL						
endrin ketone	0.16	µg/mL						
209 (SS)	0.16	µg/mL						
Pesticide C Mix - 80 µL								
oxychlordane	0.08	µg/mL						
chlorpyrifos	0.08	µg/mL						
trans-nonachlor	0.08	µg/mL						
cis-nonachlor	0.08	µg/mL						
hexachlorobenzene	0.08	µg/mL						

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Additional Compound Mix - 80 µL	
2,4'-DDD	0.16 µg/mL
2,4'-DDE	0.16 µg/mL
2,4'-DDT	0.16 µg/mL

Part 1 (See above for compounds in each standard)

Pesticide Surrogate Mix - 0.5 µg/mL
Stock Standard: Supelco Cat# 861284 200 µg/mL
Dilute 0.075 mL stock standard to final volume 30 mL with hexane

Pesticide Mix A - 80/160 ng/mL
Stock Standard: Supelco Cat# 47977 0.5-5.0 µg/mL
Dilute 1.0 mL stock standard to final volume 6.25 mL with hexane

Pesticide Mix B - 80/160 ng/mL
Stock Standard: Supelco Cat# 47977 0.5-1.0 µg/mL
Dilute 1.0 mL stock standard to final volume 6.25 mL with hexane

Pesticide Mix C - 80 ng/mL
Stock Standard: Individual component (see list in Appendix A) at 100 µg/mL from Crescent Chemical Company
Dilute 0.2 mL each stock standard to final volume 2.0 mL with hexane

Additional Compound Mix - 80 ng/mL
Stock Standard: Individual component (see list in Appendix A) at 1000 µg/mL from Restek
Dilute 0.5 µL each stock standard to final volume 6.25 mL with hexane

Part 2 (See above for compounds in each standard)

Congener Surrogate - 2 µg/mL
Stock Standard: Ultra Cat# RPC-075AS 100 µg/mL
Dilute 0.6 mL stock standard to final volume 30 mL with 50:50 hexane:acetone

Congener Spike - 4 µg/mL
Stock Standards: Ultra Scientific Cat# RPC-EPA-2-1, RPC-030AS, RPC-073AS, RPC-099AS, RPC-075AS and RPC-168AS 100 µg/mL
Dilute 0.15 mL of each stock standard to final volume 3.75 mL with hexane

Part 3 (See above for compounds in each standard)

PAH Surrogates - 10 µg/mL
Stock Standards: 2-Fluorobiphenyl Chemservice Cat #N-10359-16 Neat
O-terphenyl Chemservice Cat# S-12693X4-1ML 1000 µg/mL
Intermediate Stock Standards: 2-Fluorobiphenyl - 50 mg stock standard to final volume 25 mLwith MeCl2 2000 µg/mL
O-terphenyl 1.0 mL stock standard to final volume 10 mL with acetone 200 µg/mL
Second Intermediate Stock Standard: 2-Fluorobiphenyl - Dilute 1 mL intermediate standard to final volume 10 mL with acetone 200 µg/mL
2-Fluorobiphenyl: Dilute 2.0 mL second intermediate standard to final volume 40 mL with acetone 10 µg/mL
O-terphenyl: Dilute intermediate standard to final volume 40 mL with acetone 10 µg/mL

PAH Spike Mix - 10 µg/mL
Stock Standard: Environmental Consulting and Supplies, Inc Cat# ECS-A-032, ECS-B-032, ECS-N-032 2000 µg/mL
Intermediate Stock Standard: Dilute 2 mL of stock standard to final volume 40 mL acetone 100 µg/mL
Dilute 1.0 mL of intermediate stock standard to final volume 10 mL acetone

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Appendix B

Analyte	Tissue (ng/g)	Sediment (ng/g)	Water (µg/L)
Aldrin	**4.3**	**1**	**0.4**
Dieldrin	**1.5**	**1**	**0.20**
a-Chlordane	**2.7**	**1**	**0.030**
Trans Nonachlor	**2.7**	**1**	**0.010**
Heptachlor	**2.7**	**1**	**0.010**
Heptachlor Epoxide	**2.7**	**1**	**0.010**
Endosulfan I	**0.32**	**1**	**0.010**
Endosulfan II	**0.32**	**1**	**0.010**
Endosulfan Sulfate	**0.32**	**1**	**0.010**
4,4-DDT	**1**	**1**	**0.04**
2,4-DDT	**1**	**1**	**0.010**
4,4-DDD	**1**	**1**	**0.010**
2,4-DDD	**1**	**1**	**0.010**
4,4-DDE	**1**	**1**	**4.0**
2,4-DDE	**1**	**1**	**100**

Analyte	Tissue (ng/g)	Sediment (ng/g)	Water (µg/L)
PCB 8	**0.4**	**1**	**0.002**
PCB 18	**0.4**	**1**	**0.002**
PCB 28	**0.4**	**1**	**0.002**
PCB 44	**0.4**	**1**	**0.002**
PCB 49	**0.4**	**1**	**0.002**
PCB 52	**0.4**	**1**	**0.002**
PCB 66	**0.4**	**1**	**0.002**
PCB 87	**0.4**	**1**	**0.002**
PCB 101	**0.4**	**1**	**0.002**
PCB 105	**0.4**	**1**	**0.002**
PCB 118	**0.4**	**1**	**0.002**
PCB 128	**0.4**	**1**	**0.002**
PCB 138	**0.4**	**1**	**0.002**
PCB 153	**0.4**	**1**	**0.002**
PCB 170	**0.4**	**1**	**0.002**
PCB 180	**0.4**	**1**	**0.002**
PCB 183	**0.4**	**1**	**0.002**
PCB 184	**0.4**	**1**	**0.002**
PCB 187	**0.4**	**1**	**0.002**
PCB 195	**0.4**	**1**	**0.002**
PCB 206	**0.4**	**1**	**0.002**

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Analyte	Tissue (ng/g)	Sediment (ng/g)	Water (µg/L)
PCB 209	**0.4**	**1**	**0.002**
Acenaphthene	**50**	**100**	**N/R**
Acenapthylene	**50**	**100**	**N/R**
Anthracene	**50**	**100**	**N/R**
Fluorene	**50**	**100**	**N/R**
Naphthalene	**50**	**100**	**N/R**
Phenanthrene	**50**	**100**	**N/R**
Benzo(a)anthracene	**50**	**100**	**N/R**
Benzo(a)pyrene	**50**	**100**	**N/R**
Benzo(g,h,i)perylene	**50**	**100**	**N/R**
Benzo(b)fluoranthene	**50**	**100**	**N/R**
Benzo(k)fluoranthene	**50**	**100**	**N/R**
Chrysene	**50**	**100**	**N/R**
Dibenzo(a,h)anthracene	**50**	**100**	**N/R**
Fluoranthene	**50**	**100**	**N/R**
Indeno(1,2,3-cd)pyrene	**50**	**100**	**N/R**
Pyrene	**50**	**100**	**N/R**
1,4-dichlrorbenzene	**3900**	**100**	**N/R**

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TITLE: Preparation of Suspended Particulate Phase (SPP) and Elutriate Solutions for Marine Sediment Evaluations, based on Region II Guidelines

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Created, based on QA-1330	J. T. Provencher	01/26/18

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TITLE: Preparation of Suspended Particulate Phase (SPP) and Elutriate Solutions for Marine Sediment Evaluations, based on Region II Guidelines

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the process used for preparing Suspended Particulate Phase (SPP) elutriate solutions using a standard approach for subsequent toxicological and chemical analyses. The protocol is designed for use with samples from US EPA Region II and the New York District ACOE.

The suspended particulate phase of a proposed dredge sample represents the portion of the sediment that is likely to be dissolved and dispersed in the water column during dredging and disposal operations. This preparation process mixes volumes of sediment and water, in a 1:4 ratio for 30 minutes, in an attempt to mimic dredging and sediment handling procedures that would take place in the field. The process will wash out, or elute, contaminants that are weakly bound to the sediments and likely to be released during dredging and disposal. Created elutriate solutions may be evaluated for potential environmental impact using bioassay methods. They may also be used for chemical procedures to determine the amount of contaminant of concern present. Bioassays can directly evaluate the potential for biological impacts due to both the physical presence of suspended particles and any biologically active contaminants associated with the particulates, including the dissolved fraction.

2.0 Definitions

Elutriate: The liquid solution recovered from a mix of sediment and water after a defined mixing/settling period and subsequent filtration to removed non-dissolved material.

Suspended Particulate Phase (SPP): Term which represents the toxicity testing performed using the unfiltered supernatant as obtained from the dredged material elutriate preparation.

Sediment: An accumulation of particulate material that usually lies below a body of water's surface.

3.0 Applicable Documents and References

U.S. EPA Region I, U.S. Army Corps of Engineers, New England Division. 2004. *Regional Implementation Manual for Evaluation of Dredged Material Proposed for Disposal in New England Waters*.

U.S. EPA, U.S. Army Corps of Engineers. 1998. *Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual* . EPA-823-B-98-002.

Department of Defense (DoD), Department of Energy (DoE). 2017. *DoD Quality Systems Manual (QSM) for Environmental Laboratories.* Version 5.1.

US EPA Region 2, US Army Corps of Engineers, New York District. 2016. *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal*.

SOP QA-1113 "Cleaning and Maintenance of General Laboratory Glassware"

4.0 Materials and Apparatus

0.45 µm Filters
1 Gallon cubitainers

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1 L Plastic containers, generally Tri-pour® design
5 Gallon buckets
60 mL Luer-lock syringes, Latex and silicone oil free
Air compressor, with valves to adjust flow to maintain stirring speed
Approved site water
Electronic balance, sensitivity of 0.1 g
Funnel
Graduated cylinder, generally 1 or 2 L
Magnetic filter funnel setup
Mixing chambers, generally stainless steel stock pots
Pneumatic drill
Stainless steel paddle stirring rod
Teflon® bags, used to line 5 gallon buckets
Teflon® transfer tubing
Test sediment
Timer / clock

5.0 Methods and Procedures

5.1 Sample Collection, Preservation and Storage

5.1.1 Test substances will be clearly identified as to source, collection date, period of collection, and description of collection methods. The laboratory will identify sample storage procedures and maintain sample receipt records. Records for sample storage, usage and disposal are generated when necessary.

5.1.2 Test material is stored at 4±2°C in the original shipping container. Holding time is dependent upon specific project objectives and is variable. The maximum holding time for overlying waters used for SPP preparation is 2 weeks. The holding time for sediments used for SPP creation is 8 weeks.

5.2 Labware Preparation

5.2.1 Prior to use, all glassware and non-metallic items will be cleaned using laboratory practices outlined in SOP QA-1113 *"Glassware Use and Care"*.

5.2.2 Teflon® bags, tubing, stainless steel stock pots, and stirring paddles are washed with laboratory safe soap and thoroughly rinsed with DI water.

5.3 Elutriate Preparation

Elutriates are created by mixing sediment and water, a 1:4 ratio by volume, for a period of 30 minutes. After mixing, the mud and water is left to settle for a 1 hour period. Once settled, the completed elutriate solution may be used for chemical testing, biological testing associated with the suspended particulate phase, or both. Typically, only 50% of the liquid used to make the elutriate is recoverable after the settling period. In order to provide enough solution for testing mixture is recovered purposes, double the amount of water and sediment necessary for the final volume of solution desired should be used. See Section 7.2 for information on calculating required volumes.

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Both the SPP solution and elutriate chemical solutions may be generated at the same time. However, water for chemical analysis must be used to create the solution within 7 days of sampling. Water for SPP solution creation may be used up to 14 days after sampling.

5.3.1 Place the stainless steel stock pot under the stirring apparatus and secure the stainless steel paddle bar into the pneumatic drill located above.

5.3.2 Measure out the desired amount of water and add it to the stock pot.

5.3.3 Measure out the homogenized sub-sample of sediment to be added to the stock pot. This sediment must be screen sieved to a minimum of 2mm prior to use.

5.3.4 Open the valves from the compressor, allowing the pneumatic drill to begin to stir. Slowly add all the sediment to the stock pot while the drill is spinning. This action aids in the suspending and mixing of the sediment.

5.3.5 Move the valves from the compressor to adjust stirring speed. The stirring speed should be high enough to maintain suspension and thorough mixing of the water and sediment, but low enough to avoid the creation of a vortex. Allow mixing to occur for 30 minutes.

If the sediment is settling during the mixing process, then manual agitation and stirring may occur every 10 minutes.

5.3.6 After 30 minutes of mixing, close the valve to the compressor to stop the drill. Detach the paddle bar from the drill and move the mixture to a secondary location. Allow the mixture to settle for 1 hour.

5.4 Elutriate Siphoning and Handling

Once the sediment and water mixture has settled for 1 hour, the top layer of the mixture is siphoned off using a Teflon® tube. This clearer supernatant is used for the Suspended Particulate Phase (SPP) toxicity testing solution and/or elutriate chemical analyses.

5.4.1 Toxicity Testing

If the elutriate solution is being used for SPP biological toxicity testing, it must be used within 24 hours of preparation. If the elutriate for SPP biological testing is not used immediately, then it must be completely mixed prior to use.

The only elutriate solution that may be centrifuged for clarity is that which is associated with bivalve testing. Solutions may be centrifuged for 10 minutes at 1000 g (1925 rpms).

In cases where the salinity of the dredging site water is detrimental to the health of the test organism (too low), the dredging site water should be adjusted with EDTA free commercially available sea salts until the target salinity is obtained. Deionized water may be used to dilute the site water should the salinity be too high. The salt-amended site water should be adjusted using dilute HCl or NaOH to its original pH. Salinity adjustments should be performed prior to preparation of the 100% SPP

5.4.2 Chemical Analysis

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If the elutriate solution is being used for chemical analysis, it must be passed through a pre-cleaned 0.45 micron filter prior to analysis. Alternate clarification, by means of centrifugation, must be approved by the client and governing agencies prior to implementation.

5.5 Calibration and standardization are not applicable components of this method and procedure.

6.0 Quality Control Requirements

Acceptance criteria and detection limits are not applicable components of the quality control requirements for this Standard Operating Procedure.

7.0 Calculations and Reporting

7.1 Record the following information on the Elutriate Preparation and Use Log (see Figure 1):

- Date
- Study number
- Client / Project
- Sample ID
- Sediment ID / Water ID
- Volume of sediment / overlying water
- Mixing time start / end
- Hand mixed (yes / no)
- Centrifuged (yes / no)
- Sub-sampling for additional testing information
- Quantities sub-sampled
- Preparer's initials
- Any additional notes

7.2 To calculate the correct elutriate solution, double the 1:4 sediment to water volume using the following equations:

$S = (2V / 5)$ and $W = (2V - S)$

Where:

V = Total volume needed
S = Volume of sediment
W = Volume of water

8.0 Corrective Actions

8.1 In the event that an element of the sample preparation fails to meet the requirement of this protocol, the deviation must be documented and the Laboratory Manager notified. A Corrective Action Report (CAR) may be initiated.

8.2 Troubleshooting

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If the stirring apparatus is spinning at a rate that creates a vortex in the center of the mixture, then the mechanism must be turned down to ensure sediment is not being drawn to the center of the vessel.

9.0 Health and Safety

As with all samples, gloves should be worn when handling sediment samples and chemicals. It is advisable to wear a lab coat to protect clothing. Full body coveralls are also available. Assay and sample disposal will be conducted using procedures to minimize potential pollution of surface and groundwaters, soils, and sediments.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedures outlined in this SOP.

10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification will be based upon a review of the analyst's demonstrated abilities.

11.0 Waste Management and Pollution Prevention

Unused portions of samples are disposed of only after completion of sample analysis, submission of the final report and contracted sample retention times have been achieved. All conditions of disposal and all records and correspondence concerning the final disposition of the physical sample shall be recorded and retained. Records shall indicate the date of disposal and the name of the individual who performed the task.

11.1 Aqueous Samples

After testing, aqueous samples will be disposed of through ESI's aqueous waste UV treatment system. This includes all unused samples, diluted samples and dilution waters that are not preserved. Access points for aqueous waste disposal in compliance with state and federal regulations are located throughout the laboratory and include streams for acidified samples, alkaline samples, and unpreserved samples. Any samples found to be toxic may require further testing or treatment in order to determine proper disposal technique to assure compliance with all local, state and federal regulations.

11.2 Solid Materials

ESI is a fully licensed and approved hazardous waste generator and storage facility. Waste storage, transfer and disposal will be in compliance with NH DES and US EPA waste disposal regulations. Non-hazardous solid materials, sediments and soils will be disposed of through an appropriate waste disposal site. All non-hazardous solid waste will be drained of as much free liquid as possible. The remaining sample is removed from its original container and placed with other non-hazardous waste samples in a 55 gallon metal drum.

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Once full, these containers will then be transferred to an approved waste disposal facility. This transfer is recorded on a waste manifest and they are retained for a minimum of 3 years.

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TITLE: Preparation of Suspended Particulate Phase (SPP) and Elutriate Solutions for Marine Sediment Evaluations, based on Region II Guidelines

Figure 1. Elutriate Preparation & Use Log

ELUTRIATE PREPARATION & USE LOG

Date: ESI Study:

Client: Project:

VOLUMES

Sample ID

Amount of Sediment L

Amount of Overlying Water L

Preparer's Initials

TIMES

Time Mixing Started

Hand Mixed Every 10 Minutes? Yes No

Time Mixing Stopped

Time Elutriate Siphoned Off

Centrifuged? Yes No

Lab Code	Sediment ID	Overlying Water ID

Lab Code	Elutriate Use	Volume

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Title: Sediment Extraction for Organic Compounds - Region II Testing

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	A. MacKinnon	01/2018

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Title: Sediment Extraction for Organic Compounds - Region II Testing

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the procedure for extracting sediment samples for organic compounds using elements of US EPA method 3570. This protocol is designed for use with samples from US EPA Region II and the New York District ACOE. The procedure for florisil column cleanup uses US EPA method 3620 and the procedure for silica gel column cleanup uses US EPA method 3630. The procedure for sulfur cleanup uses US EPA method 3660B and the procedure for sulfuric acid cleanup uses US EPA method 3665A.

Homogenized samples are spiked with an appropriate surrogate solution and extracted with solvent in glass. The solvent extracts are collected and adjusted to the appropriate final volume, typically 0.2 to 2.0 mL, by ebullation on a warming bath, such as Meyer N-Evap, or using a K-D apparatus. The extract is stored in glass and refrigerated until GC or GC/MS analysis. Samples may require additional clean-up.

Cleanup options may include, but are not limited to:

Pesticides (PEST): Florisil column chromatographic cleanup (see section 5.2).
Polynuclear Aromatic Hydrocarbons (PAH): Silica gel column chromatographic cleanup (see section 5.3).
PCB Congeners (CGR) and Aroclors (ARO): Treatment with concentrated sulfuric acid (see section 5.4).

2.0 Definitions

See Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Method 3510C (SW-846). (This method should be consulted for interferences and pollution prevention.)

US EPA. 2002. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Method 3570. (SW-846).

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Methods 3620C and 3630C (SW-846).

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Methods 3660B (SW-846).

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Methods 3665A (SW-846).

US EPA. 1983. *Methods for Chemical Analysis of Water and Wastes.* Method 608.

US EPA. 1988. *Statement of Work for Organic Analysis, Multi-Media Concentration.* Pesticide D-11.

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US EPA. 1985. *Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry.* Method 680.

Agilent Technologies. 1999. *Hardware Manual.* 5973 Network Mass Selective Detector.

Agilent Technologies. 1997-2000. *Operation Guide.* Agilent 7683 Automatic Liquid Sampler.

Agilent Technologies. 2000. *Operating Manual.* Agilent 6890 Series Gas Chromatograph.

Agilent Technologies. 1989-2001. *G1701DA Version D.00.00.38 19-Nov 2001.* Environmental ChemStation.

Department of Defense (DoD), Department of Energy (DoE). 2017. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.1.

4.0 Materials and Apparatus

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable logbook), and expiration date. In-house stock standard solutions may be prepared from neat stocks or purchased.

4.1 Reagents and Standards

Acetone, pesticide grade
Cyclohexane, pesticide grade
Florisil, activated at 675°C for 4 hours
Hexane, pesticide grade (typically used as solvent for PCB Aroclors (ARO) and pesticide extractions)
Methylene chloride, pesticide grade (typically used as solvent for PCB Congeners (CGR) and semi-volatile organic compound (SVOC) extractions)
Pentane, pesticide grade
Silica gel, activated at 130°C for 16 hours
Sodium sulfate, heated at 400°C for 4 hours to remove phthalates and other interfering organic substances
Pesticide surrogate and spike standards, provided by Supelco or other acceptable vendors (see Appendix A: Part 1 for standard preparation instructions)
Congener surrogate and spike standards, provided by Ultra or other acceptable vendors (see Appendix A: Part 2 for standard preparation instructions)
PAH surrogate and spike standards, provided by Environmental Consulting and Supplies (ECS) or other acceptable vendors (see Appendix A: Part 3 for standard preparation instructions)
Stock semi-volatile organic standards, provided by ECS or other acceptable vendors (see Appendix A: Part 4 for standard preparation instructions)
PCB aroclor surrogate and spike standards, provided by Restek and Ultra or other acceptable vendors (see Appendix A: Part 5 for standard preparation instructions)

4.2 Equipment and Supplies

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250 mL glass Boston bottles
Analytical balance, capable of weighing to 0.01 g
Copper powder
Evaporation containers (e.g. centrifuge test tubes)
Florisil
Gas-tight volumetric syringes
Glass chromatography columns
Glass extract collection vials 2 mL with Teflon lined lids
Glass funnels
Kimwipes®
Kuderna-Danish apparatus
Meyer N-Evap Analytical Evaporator
Mortar and pestle
Silanized glass wool
Silica Gel
Water/Steam bath (for K-D solvent evaporation)
Zymark TurboVap Evaporator

5.0 Methods and Procedures

Samples should be stored in glass jars with Teflon lined caps. Sediment samples must be refrigerated at 4±2°C until extracted. It is recommended that extractions be performed as soon as possible, but must be done within 14 days (possibly longer if kept frozen).Aqueous samples should be collected in 1 L glass jars with Teflon lined caps. Remove known free residual chlorine with ascorbic acid. Adjust pH to 5 - 9 SU if needed. Store samples at 4±2°C. Holding time for aqueous samples is 7 days. Samples should be stored in glass jars with Teflon lined caps. Tissue samples must be frozen until extracted. It is recommended that extractions be performed as soon as possible, but should be done within 360 days.

5.1 Extraction Procedure

Ensure all glassware is clean and dry and thoroughly solvent rinse all glassware, mortar and pestle with the appropriate solvent before proceeding with extractions.

5.1.1 Measure and add approximately 10 to 20 g dry weight (depending on the project) of homogenized sample into mortar. Use sodium sulfate to grind mixture until sample turns to a fine, dry, free-flowing powder. Start with approximately 5 g of sodium sulfate and add more as needed. Using a funnel, transfer the sample from the mortar into a labeled extraction vessel with a Teflon lined cap.

Note that this step deviates from the order of procedures in method 3570. Method 3570 requires the addition of sodium sulfate to the extraction vessel before the sample and does not require the sodium sulfate and sample to be mixed in a mortar prior to being transferred into the extraction vessel.

5.1.2 Unless otherwise specified, 5 g sodium sulfate will serve as the laboratory blank and the Laboratory Control Sample (LCS).

5.1.3 Using a gas-tight volumetric syringe, carefully add the appropriate amount of surrogate solution to each sample, including the blank and LCS. Add the appropriate

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amount of spiking solution to the LCS, and to the Matrix Spike (MS), Matrix Spike Duplicate (MSD), and Matrix Spike Triplicate (MST) samples when available. Spiked samples must be spiked at 3-5x the reporting limit. See appendix B for the appropriate reporting limits. The reference/control sediment will be used to perform MS/MSD/MST for sediment analysis. Site water will be used to preform aqueous analysis and pretest tissue will be used to perform tissue analysis.

Note that the amount of surrogate and spike compounds added to the soil deviates from Method 3570 specification of 50 µg and not all standards are made in methylene chloride. See Appendix A for standard concentrations and recipes.

5.1.4 Add approximately 50 mL of solvent (see Appendix A for appropriate extraction solvent) to all sample vials and shake each vial for 2 minutes. If required, samples may be rotated end over end for at least 4 hours for the first extraction. Let settle for 10 minutes and if necessary, vials may be centrifuged for 15 minutes at 500 rpm.

Note that volume and type of solvent used for extraction deviates from method 3570 specification of 12 mL of methylene chloride.

5.1.5 Decant extract into a glass vial for concentration. If necessary, extract can be filtered through a glass funnel containing sodium sulfate with glass wool plug or filter paper into a glass vessel. Sodium sulfate must be pre-rinsed and wetted with appropriate solvent prior to use. Once sample has been filtered, rinse sodium sulfate with appropriate solvent immediately, ensuring the sodium sulfate does not go dry. Before recapping vial, ensure the vial lip and threads are free from debris and wipe with a Kimwipe® as necessary.

5.1.6. Repeat section 5.1.4 and 5.1.5 twice more. Glass vessel should be capped while not actively being added to. After final extraction, rinse sodium sulfate with 2 - 3 mL of extraction solvent to ensure complete transfer.

5.1.7. Combine all three extracts and clean up as necessary (see sections 5.2 - 5.5). Adjust to the appropriate final volume (typically 0.2 mL to 2.0 mL) by ebullition on a warming bath, such as the Meyer N-Evap or K-D apparatus.

If K-D is used, add Teflon boiling stick or other appropriate material to the concentration vessel and attach micro-Snyder column Pre-wet Snyder column with 0.5 mL of extraction solvent. Place in a water bath maintained at a constant temperature, approximately 60 - 65°C. Temperature and position of K-D apparatus should be adjusted so the solvent boils evenly, the Snyder column balls chatter and the columns do not flood. Concentrate extract until about 1 mL is remaining.

5.1.8 Let the final extract cool (and drain if K-D was used) then transfer to a screw cap Teflon lined septum glass vial for analysis. Rinse the evaporation container with solvent for complete transfer, and prepare the exact final volume with a gas tight syringe. See Appendix A for appropriate final volumes. If the sample residue prevents concentrating to the desired volume, then adjust the final volume upward with solvent and record to the nearest 0.01 mL.

5.1.9 Extract condition and preparation data are verified by the laboratory supervisor and then transferred to the organics extract refrigerator maintained at 4±2°C until time

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of analysis.

5.2 Florisil Column Cleanup

Florisil columns may be useful in cleaning up pesticide extracts. Following the florisil column cleanup, proceed to finalize the extract volume and prepare for analysis (see steps 5.1.7 through 5.1.9).

5.2.1 Prepare a Florisil column for each extract using a 10 mL glass chromatography column fitted with silanized glass wool and a sodium sulfate plug at the bottom. Fill the column with 6 to 7 cc of florisil and tap the column gently to settle the powder. Insert a sodium sulfate plug into the top of the column.

5.2.2 Secure the column on a pipette stand in a fume hood. Pre-clean the column with approximately 25 mL of $MeCl_2$ and discard to waste. Pre-elute the column with approximately 25 mL of hexane and allow to drain into a waste container to be discarded. Keep the column wet with solvent above the sodium sulfate.

5.2.3 Transfer all or a known portion of the sample extract to the top of the florisil column using a gas tight syringe. Record the amount of sample (mL) taken for the cleanup. Add sufficient hexane to the sample vial to rinse it, then transfer rinse to the column. Elute the column with 50 mL of $MeCl_2$. Collect the eluate in a clean, labeled, solvent rinsed evaporation container. Extracts must be exchanged into hexane before the extract is prepared for analysis.

5.3 Silica Gel Column Cleanup

Silica gel columns may be useful for PAH extracts. Following the silica gel column cleanup, proceed to finalize the extract volume and prepare for analysis (see steps 5.1.7- 5.1.9).

5.3.1 Prepare a silica gel column for each extract using a 10 mL glass chromatography column fitted with silanized glass wool and a sodium sulfate plug at the bottom. Fill the column with 6 to 7 cc of silica gel and tap the column gently to settle the powder. Insert a sodium sulfate plug into the top of the column.

5.3.2 Secure the column on a pipette stand in a solvent delivery system in a fume hood. Pre- clean the column with approximately 25 mL of $MeCl_2$ and discard to waste. Pre-eulte the column with 25 mL of pentane into a waste container to be discarded. Keep the column wet with solvent above the sodium sulfate.

5.3.3 Transfer all or a known portion of the sample extract to the top of the silica gel column using a gas tight syringe. Extracts must be exchanged into hexane before being transferred to the column. Record the amount of sample (mL) taken for cleanup. Add sufficient hexane to the sample vial to rinse, then transfer the rinse to the column. Elute the column with 50 mL of 1+1 $MeCl_2$/pentane. Collect the eluate in a clean, labeled, solvent rinsed evaporation container.

5.4 Sulfuric Acid Cleanup

Sulfuric acid is used to clean up CGR and aroclors. Any column cleanup should occur before the acid cleanup. Extracts must be exchanged into hexane before adding any acid. Add approximately 0.5 mL of concentrated sulfuric acid to the extract, cap and shake. Allow the

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extract to settle for at least one hour, where the extract will separate into two distinct layers. Carefully pull off the top layer (the organic layer) using a gas tight syringe. Transfer to clean vial for analysis. Be very careful not to get any acid in the extract for analysis. Record the extract volume recovered.

5.5 Copper Cleanup

Copper is used to remove sulfur from sample extracts. The copper must be very reactive prior to use. All oxides must be removed so the copper is shiny and bright. If the copper does not look this way, then it needs to be reactivated by rinsing with dilute HNO_3 acid, Milli-Q® water, and then acetone. Approximately 2 g of cleaned copper is added to the sample extract and agitated for a minimum of one minute. Copper should be added to extract until there is no sulfur left to react with the copper (copper will stop turning black once all sulfur has been removed). Once this has been achieved, the extract should be removed from the copper and transferred to a clean vial for analysis.

6.0 Quality Control Requirements

Quality control elements may be added, removed, or altered to meet specific program or project requirements. Alternate acceptance criteria may be set for specific programs or projects, and reporting limits are specific to each parameter and client protocols. For DoD-specific projects see the QSM 5.1, as appropriate.

Note that all QC elements and samples must be spiked with a surrogate standard.

6.1 Demonstrations of Capability (DOC)

An analyst must complete a DOC before working independently on this procedure and yearly thereafter. A DOC is also required with any changes to instrument, method, or analyst. To perform a DOC, four replicates of a prepared quality control sample must be prepared. DOC samples must meet LCS acceptance criteria. For more information on DOCs see SOP QA-1108 “Training”.

6.2 Method Detection Limit (MDL)

The MDL is calculated using the appropriate student’s t-parameter value multiplied by the standard deviation of 8 or more replicates of spiked samples or standards over the course of the year. MDLs are performed annually and with changes in instrument or method. The calculated MDL must be lower than the reporting limit.

The t-parameter value is from a one-tailed Student’s t-Test at the 99% confidence interval using n-1 degrees of freedom, with “n” being the number of replicates.

The most recent MDL study can be found in the inorganics quality control directory on the ESI network. For more information on MDLs see SOP QA-1111 “Determination of Method Detection and Reporting Limits”.

6.3 Limit of Detection (LOD)

LOD determination will be conducted on a quarterly basis. For a single-analyte standard, the

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LOD is a single quality control sample that is approximately 2 to 3 times the MDL but no greater than the reporting limit. For a multi-analyte standard, the LOD is a single quality control sample that is approximately 1 to 4 times the MDL but no greater than the reporting limit.

The apparent signal to noise ratio at the LOD must be at least 3 or for methods that do not produce a signal to noise the LOD result must be at least 3 standard deviations greater than the mean method blank.

6.4 Limit of Quantitation (LOQ)

LOQ confirmation will be conducted on a quarterly basis. The LOQ is a single quality control sample no more than 1 to 2 times the reporting limit. The LOQ standard acceptance criteria is 30-150%. For more information on LODs and LOQs see SOP QA-1147 “Determination of LOD and LOQ Quality Control Samples”.

6.5 Blank, LCS, MS/MSD/MST, Standard Reference Material (SRM), and Relative Standard Deviation (RSD)

6.5.1 A blank, LCS, SRM and MS/MSD/MST will be analyzed every batch or every 20 samples, whichever is more frequent.

6.5.2 Acceptance criteria for all blanks is less than the reporting limit. Reporting limits can be found in Appendix B.

6.5.3 Acceptance criteria for LCS recovery is 50-150%. The LCS will be spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix B.

6.5.4 Acceptance criteria for MS/MSD/MST recovery is 50-150%. Acceptance criteria for RSD between MS/MSD/MST samples is 50%. MS/MSD/MST should be performed at a rate of 1 per 20 samples of 5% frequency. For sediment analysis reference sediment will be used to perform all MS/MSD/MST samples. For tissue analysispretest tissue will be used to perform all MS/MSD/MST samples. The MS/MSD/MST samples will be spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix B.

6.5.5 Acceptance criteria for surrogate standard recovery is 30-150%. All applicable surrogate standards must be within the acceptance range to be considered acceptable. If the surrogate recoverys fall outside of the acceptable range the client must be notified and the sample must be re extracted.

6.5.6 Acceptance criteria for SRM recovery is 50-150%. The SRM is evaluated for analytes at >3x the reporting limit. Reporting limits can be found in Appendix B.

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6.6 Interferences

Solvents, standards, and labware may be contaminated with artifacts that would affect results. Phthalate esters can pose a contamination issue with all analytes not just phthalate esters themselves.

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7.0 Calculations and Reporting

7.1 The following information should be recorded when performing a sediment extraction for organic compounds:

- Date
- Analyst's initial
- Batch
- Sample ID, with dilution factor if applicable
- Sample code / standard code
- Solvent lot ID
- Surrogate ID and amount, with dilution factor (if applicable)
- Spike ID and amount, with dilution factor (if applicable)
- Initial weight of wet sample
- Final volume
- Volume taken for cleanup
- Volume of cleaned sample
- Method
- Balance identification number
- Filter paper lot number
- Silica gel, florisil, and glass wool lot numbers (if applicable)
- Notes, if any

7.2 Calculations

Calibrate the system software as per the Agilent manuals referenced in Section 3.0. When reasonable, use the system software to calculate the values for QC elements and samples. Review the data to ensure it is correct and that all quality control requirements have been met. When needed, manually integrate the values according to ESI SOP QA-1132 "Manual Chromatographic Peak Integration".

7.2.1 Calculate LCS/Surrogate Recovery as follows:

$$\frac{\text{LCS/SUR Concentration}}{\text{Concentration Added}} \times 100 = \%\text{Recovery}$$

7.2.2 Calculate %RSD as follows:

$$\frac{\text{Std}(\text{Result 1 - Result 2})}{\text{Avg}(\text{Result 1 - Result 2})} \times 100 = \%\text{RSD}$$

7.2.3 Calculate Spike Recovery as follows:

$$\frac{\text{Spiked Sample Concentration - Sample Concentration}}{\text{Concentration Added}} \times 100 = \%\text{Recovery}$$

8.0 Corrective Actions

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8.1 Data Qualification

8.1.1 If DOCs, MDLs, LODs, or LOQs do not meet acceptability criteria, then repeat until acceptability criteria is met.

8.1.2 If the LCS does not meet percent recovery acceptance criteria, then they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (using the “J” qualifier).

8.1.3 If samples analyzed with blanks are not meeting acceptance criteria, then they may be reanalyzed or reported with qualification (using the “B” qualifier).

8.1.4 If matrix spike duplicate and triplicate samples do not meet percent recovery acceptance criteria, then they may be reanalyzed or reported with qualification (using the “J” qualifier). If the sample result is less than 5 times the reporting limit, then the RSD does not need to be evaluated.

8.1.5 If the MS recovery is not within acceptance criteria, then samples may be reanalyzed or reported with qualification (using the “J” qualifier). If the sample result is greater than 5 times the spike amount, then the percent recovery need not be calculated.

8.1.6 If the surrogate recovery is not within the acceptable range, then the client is notified and the sample is re-extracted whenever possible. Sample matrix effect will be proven if the results obtained from the re-extracted sample show similar results as the first recovery. When this occurs, both results are reported with an explanation. If the results of the re-extracted sample are in the acceptable range, then there is a problem with the first extraction and only the compliant results are reported. If no additional sample can be obtained, then results are flagged in the report and a narrative is included qualifying the data.

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified.

- Quality and expiration date of reagents
- Cleanliness of containers, equipment, and labware
- Integrity of standards
- GC and/or GC/MS operation

9.0 Health and Safety

9.1 As with all samples, gloves and safety glasses should be worn when handling samples, reagents, and standards. It is necessary to wear a lab coat to protect clothing. All solvent usage must occur in properly working fume hoods.

9.2 Methylene chloride, pentane, and cyclohexane are extremely hazardous to everyone in the laboratory if handled improperly. It is important that all necessary safety precautions are

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followed.

9.3 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding Materials Safety Data Sheet (MSDS).

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptance criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP, recording all data as described in Section 7.1, and reviewing all QC results for compliance with acceptance criteria.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities and DOC performance.

11.0 Waste Management and Pollution Prevention

Unused non-hazardous sediment sample may be disposed of as general trash. Solvents not evaporated in a hood are collected and disposed of as hazardous waste in designated solvent water waste containers. Extracted tissue, once dried should not be hazardous but may be disposed of as hazardous waste. Extracts and standard residues are deemed hazardous and are disposed of as such. Minimize solvent use and prepare standards in amounts that will be used before expiration.

11.1 Solid Materials

Waste storage, transfer and disposal will be in compliance with NH DES and US EPA waste disposal regulations. Non-hazardous solid materials, sediments and soils will be disposed of through an appropriate waste disposal site. All non-hazardous solid waste will be drained of as much free liquid as possible. The remaining sample is removed from its original container and placed with other non-hazardous waste samples in a 55 gallon metal drum. Once full, these containers will then be transferred to an approved waste disposal facility. This transfer is recorded on a waste manifest and they are retained for a minimum of 3 years.

11.2 Hazardous Waste Samples

Hazardous waste samples must be handled by properly trained employees. Only the ESI hazardous waste coordinator may train ESI employees on handling hazardous waste. There are 3 main hazardous waste streams at ESI, "dilute metals acidic", "solvents" and "Lachat wastes". When a satellite container needs to be emptied, it is properly transported to the waste shed and added into the appropriately labeled container for that waste stream. Logbooks are kept as a record for volumes added to each drum of any specific waste stream and shipping manifests reflect when they are picked up for transport.

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Title: Sediment Extraction for Organic Compounds - Region II Testing

Appendix A

Pesticides			Congener			PAH		
Initial Volume: 20 g			Initial Volume: 20 g			Initial Volume: 20 g		
Final Volume: 1.0 mL			Final Volume: 1.0 mL			Final Volume: 1.0 mL		
Solvent: 1:1, Hexane:Acetone			Solvent: 1:1, $MeCl_2$: Acetone			Solvent: 1:1, $MeCl_2$: Acetone		
Surrogate - 100 µL			Surrogate - 100 µL			Surrogate - 100 µL		
Tetrachloro-m-xylene	0.5	µg/mL	198	2	µg/mL	o-terphenyl	10	µg/mL
Decachlorobiphenyl	0.5	µg/mL				2-fluorobiphenyl	10	µg/mL
			Spike - 50 µL					
Pesticide A Mix - 250 µL			8	4	µg/mL	Spike - 100 µL		
TCMX (SS)	0.08	µg/mL	18	4	µg/mL	naphthalene	10	µg/mL
alpha-BHC	0.08	µg/mL	28	4	µg/mL	acenaphthylene	10	µg/mL
gamma-BHC (Lindane)	0.08	µg/mL	44	4	µg/mL	acenaphthene	10	µg/mL
heptachlor	0.08	µg/mL	52	4	µg/mL	fluorene	10	µg/mL
endosulfan I	0.08	µg/mL	66	4	µg/mL	phenanthrene	10	µg/mL
dieldrin	0.16	µg/mL	77	4	µg/mL	anthracene	10	µg/mL
endrin	0.16	µg/mL	101	4	µg/mL	fluoranthene	10	µg/mL
4,4'-DDD	0.16	µg/mL	105	4	µg/mL	pyrene	10	µg/mL
4,4'-DDT	0.16	µg/mL	118	4	µg/mL	benzo[a]anthracene	10	µg/mL
methoxychlor	0.8	µg/mL	126	4	µg/mL	chrysene	10	µg/mL
209 (SS)	0.16	µg/mL	128	4	µg/mL	benzo[b]fluoranthene	10	µg/mL
			138	4	µg/mL	benzo[k]fluoranthene	10	µg/mL
Pesticide B Mix - 250 µL			153	4	µg/mL	benzo[a]pyrene	10	µg/mL
TCMX (SS)	0.08	µg/mL	170	4	µg/mL	indeno[1,2,3-cd]pyrene	10	µg/mL
beta-BHC	0.08	µg/mL	180	4	µg/mL	dibenz[a,h]anthracene	10	µg/mL
delta-BHC	0.08	µg/mL	187	4	µg/mL	benzo[g,h,i]perylene	10	µg/mL
aldrin	0.08	µg/mL	195	4	µg/mL			
heptachlor epoxide	0.08	µg/mL	206	4	µg/mL			
gamma-chlordane	0.08	µg/mL	209	4	µg/mL			
alpha-chlordane	0.08	µg/mL	49	4	µg/mL			
4,4'-DDE	0.16	µg/mL	183	4	µg/mL			
endosulfan II	0.16	µg/mL	87	4	µg/mL			
endrin aldehyde	0.16	µg/mL	184	4	µg/mL			
endosulfan sulfate	0.16	µg/mL						
endrin ketone	0.16	µg/mL						
209 (SS)	0.16	µg/mL						
Pesticide C Mix - 250 µL								
oxychlordane	0.08	µg/mL						
chlorpyrifos	0.08	µg/mL						
trans-nonachlor	0.08	µg/mL						
cis-nonachlor	0.08	µg/mL						
hexachlorobenzene	0.08	µg/mL						

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Title: Sediment Extraction for Organic Compounds - Region II Testing

PCB Aroclor			SVOC	
Initial Volume: 20 g			Initial Volume: 20 g	
Final Volume: 1.0 mL			Final Volume: 1.0 mL	
Solvent: 1:1, Hexane: Acetone			Solvent: 1:1, MeCl2: Acetone	
Surrogate - 100 µL			Surrogate - 1.0 mL	
Tetrachloro-m-xylene	0.5	µg/mL	2,4,6-tribromophenol	200 µg/mL
Decachlorobiphenyl	0.5	µg/mL	2-fluorophenol	200 µg/mL
			2-fluorobiphenyl	100 µg/mL
Spike - 125 µL			terphenyl-d14	100 µg/mL
Aroclor 1016	10	µg/mL	nitrobenzene-d5	100 µg/mL
Aroclor 1260	10	µg/mL	phenol-d5	200 µg/mL
			Spike - 1.0 mL	
			2-chlorophenol	200 µg/mL
			phenol	200 µg/mL
			4-chloro-3-methylphenol	200 µg/mL
			4-nitrophenol	200 µg/mL
			pentachlorophenol	200 µg/mL
			1,2,4-trichlorobenzene	100 µg/mL
			di-n-butylphthalate	100 µg/mL
			1,4-dichlorobenzene	100 µg/mL
			acenaphthene	200 µg/mL
			N-nitroso-di-n-propylamine	100 µg/mL
			2,4-dinitrotoluene	100 µg/mL
			pyrene	200 µg/mL
			naphthalene	100 µg/mL
			phenanthrene	100 µg/mL
			anthracene	100 µg/mL
			fluoranthene	100 µg/mL
			benzo(a)anthracene	100 µg/mL
			benzo(b)fluoranthene	100 µg/mL
			benzo(a)pyrene	100 µg/mL
			dibenzo(a,h)anthracene	100 µg/mL
			fluorene	100 µg/mL
			acenaphthylene	100 µg/mL
			chrysene	100 µg/mL
			benzo(k)fluoranthene	100 µg/mL
			indeno(1,2,3-cd)pyrene	100 µg/mL
			Benzo(k)fluoranthene	100 µg/mL
			benzo(g,h,i)perylene	100 µg/mL

Part 1 (See above for compounds in each standard)

Pesticide Surrogate Mix - 0.5 µg/mL
Stock Standard: Supelco Cat# 861284 200 µg/mL
Dilute 0.075 mL stock standard to final volume 30 mL with hexane

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Pesticide Mix A - 80/160 ng/mL
Stock Standard: Supelco Cat# 47977 0.5-5.0 µg/mL
Dilute 1.0mL stock standard to final volume 6.25 mL with hexane

Pesticide Mix B - 80/160 ng/mL
Stock Standard: Supelco Cat# 47977 0.5-1.0 µg/mL
Dilute 1.0 mL stock standard to final volume 6.25 mL with hexane

Pesticide Mix C - 80 ng/mL
Stock Standard: Individual component (see list in Appendix A) at 100 µg/mL from Crescent Chemical Company
Dilute 0.2 mL each stock standard to final volume 2.0 mL with hexane

Part 2 (See above for compounds in each standard)

Congener Surrogate - 2 µg/mL
Stock Standard: Ultra Cat# RPC-075AS 100 µg/mL
Dilute 0.6 mL stock standard to final volume 30 mL with 50:50 hexane:acetone

Congener Spike - 4 µg/mL
Stock Standards: Ultra Scientific Cat# RPC-EPA-2-1, RPC-030AS, RPC-073AS, RPC-099AS, RPC-075AS and RPC-168AS 100 µg/mL
Dilute 0.15 mL of each stock standard to final volume 3.75 mL with hexane

Part 3 (See above for compounds in each standard)

PAH Surrogates - 10 µg/mL
Stock Standards: 2-Fluorobiphenyl Chemservice Cat #N-10359-16 Neat
O-terphenyl Chemservice Cat# S-12693X4-1ML 1000 µg/mL
Intermediate Stock Standards: 2-Fluorobiphenyl- 50 mg stock standard to final volume 25 mLwith MeCl2 2000 µg/mL
O-terphenyl - 1.0 mL stock standard to final volume 10 mL with acetone 200 µg/mL
Second Intermediate Stock Standard: 2-Fluorobiphenyl - Dilute 1 mL intermediate standard to final volume 10 mL with acetone 200 µg/mL
2-Fluorobiphenyl: Dilute 2.0 mL second intermediate standard to final volume 40 mL with Acetone 10 µg/mL
O-terphenyl: Dilute intermediate standard to final volume 40 mL with acetone 10 µg/mL

PAH Spike Mix- 10 µg/mL
Stock Standard: Environmental Consulting and Supplies, Inc Cat# ECS-A-032, ECS-B-032, ECS-N-032 2000 µg/mL
Intermediate Stock Standard: Dilute 2 mL of stock standard to final volume 40 mL acetone 100 µg/mL
Dilute 1.0 mL of intermediate stock standard to final volume 10 mL acetone

Part 4 (See above for compounds in each standard)

SVOC Surrogate - 100-200 µg/mL
Stock Standard: ECS Cat# ECS-N-027 1000-2000 µg/mL
Dilute 2 mL stock standard to final volume 20 mL with acetone

SVOC Spike Mixes - 100-200 µg/mL
Stock Standards: Acid Mix ECS Cat #ECS-N-004 2000 µg/mL
Base/Neutral Mix ECS Cat# ECS-N-005 1000 µg/mL
PAH Mix ECS Cat# ECS-N-032 2000 µg/mL
Acid Mix - Dilute 2.0 mL stock standard to 20 mL with acetone (200 µg/mL)
Base/Neutral Mix - Dilute 2.0 mL stock standard to final volume 20 mL with acetone (100 µg/mL)
PAH Mix - Dilute 2.0 mL stock standard to final volume 40 mL with acetone (100 µg/mL)

Part 5 (See above for compounds in each standard)

PCB Surrogate Mix - 0.5 µg/mL
Stock Standard: Supelco Cat# 861284 200 µg/mL
Dilute 0.075 mL stock standard to final volume 30 mL with hexane

PCB Aroclor Spike Mix - 10 µg/mL
Stock Standards: Aroclor 1016 Ultra Scientific, Cat#PP-282-1 100 µg/mL
Aroclor 1260 Ultra Scientific Cat#PP-362-1 100 µg/mL

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Aroclor 1016 1.0 mL stock standard to final volume 10.0 mL with hexane
Aroclor 1260 1.0 mL stock standard to final volume 10.0 mL with hexane

Appendix B Reporting Limits

Analyte	Tissue (ng/g)	Sediment (ng/g)	Water (µg/L)
Aldrin	**4.3**	**1**	**0.4**
Dieldrin	**1.5**	**1**	**0.20**
a-Chlordane	**2.7**	**1**	**0.030**
Trans Nonachlor	**2.7**	**1**	**0.010**
Heptachlor	**2.7**	**1**	**0.010**
Heptachlor Epoxide	**2.7**	**1**	**0.010**
Endosulfan I	**0.32**	**1**	**0.010**
Endosulfan II	**0.32**	**1**	**0.010**
Endosulfan Sulfate	**0.32**	**1**	**0.010**
4,4-DDT	**1**	**1**	**0.04**
2,4-DDT	**1**	**1**	**0.010**
4,4-DDD	**1**	**1**	**0.010**
2,4-DDD	**1**	**1**	**0.010**
4,4-DDE	**1**	**1**	**4.0**
2,4-DDE	**1**	**1**	**100**

Analyte	Tissue (ng/g)	Sediment (ng/g)	Water (µg/L)
PCB 8	**0.4**	**1**	**0.002**
PCB 18	**0.4**	**1**	**0.002**
PCB 28	**0.4**	**1**	**0.002**
PCB 44	**0.4**	**1**	**0.002**
PCB 49	**0.4**	**1**	**0.002**
PCB 52	**0.4**	**1**	**0.002**
PCB 66	**0.4**	**1**	**0.002**
PCB 87	**0.4**	**1**	**0.002**
PCB 101	**0.4**	**1**	**0.002**
PCB 105	**0.4**	**1**	**0.002**
PCB 118	**0.4**	**1**	**0.002**
PCB 128	**0.4**	**1**	**0.002**
PCB 138	**0.4**	**1**	**0.002**
PCB 153	**0.4**	**1**	**0.002**
PCB 170	**0.4**	**1**	**0.002**
PCB 180	**0.4**	**1**	**0.002**
PCB 183	**0.4**	**1**	**0.002**
PCB 184	**0.4**	**1**	**0.002**
PCB 187	**0.4**	**1**	**0.002**
PCB 195	**0.4**	**1**	**0.002**

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PCB 206	**0.4**	**1**	**0.002**
PCB 209	**0.4**	**1**	**0.002**

Analyte	Tissue (ng/g)	Sediment (ng/g)	Water (µg/L)
Acenaphthene	**50**	**100**	**N/R**
Acenapthylene	**50**	**100**	**N/R**
Anthracene	**50**	**100**	**N/R**
Fluorene	**50**	**100**	**N/R**
Naphthalene	**50**	**100**	**N/R**
Phenanthrene	**50**	**100**	**N/R**
Benzo(a)anthracene	**50**	**100**	**N/R**
Benzo(a)pyrene	**50**	**100**	**N/R**
Benzo(g,h,i)perylene	**50**	**100**	**N/R**
Benzo(b)fluoranthene	**50**	**100**	**N/R**
Benzo(k)fluoranthene	**50**	**100**	**N/R**
Chrysene	**50**	**100**	**N/R**
Dibenzo(a,h)anthracene	**50**	**100**	**N/R**
Fluoranthene	**50**	**100**	**N/R**
Indeno(1,2,3-cd)pyrene	**50**	**100**	**N/R**
Pyrene	**50**	**100**	**N/R**
1,4-dichlrorbenzene	**3900**	**100**	**N/R**

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Title: Analysis of Semi-volatile Compounds by GC/MS - Region II Testing

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Draft Preparation	C. Sasso	01/2018

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1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the procedure used to analyze extractable Acid and Base Neutral (ABN) Compounds in water, soil, and waste matrices using gas chromatographic/mass spectrometric (GC/MS) EPA Methods 625 and SW-846 8270. This protocol is designed for use with samples from US EPA Region II and the New York District ACOE.

Specific analytes include those compounds on the EPA target compound list as well as aniline, benzidine, and azobenzene (see Appendix 1). Detection limits are at the parts per billion level or greater; references to ppm levels in this document refer to extract, and not to sample analyte concentrations. Instrumental procedures and their requirements as specified in referenced EPA methods will be described. Performance of this method is limited to analysts experienced in GC/MS operation and in the interpretation of gas chromatograms and mass spectra.

2.0 Definitions

Surrogate: An organic compound that is similar to analytes of interest in chemical composition, extraction and chromatography, but is not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

See ESI's Quality Assurance Manual, Section 13.0: Terms and Definitions.

3.0 Applicable Documents and References

US EPA. 1996. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* Third Edition. Method 8270 C. SW-846.

US EPA. 1983. *Methods for Chemical Analysis of Water and Wastes.* Method 625.

Agilent Technologies. 1999. *Hardware Manual*. Agilent 5973 Network Mass Selective Detector.

Agilent Technologies. 2000. *Operation Guide*. Agilent 7683 Automatic Liquid Sampler.

Agilent Technologies. 2000. *Operating Manual.* Agilent 6890 Series Gas Chromatograph.

Agilent Technologies. 2001. *G1701DA Version D.00.00.38 19-Nov 2001.* Environmental ChemStation.

Department of Defense (DoD), Department of Energy (DoE). 2017. *DoD Quality Systems Manual (QSM) for Environmental Laboratories.* Version 5.1.

SOP QA-1132 "Manual Chromatographic Peak Integration"
SOP QA-1367 "Extraction of Pentachlorophenol and other Semi-volatile Organic Compounds in Aqueous Samples"

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4.0 Materials and Apparatus

4.1 Reagents and Standards

All standards and reagents shall be ACS reagent grade or equivalent unless specifically noted. Each standard and reagent made shall be labeled with solution name, reference number (obtained from applicable log book), and expiration date. All new reagent lots are tested prior to use for contamination. New standards lots are checked against identical standards from an independent source to verify concentration and purity.

4.1.1 Methylene chloride ($MeCl_2$)

Pesticide grade or equivalent for diluting samples and standards. Checked for traces of target analytes. Avoid storage at high temperatures. Keep material dry and away from moisture. Released material will evaporate.

4.1.2 Internal Standards (IS)

Comprised of compounds: 1,4 Dichlorobenzene-d4, Napthalene-d, Chrysene-d12, Phenanthrene-d10, Acenapthene-d10, and perylene-d12. Store tightly capped out of the light.

4.1.3 Surrogate Standards (SS)

Comprised of compounds: phenol-d6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d5, 2-fluorobiphenyl, and p-terphenyl-d14 at 100/200 µg/mL for ABN and SVOC compounds. For PAH compounds, SS include o-terphenyl and 2-fluorobiphenyl. Store tightly capped at 4±2°C. For pentachlorophenol, 2,4,6-tribromophenol is used for the surrogate. Store tightly capped at room temperature.

4.1.4 50 ng/µL Decaflurotriphenylphosphine (DFTPP) GCMS Tuning Standard

A methylene chloride solution containing 50 µg/mL of DFTPP, 4,4'-DDT, pentachlorophenol, and benzidine. Used to check column performance and verify injection port inertness. Store tightly capped at 4±2°C.

4.1.5 Standard Stock Mixes

Standard Stock Mixes are purchased from reputable suppliers. These mixes arrive in sealed amber ampules may be used for up to one year or more when stored in the ampule and refrigerated at 4±2°C. See Appendix 1 for a list of compounds and mixes.

4.2 Equipment and Supplies

Agilent 6890/5973 GC/MS
Autosampler Agilent 7683
Data software package system
Gas tight syringes
Sample vials with screw caps
Analytical balance for weighing standards and solids

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Class A volumetric flasks as required
Refrigerator for storage of standards and extracts
Column

5.0 Methods and Procedures

Sample handling and extraction are performed according to the procedures detailed below. A tune check is performed using the DFTPP standard. The results are checked and saved to a file in the SVOC directory on the ESI network. A calibration is then run using standard(s) and the calibration curve is checked. Analysis of samples may only begin once the tune check and calibration curve passes the set quality control criteria.

Sample extracts are then assembled on the autosampler trays in accordance with the sample table information on the connected software for the GC/MS. The file numbers and sample information is recorded in the run log book. Once the data has been collected, peak integrations may be done, saved to the SVOC directory on the ESI network, printed, and then calculated.

5.1. Sample Handling

Samples should be collected in 1 L glass jars with teflon lined caps. Those with free residual chlorine should be treated with sodium thiosulfate. Prior to extraction, the samples are stored at 4±2°C in the sample management area. Holding time is 7 days from sampling for waters and 14 days from sampling for soils (up to one year if kept frozen).Tissue samples must be frozen until extracted. It is recommended that extractions be performed as soon as possible, but should be done within 360 days.

5.2 Sample Preparation

The sample preparation procedures for ABN or PAH extraction is described in ESI SOPs: QA-1367 “Extraction of Pentachlorophenol and other ABN Compounds in Aqueous Samples”, QA- 1377 “Tissue Extraction of Organic Compounds”, and QA-1390 “Sediment Extraction for Organic Compounds”.

In general, samples are serially extracted with $MeCl_2$, extracts are combined, dried over sodium sulfate and cleaned if necessary. The extract is concentrated to the desired final volume. Blanks and LCSs are extraced with every batch of samples; a set of matrix spikes (MS) are performed every 20 samples or by client request. Moisture content is determined for solid samples. When concentration to the desired final volume is achieved, the extracts are transferred to 2.0 mL vials with screw tops and teflon lined septa and are stored at 4±2°C when not under analysis. All extracts must be analyzed within 40 days..

5.3 Standard Stock Mixes

Standards are stable for 6 to 12 months if stored at 4°C. Stock standards are checked for signs of degradation or evaporation. Record standards in the appropriate standards log.

5.3.1 ABN Working Standards

Prepare ABN working standards by diluting the appropriate stock standards. Commercial ABN surrogate standard mix is diluted to the desired concentration. The

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ABN spikes (acid and base neutral) used in matrix spike and lab control samples are made from diluting commercial acid extractable and base/neutral spiking mixes. The initial multi point calibration is made from diluting commercial mixes which includes: PAH analyte mix, add-ons mix, base/neutrals mix, benzidines mix, and phenols mix. Dilutions are made in methylene chloride to achieve the desired concentrations. See Appendix 7 for a list of standards and concentrations.

5.3.2 Pentachlorophenol (PCP) Working Standards

Prepare PCP working standards by diluting the appropriate stock standards. Commercial PCP surrogate standard is diluted to the desired concentration. The initial multi point calibration is made from diluting a commercial standard. Dilutions are made in methylene chloride to achieve the desired concentrations. See Appendix 7 for a list of standards and concentrations.

5.3.3 Polycyclic Aromatic Hydrocarbons (PAH) Working Standards

Prepare PAH working standard by diluting the appropriate stock standard. Commercial PAH surrogate standard mix is diluted to the desired concentration. The initial multi point calibration is made from diluting commercial mixes which includes target PAH analyte mix. Dilutions are made in methylene chloride to achieve the desired concentrations. See Appendix 7 for a list of standards and concentrations.

5.4 Mass Spectrometer (MS) Tuning

In order for mass spectra to be comparable among laboratories, all MS instruments must be tuned similarly. The MS instruments must be tuned to meet the manufacturer's specifications using a suitable calibrant. Strict reference fragmentation criteria have been set for the tuning compound DFTPP (see Appendix 2). The mass spectrometer must be hardware tuned to meet the criteria. Further, the tune must be verified each day that analyses are made and every 12 hours of analysis time. More frequent checks may be made according to the client's Data Quality Objectives (DQOs). Specifically, the tune is checked in the following manner:

DFTPP at 50 ng/µL is analyzed using tune parameters that will be used for samples (see Appendix 3).

DFTPP mass spectrum obtained is evaluated against the criteria using the system software. A report is generated that includes the criteria table, mass spectrum of passing scan, tabulation of ion fragment abundances normalized to m/e 198, and the abundance of m/e 198. The passing spectrum is archived. Scans under the peak may be averaged, and background scans subtracted prior to evaluation. Benzidine and pentachlorophenol are monitored for tailing factors greater than 3, which would require possible GC/MS maintenance before proceeding with sample analysis. A text file of the passing tune report is saved.

5.5 Instrument Calibration

Instrument calibrations are run to obtain response factors from standards of known concentration. Multipoint calibrations determine the linearity of the response over a range of concentrations, as well as indicate instrument stability. It is appropriate to use the average

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of all the response factor points in calculating target concentrations when the response of a compound is found to be linear over the calibration range (the plot of area versus concentration as a straight line). Is it also appropriate to use the response factors of a single point in that range, as is done with continuing calibration standards. In most semi-volatile methods, response is linear within the calibration and sometimes well beyond it.

A five point (multipoint) initial calibration is run once to verify linearity and to obtain an acceptable average response factor over the calibration range for each analyte. Then each day afterwards, or according to project requirements, a continuing calibration check is run to verify the calibration.

5.5.1 Internal Standards

Working level standards for ABN compounds are prepared from standard stock mixes at as many as eight levels: 0.5, 2, 5, 10, 20, 40, 80, 100, 120, 160 and 200 ppm. Additional mid level standards are prepared as needed for continuing calibration check standards.

Quantitation is done using the internal standard method. For every sample that is run, a quantitation report is generated on the host computer. The ratio of target analyte response (e.g., area per nanogram) to internal standard response defines the response factor of that target analyte. See Section 7.3.1 for response factor calculation. The computer uses a target analyte's response factor from the initial calibration to calculate the analyte concentration in a sample extract. See Section 7.3.2 for target concentration calculation.

Internal standard containing 6 compounds is added to all standards and samples to provide a concentration of 40 µg/mL prior to analysis for analysis of ABN and SVOC compounds. For PCP and PAH analysis, the internal standard is added to all standards and samples to provide a concentration of 1ug/mL.

5.5.2 Initial Multipoint Calibration

5.5.2.1 An initial five to eight point calibration standard set is analyzed using standard levels between 0.5 and 200 µg/mL. Preparation of these standards is described in the Standard Stock Mixes section on page 4 and in more detail in Appendix 7. The standards must be run within a valid tune window, though not necessarily all in the same one.

5.5.2.2 Check for Method Compliance

Listed in Appendix 4 are the System Performance Check Compounds (SPCCs) and Calibration Check Compounds (CCCs) and their criteria as specified by EPA method 8270. SPCCs must have a minimum response factor of 0.05, and the CCCs have maximum Percent Relative Standard Deviation (%RSD) of 20%. Full compliance with EPA CLP criteria is desirable; however, the acceptable limits are always a function of the project Data Quality Objectives (DQOs).

Any CCCs not included in the list of analytes for a project do not need to be

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included in the calibration standards. A text file table containing the response factors for every compound in each of the standards and %RSD may be generated with the Agilent software. If the initial calibration meets acceptance criteria, the method may be used.

5.5.3 Continuing Calibration

Instrument calibration must be checked and updated prior to sample analysis every 10 samples or mid batch for batches under 20. This is done by the injection of a upper or mid-level calibration standard.

As with the initial calibration, the continuing calibration is checked for EPA method 8270 compliance with CCC and SPCC criteria (see Appendix 4). Compliance with EPA method 625 requires continuing calibration compounds to have response factors that deviate no more than 20% recovery to the initial calibration. Compliance with CLP limits is desirable, but compliance with the specific project-specific DQOs is required. Any exception to CLP criteria should be approved by the Laboratory Manager.

5.5.4 Instrumental Analysis

Detailed instrument operating conditions may be found in Appendix 3.

5.5.4.1 Screening

Sample extracts may be screened by Gas Chromatography- Flame Ionization Detector (GC-FID) prior to analysis in order to determine the level of contamination present and if a dilution is necessary. Dilutions are made to keep all targets within the calibration range. In order to avoid raising the detection limit unnecessarily, the rule for dilutions is that the most concentrated analytes must fall in the upper half of the calibration range of 80 - 160 ppm unless high levels of non-target compounds are present. While some contracts require that no analyte exceed 160 ppm, accurate quantitation occurs anywhere in an analyte's linear range. Remaining within the demonstrated linear range satisfies the data quality objectives of most projects.

5.5.4.2 GC/MS Analyses

After the instrument is tune checked with DFTPP and the calibration check is performed, high quality data is generated and substantiated by running the samples with associated blanks and spikes.

Operation of the GC/MS is as described in the manufacturer's manuals. Certain parameters are monitored to ensure that the analytes of interest are being detected and quantitated reliably. These parameters include quality control elements such as blanks, spikes, internal standard areas, and surrogate recoveries.

5.5.4.3 Internal Standards

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Areas of internal standards are monitored to check system stability. Samples must have internal standard areas 1/2 - 2 times those of the standard. Internal standard retention times must fall within 30 seconds of those in the standard. Upholding these criteria ensures that accurate concentrations are calculated from previously generated response factors. The time windows ensure that target analytes will be detected reliably. Failure to meet the requirements means the sample must be reanalyzed. Internal standard area trend files are maintained on the computer system, and are available for review.

5.5.4.4 Surrogates

The role of surrogates is to indicate extraction efficiency, and the presence of matrix effects. Failure to meet surrogate acceptance criteria may be indicative of a systems failure and should be monitored. If sufficient sample volume is available, re-extraction may occur. The re-extract either confirms the presence of a matrix effect or replaces the original aberrant extract. Ash matrix has been proven to interfere with surrogate recoveries; these are not re-extracted. Samples with matrix effects are also noted on their reports. Data from samples with insufficient material for re-extraction are considered in the context of other associated quality control data, then qualified accordingly. Trend files for water and soil surrogate recoveries are maintained on the computer and are available for review.

6.0 Quality Control Requirements

Quality Control elements may be added, removed, or altered to meet specific program or project requirements. Acceptance criteria shall be updated based on the associated control charts once sufficient data points have been collected (at least 20 data points) and yearly thereafter. Alternate acceptance criteria may be set for specific programs or projects. For DoD-specific projects reference QSM 5.1, as appropriate.

Note that all quality control elements and samples must be spiked with a surrogate standard. All quality control elements, samples, and calibration standards must be spiked with internal standards.

6.1 Demonstration of Capability (DOC)

An analyst must complete a DOC before working independently on this procedure and yearly thereafter. A DOC is also required with any changes to instrument, method, or analyst. To perform a DOC, four replicates of a prepared quality control sample must be prepared. DOC samples must meet LCS acceptance criteria. For more information on DOCs see SOP QA-1108 “Training”.

6.2 Method Detection Limit (MDL)

The MDL is calculated using the appropriate student’s t-parameter value multiplied by the standard deviation of 7 or more replicates of spike samples or standards. MDLs are performed annually and with changes in instrument or method. The calculated MDL must be lower than the reporting limit.

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The t-parameter value is from a one-tailed Student's t-Test at the 99% confidence interval using n-1 degrees of freedom, with "n" being the number of replicates.

The most recent MDL study can be found in the Quality Control directory on the ESI network. For more information on MDLs see SOP QA-1111 "Determination of Method Detection and Reporting Limits".

6.3 Limit of Detection (LOD)

LOD determination will be conducted on a quarterly basis. For a single-analyte standard, the LOD is a single quality control sample that is approximately 2 to 3 times the MDL but no greater than the reporting limit. For a multi-analyte standard, the LOD is a single quality control sample that is approximately 1 to 4 times the MDL but no greater than the reporting limit.

The apparent signal to noise ratio at the LOD must be at least 3. For methods that do not produce a signal to noise, the LOD result must be at least 3 standard deviations greater than the mean method blank.

6.4 Limit of Quantitation (LOQ)

LOQ confirmation will be conducted on a quarterly basis. The LOQ is a single control sample no more than 1 to 2 times the reporting limit. The LOQ standard acceptance criteria is 30-150%. For more information on LODs and LOQs see SOP QA-1147 "Determination of LOD and LOQ Quality Control Samples".

6.5 Blank, LCS, Matrix Spike (MS/MSD/MST) Samples, Standard Reference Material (SRM), Relative Standard Deviation (RSD)

6.5.1 Analyze a blank, LCS, MS, MS duplicate (MSD), MS triplicate (MST), and SRM every analytical batch or 20 samples, whichever is more frequent, or as required in the prepatory method.

6.5.2 Acceptance criteria for all blanks is less than the reporting limit. Reporting limits can be found in Appendix 8.

6.5.3 Acceptance criteria for LCS recovery is 50%-150%. The LCS will be spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix 8.

6.5.4 Acceptance criteria for spiked sample recovery is 50%-150%. The MS/MSD/MST samples will be spiked at 3-5x the reporting limit. Reporting limits can be found in Appendix 8.

6.5.5 MS/MSD/MST samples are extracted either per client request or per project requirements. Every attempt will be made to obtain enough sample to perform a matrix spike with 5% frequency. If a single sample needs to be extracted and there is not enough sample volume for a matrix spike, a synthetic sample, or LCS

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duplicate may be extracted and analyzed to provide 5% spike occurrence. For sediment analysis reference sediment will be used to perform all MS/MSD/MST samples. For tissue analysispretest tissue will be used to perform all MS/MSD/MST samples.

6.5.6 Acceptance criteria for surrogate standard recovery is 30-150%. All applicable surrogate standards must be within the acceptance range to be considered acceptable.If the surrogate recoverys fall outside of the acceptable range the client must be notified and the sample must be re extracted.

6.5.7 Acceptance criteria for standard reference material recovery is 50-150%. The SRM is evaluated for analytes at >3x the reporting limit. Reporting limits can be found in Appendix 8.

6.7 ICV, CCV, ICB and CCB

Note that the system calibration may be updated with the CCV and CCB, provided that they are followed by a CCV, CCB, and reporting limit standard re-analysis.

6.7.1 Initial calibration consists of a blank and a minimum of five standards. Acceptance criteria for initial calibration requires the relative standard deviation of response factors be less than 20%.

6.7.2 ICV and ICB are performed following analysis of initial calibration standards. Acceptance criteria for the ICV requires the recovery compared to the initial calibration be ±20%.

6.7.3 CCV and CCB are performed at the beginning of every analytical session, as well as every 10 samples or mid batch for batches of less than 20 samples. Acceptance criteria for the CCV requires the system performance check compounds (SPCC) and the calibration check compounds (CCC) to have an recovery of ±20% compared to the initial calibration

6.8 Tune MS with DFTPP; see Appendix 2 for DFTPP standard criteria.

6.10 See Appendix 5 for internal standard acceptance criteria.

6.11 Interferences

The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510. Method 3510 is preferred over Method 3520 for the analysis of these classes of compounds. However, the recovery of phenols may be optimized by using Method 3520, and performing the initial extraction at the acid pH. Refer to EPA method 3500 for a full list of interferences.

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7.0 Calculations and Reporting

7.1 The following information should be recorded when performing an analysis of ABN compounds by GC/MS:

- Date
- Analyst's initial
- Batch
- Sample ID, with dilution factor if applicable
- Standard ID, with dilution factor if applicable
- Data file name
- Acquisition number
- Notes, if any

7.2 Reporting

The quantitation report generated on the host computer for every sample that is run lists the retention times and areas for all peaks which match the standard library criteria. For GC/MS, the criteria includes elution time within a narrow window and a match with standard spectra. The concentration is also reported, based on response factors calculated from the daily standard. The concentration is the value for analytes in the extract itself; it does not account for dilutions or exact volume/mass of sample used to prepare the extract. Retention time of hits are compared to those of the standard, and questionable integrations and spectra are checked by the analyst. The chromatogram is inspected for unintegrated peaks. Large nontargets are identified by library searching.

7.2.1 The results to be listed on the final report are calculated using the system software generated analyte value, analyst provided instrument dilution factor, laboratory provided sample volume or weight, moisture content if needed and extract final volume(s). See Section 7.3.3 for this calculation.

7.2.2 The concentrations for solids are reported on a dry weight basis. A draft of the final report is printed in the laboratory for validation by the analyst. This is forwarded to the Laboratory Manager for final review and sign-off before generation of the finished report for the client.

7.3 Calculations

7.3.1 Calculate the Response Factor as follows:

$$\text{Response Factor} = \frac{(\text{Area Target})(\text{Interal Standard Concentration})}{(\text{Area Internal Standard})(\text{Target Concentration})}$$

7.3.2 Calculate the Target Concentration as follows:

$$\text{Target Concentration} = \frac{(\text{Area Target in Sample})(\text{Interal Standard Concentration})}{(\text{Area Internal Standard in Sample})(\text{Analyte Response Factor})}$$

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7.3.3 Calculate the Report Value as follows:

$$\text{Report Value} = \frac{\text{(Inst Value) (Final Volume) (Prep DF) (Primary ABN Volume)}}{\text{(AMT Extracted) (\%Dry) (ABN Aliquot)}}$$

Where:

AMT Extracted = The inital volume of sample extracted in grams or mL.
%Dry = 1 - moisture content of the solid sample. (Water = 1)
ABN Aliquot = Sub sample of primary ABN volume diluted to a final volume.
Prep DF = Dilution of the sample run on the GCMS.
Primary ABN Volume = Sample extract at an exact volume.
Inst Value = Lowest concentration point of inital calibration.

7.3.4 Calculate Standard Recovery as follows:

$$\frac{\text{Standard Concentration}}{\text{Concentration Added}} \times 100 = \text{\% Recovery}$$

7.3.5 Calculate %RSD as follows:

$$\frac{\text{Std(Result 1 - Result 2)}}{\text{Avg(Result 1 - Result 2)}} \times 100 = \text{\%RSD}$$

7.3.6 Calculate Sample Spike Recovery as follows:

$$\frac{\text{Spike Concentration - Sample Concentration}}{\text{Concentration added to Spike}} \times 100 = \text{\% Recovery}$$

8.0 Corrective Actions

8.1 Data Qualification

All quality control measures have associated acceptance windows. A failure of any of these measures may necessitate corrective action. The action may be to correct performance of a specific technique or to repair a hardware defect. Affected samples should be re-analyzed or reextracted first if necessary and then reanalyzed. Data that is unavoidably compromised and cannot be regenerated must be flagged. In such cases, the client is contacted promptly for more sample.

8.1.1 If DOCs, MDLs, LODs, or LOQs do not meet acceptability criteria, then repeat analysis or adjust criteria until acceptability is met.

8.1.2 If the LCS does not meet percent recovery acceptance criteria, then they should be reanalyzed along with all associated QC and samples. Samples may be reported with qualification (using the “J” qualifier).

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8.1.3 If samples analyzed with blanks (prep, initial, continuing) are not meeting acceptance criteria, then they may be reanalyzed or reported with qualification (using the "B" qualifier).

8.1.4 If the matrix spike sample or duplicate recovery is not within acceptance criteria, then samples may be reanalyzed or reported with qualification (using the "J" qualifier). If the sample result is greater than 5 times the spike amount, then the percent recovery need not be calculated.

8.1.5 If the matrix spike or duplicate do not meet percent recovery acceptance criteria, then the sample result may be reported with qualification (using the "J" qualifier) or reanalyzed.

8.1.6 If the DFTPP standard does not meet the tuning check acceptability criteria, then the DFTPP standard must be reanalyzed or the hardware must be re-tuned.

8.1.7 If the initial calibration curve is not within acceptance criteria, then recalibrate or qualify any positive sample values as estimated (using the "J" qualifier).

8.1.8 If the ICV does not meet acceptance criteria, then prepare a new curve and ICV or qualify any positive sample values as estimated (using the "J" qualifier).

8.1.9 If a CCV does not meet acceptance criteria, then all associated samples must be reanalyzed or qualified as estimated (using the "J" qualifier).

8.1.1 If surrogate recovery is not within the acceptable range, then the client must be notified and the sample is re-extracted whenever possible. Sample matrix effect is proven if the results obtained from the re-extracted sample show similar results. When this occurs, both results are reported with an explanation. A problem with the first extraction is indicated when the results of the re-extracted sample are in the acceptable range. In this case, only the compliant results are reported. If no additional sample can be obtained, then results are flagged in the report and a narrative is included qualifying the data.

8.1.11 If an analyte's continuing calibration response factor exceeds 20% recovery relative to the initial calibration, then a new initial calibration must be must be obtained or associated analyte values qualified as estimated.

8.1.12 If the internal standards do not meet acceptance criteria, then all associated samples must be reanalyzed or qualified as estimated (using the "J" qualifier).

8.2 Troubleshooting

In the event that quality control data are repeatedly beyond acceptance limits, the following parameters should be evaluated and modified.

- Quality and expiration date of standards
- Cleanliness of containers, equipment, and labware

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- Integrity of standards
- Instrument operation

9.0 Health and Safety

9.1 Methylene chloride and semi-volatile standards are extremely hazardous to everyone in the laboratory if handled improperly.Gloves and safety glasses must be worn when handling samples, reagents, and standards. It is necessary to wear a lab coat to protect clothing. All solvent usage must occur in properly working fume hoods. Use precautions to prevent spills; if any spills do occur, clean up all spills immediately and notify supervisor.

9.2 Analysts should familiarize themselves with any chemical substances which are named along with their corresponding Material Safety Data Sheet (MSDS).

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure all acceptance criteria has been set and SOPs are updated as required. The Laboratory Manager must also ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedure outlined in this SOP, recording all data as described in Section 7.1, and reviewing all QC results for compliance with acceptance criteria.

10.2 Prior to any staff member working unsupervised, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification is based upon a review of the analyst's demonstrated abilities and DOC performance.

11.0 Waste Management and Pollution Prevention

Unused portions of samples are disposed of only after completion of sample analysis, submission of the final report and contracted sample retention times have been achieved. All conditions of disposal and all records and correspondence concerning the final disposition of the physical sample shall be recorded and retained. Records shall indicate the date of disposal, the nature of disposal and the name of the individual who performed the task.

Unused non-hazardous sample may be disposed of as wastewater. Solvents and extracted sample not evaporated in a hood are collected and disposed of as hazardous waste in designated solvent water waste containers. Minimize solvent use and prepare standards in amounts that will be used before expiration. Commercial extracts are disposed of after approximately 2 months. CLP extracts are disposed of according to the contract.

11.1 Aqueous Samples

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After testing, aqueous samples may be disposed of through ESI's aqueous waste UV treatment system located on the first floor. This includes all unused samples, diluted samples and dilution waters that have a pH >6 and <8.

Any samples found to be toxic may require further testing or treatment in order to determine proper disposal technique to assure compliance with all local, state and federal regulations.

11.2 Solid Materials

Waste storage, transfer and disposal will be in compliance with NH DES and US EPA waste disposal regulations. Non-hazardous solid materials, sediments and soils will be disposed of through an appropriate waste disposal site. All non-hazardous solid waste will be drained of as much free liquid as possible. The remaining sample is removed from its original container and placed with other non-hazardous waste samples in a 55 gallon metal drum. Once full, these containers will then be transferred to an approved waste disposal facility. This transfer is recorded on a waste manifest and they are retained for a minimum of 3 years.

11.3 Hazardous Waste Samples

Hazardous waste samples must be handled by properly trained employees. Only the ESI hazardous waste coordinator(s) may train ESI employees on handling hazardous waste. There are 3 main hazardous waste streams at ESI, “dilute metals acidic”, “solvent water” and “Lachat wastes”. When a satellite container needs to be emptied, it is properly transported to the waste shed and added to the appropriately labeled container for that waste stream. The volume of waste added and the date are recorded in the Hazardous Waste Logbook. Shipping manifests reflect the volume and date the wastes are picked up for transport.

Appendix 1. Target Analytes

Phenols	Base/ Neutrals	Add- Ons	Polycycic Aromatic Hydrocarbons (PAH)
Phenol	1, 2,4-Trichlorobenzene	2,4,5-Trichlorophenol	naphthalene
Pentchlorophenol	1,2-Dichlorobenzene	2-Methylnaphthalene	2-methylnapthalene
4-Nitrophenol	1,3-Dichlorobenzene	2-Methylphenol	acenaphthylene
4-Chloro-3-methylphenol	1,4-Dichlorobenzene	2-Nitroaniline	acenaphthene
4,6-Dinitro-2-methylphenol	2,4-Dinitrotoluene	3-Nitroaniline	fluorene
2-Nitrophenol	2,6-Dinitrotoluene	4-Chloroaniline (p-Chloroaniline)	phenanthrene
2-Chlorophenol	2-Chloronaphthalene	4-Methylphenol	anthracene
2,4-Dintrophenol	4-Bromodiphenyl ether	4-Nitroaniline	fluoranthene
2,4-Dimethylphenol	4-Chlorophenyl-phenyl ether	Aniline	pyrene
2,4-Dichlorophenol	Azobenzene	Benzoic acid	benzo(a)anthracene
2,4,6-Trichlorophenol	Bis(2-chloro-1-methylethyl) ether	Benzyl alcohol	chrysene
	bis(2-Chloroethoxy)methane	Dibenzofuran	benzo(b)fluoroanthene
Benzidines	bis(2-Chloroethyl)ether		benzo(k)fluoroanthene
Benzidene	Bis(2-Ethylhexyl)phthalate		benzo(a)pyrene
3,3-Dichlorobenzidine	Butylbenzyl phthalate		indeno(1,2,3-cd)pyrene

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Carbazole
Diethyl phthalate
Dimethyl phthalate
Di-n-butyl phthalate
Di-n-octyl phthalate
Hexachlorobenzene
Hexachlorobutadiene
Hexachlorocyclopentadiene
Hexachloroethane
Isophorone
Nitrobenzene
N-Nitrosodimethylamine
N-Nitrosodi-n-propylamine
N-Nitrosodiphenylamine
Pyridine
dibenzo(a,h)anthracene
benzo(g,h,i)perylene

Appendix 2. DFTPP Tuning Criteria[1]

GC/MS Performance Standard - Decafluorotriphenylphospine (DFTPP)

Ion	Abundance m/z Criteria
51	30-60% of mass 198
68	Less than 2% of mass 69
69	(reference only)
70	Less than 2% of mass 69
127	40-60% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
275	5-9% of mass 198
365	Greater than 1% of mass 198
441	Present but less than mass 443
442	Greater than 40% of mass 198
443	17-23% of mass 442

[1] Table adapted from US EPA Statement of Work: 2/88, E-30/SV

Note: Mass 125 and 275 abundance ranges relative to 198 may be extended for enhanced target compound sensibility when selected ion mode analysis is performed.

Appendix 3. Semi-Volatile Run Conditions

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Title: Analysis of Semi-volatile Compounds by GC/MS - Region II Testing

INSTRUMENT_____AGILENT 6890GC/5973MSD

METHOD QASOP#

AC
3. 625/8270 ABN 5200

TEMPERATURE PROGRAMS

1. DFTPP: START AT 100°C, RAMP 20/MIN, FINAL 5MIN AT 300°C. RUN TIME=15MIN MS SCANS 35-500 AMU, A/D RATE =2
A/S PARAMETES: 1UL VOLUME INJECTION
#SAMPLE WASHES (1)____ #SAMPLE PUMPS (6)_____.
SOLVENT A WASHES (3)____ SOLVENT B WASHES (3)____.
SAMPLE VISCOSITY WAIT (0) INJECTION MODE IS FAST
SOLVENT A = 1:1 TOLUENE/$MECL_2$ SOLVENT B = $MECL_2$

2. 625/8270°C ABN: FOR 2MIN, RAMP 10/MIN, FINAL 8MIN AT 300°C. RUN TIME = 35MIN MS SCANS 35-500 AMU, A/D RATE =2
A/S PARAMETES: 1UL VOLUME INJECTION
#SAMPLE WASHES (1)____ #SAMPLE PUMPS (6)_____.
SOLVENT A WASHES (3)____ SOLVENT B WASHES (3)____.
SAMPLE VISCOSITY WAIT (0) INJECTION MODE IS FAST
SOLVENT A = 1:1 TOLUENE/$MECL_2$ SOLVENT B = $MECL_2$

3. 625/8270 BN: 50°C FOR 2MIN, RAMP 10/MIN FINAL 15MINAT 300°C. RUN TIME = 42MIN MS SCANS 35-500 AMU, A/D RATE =2
A/S PARAMETES: 1UL VOLUME INJECTION
#SAMPLE WASHES (1)____ #SAMPLE PUMPS (6)_____.
SOLVENT A WASHES (3)____ SOLVENT B WASHES (3)____.
SAMPLE VISCOSITY WAIT (0) INJECTION MODE IS FAST
SOLVENT A = 1:1 TOLUENE/$MECL_2$ SOLVENT B = $MECL_2$

INJECTOR TEMP = 275°C, INTERFACE (DET B) = 300°C
SPLITLESS VALVE TIME (.85MIN)_____,OVEN EQIULIB = 2MIN
COLUMN HEAD PRESSURE (7PSI=50KPA)_____.
TOTAL FLOW (1000ML/MIN)_____, SEPTUM PURGE (1ML/MIN)____.
GOAL IS 1ML/MIN ON COLUMN WITH 99% OF TOTAL FLOW EXITING SPLIT VENT

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Appendix 4. Semi-Volatile Calibration Requirements

[2]Compounds not requiring 20 standard in five point calibration:

Compound # Quant	HP Ion	Compound
23	122	Benzoic acid
37	196	2,4,5-Trichlorophenol
39	65	2-Nitroaniline
42	138	3-Nitroaniline
44	184	2,4-Dinitrophenol
45	109	4-Nitrophenol
52	138	4-Nitroaniline
54	198	4,6-Dinitro-2-Methylphenol
59	266	Pentachlorophenol

[2] Table adapted from US EPA Statement of work: 2/88 E-30/SV, Section 2.1.1

[3] CCC and SPCC Compounds:

Compound # Quant	HP Ion	Compound	Type
4	94	Phenol	CCC
9	146	1,4-Dichlorobenzene	CCC
15	70	N-Nitroso-di-n-propylamine	SPCC
21	139	2-Nitrophenol	CCC
25	162	2,4-Dichlorophenol	CCC
29	225	Hexachlorobutadiene	CCC
30	107	4-Chloro-3-methylphenol	CCC
35	237	Hexachlorobutadiene	SPCC
36	196	2,4,6-Trichlorophenol	CCC
43	153	Acenaphthene	CCC
44	184	2,4-Dinitrophenol	SPCC
45	109	4-Nitrophenol	SPCC
55	169	N-Nitrosodiphenylamine	CCC
59	266	Pentachlorophenol	CCC
63	202	Fluoranthene	CCC
74	149	Di-n-octylphthalate	CCC
77	252	Benzo(A)pyrene	CCC

- %RSD for CCCs ≤ to 30.0% for five point.
- %Difference for CCCs of continuing calibration ≤ to 25.0%
- Minimum RRF for SPCCs is 0.050.

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[3] Table adapted from US EPA Statement of Work: 2/88, E-31-35/SV, Sections 2.3.1, 2.4.1, 2.6.1, 2.6.2.1, Table 2.3

Appendix 5. Internal Standards

Compound # Quant.	HP Extrel	Ion	Compound	% Recovery
1	1S	152	1,4-Dichlorobenzene-D4	50-200
17	2S	136	Naphthalene-D8	50-200
32	3S	164	Acenaphthene-D10	50-200
53	4S	188	Phenanthrene-D10	50-200
65	5S	240	Chrysene-D12	50-200
73	6S	264	Perylene-D12	50-200

Appendix 6. Method 625 LCS Acceptance Critera

[4] **Table 6—QC Acceptance Criteria—Method 625**

Parameter	Test conclusion (µg/L)	Limits for s (µg/L)	Range for (µg/L)	Range for P, P (Percent)s
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27-133
Benzo(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(ghi)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
P-BHC	100	31.5	41.5-130.6	24-149
o-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl)ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl)ether a	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118

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1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4,-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dhlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26-155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
PCB-1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Dichlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation for four recovery measurements, in µg/L (Section 8.2.4).
P = Average recovery for four recovery measurements, in µ/L (Section 8.2.4). P, P = Percent recovery measured (Section 8.3.2, Section 8.4.2).

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D = Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limts to concentrations below those used to develop Table 7.

[a] The proper chemical name is 2,2'oxybis(1-chloropropane).

[4] Table adapted from US EPA Method 625.

Appendix 7. Calibration Preparation

In each part, use the described stock standards to make the appropriate working standards. (FV = Final Volume)

Part A: SVOC Calibration Curve and Continuing Calibration Recipe

Stock Standards

SVOC Surrogates Mix	1000/2000 µg/mL
Phenols Mix	2000 µg/mL
Benzidines Mix	2000 µg/mL
Base/ Neutrals Mix	2000 µg/mL
8270 Add- Ons Mix	2000 µg/mL
PAH Analyte Mix	2000 µg/mL
Internal Standard Mix	4000 µg/mL

Working Standards

Calibration Mix Solution prepared by adding 200 µL of each mix listed above brought to FV 2.0 mL with MeCl2 = 200 µg/mL.
400 µL Cal Mix + 4 µL Internal Standard = 200 µg/mL
320 µL Cal Mix to FV 400 µL + 4 µL Internal Standard = 160 µg/mL
240 µL Cal Mix to FV 400 µL + 4 µL Internal Standard =120 µg/mL
400 µL Cal Mix to FV 1000 µL + 10 µL Internal Standard = 80 µg/mL
200 µL Cal Mix to FV 1000 µL + 10 µL Internal Standard = 40 µg/mL
100 µL Cal Mix to FV 1000 µL + 10 µL Internal Standard = 20 µg/mL
CCV 80 µg/mL- 4000 µL Cal Mix to FV 1000 µL +10 µL Internal Standard
CCB 1000 µL $MeCl_2$ + 10 µL Internal Standard

Part B: SVOC Initial Calibration Verification

Stock Standards

Restek 8270 Calibration Mixes 1-5	2000 µg/mL
Internal Standard Mix	4000 µg/mL

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Working Standards

ICV 120 µg/mL 60 µL of each 8270 Cal Mix 1-5 to FV 1 mL $MeCl_2$ +10 µL Internal Standard
ICB 1 mL $MeCl_2$ +10 µL Internal Standard

Part C: PAH Calibration Curve and Continuing Calibration Recipe

Stock Standards

2-fluorobiphenyl (SS)	200 µg/mL
o-terphenyl (SS)	200 µg/mL
PAH mix	100 µg/mL
Internal Standard Mix	40 µg/mL

Working Standards

Calibration Mix Solution prepared by adding 300 µL of the PAH Mix and 150 µL of each of the SS standards to a FV of 3.0 mL in $MeCl_2$ = 10 µg/mL

10 µg/mL = 500 µL Cal Mix + 12.5 µL Internal Standard
8 µg/mL = 800 µL Cal Mix to FV 1.0 mL $MeCl_2$ + 25 µL Internal Standard
6 µg/mL = 600 µL Cal Mix to FV 1.0 mL $MeCl_2$ + 25 µL Internal Standard
4 µg/mL = 400 µL Cal Mix to FV 1.0 mL $MeCl_2$ + 25 µL Internal Standard
2 µg/mL = 200 µL Cal Mix to FV 1.0 mL $MeCl_2$ + 25 µL Internal Standard
1 µg/mL = 100 µL Cal Mix to FV 1.0 mL $MeCl_2$ + 25 µL Internal Standard
0.2 µg/mL = 20 µL Cal Mix to FV 1.0 mL $MeCl_2$ + 25 µL Internal Standard
CCV 2ug/mL= 200 µL Cal Mix to FV 1.0 mL $MeCl_2$ + 25 µL Internal Standard
CCB 1.0 mL $MeCl_2$ + 25 µL Internal Standard

Part D: PAH Initial Calibration Verification

Stock Standards

PAH mix	2000 µg/mL

Working Standards

ICV 2 µg/mL 1 µL PAH Mix to FV 1.0 mL $MeCl_2$ + 25 µL Internal Standard
ICB 1 mL $MeCl_2$ +10 µL Internal Standard

Part E: Pentachlorophenol (PCP) Calibration Curve and Continuing Calibration Recipe

Stock Standards

2,4,6-tribromophenol (SS)	100 µg/mL
Pentachlorophenol	100 µg/mL

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Internal Standard Mix 40 µg/mL

Working Standards

Calibration Mix Solution prepared by adding 500 µL of the PCP Surrogate and 500 µL of the PCP standard to a FV 2.0 mL with $MeCl_2$ = 25 µg/mL

25 µg/mL = 500 µL Cal Mix + 12.5 µL Internal Standard
20 µg/mL = 400 µL Cal Mix to FV 0.5 mL $MeCl_2$ + 12.5 µL Internal Standard
15 µg/mL = 300 µL Cal Mix to FV 0.5 mL $MeCl_2$ + 12.5 µL Internal Standard
10 µg/mL = 200 µL Cal Mix to FV 0.5 mL $MeCl_2$ + 12.5 µL Internal Standard
5 µg/mL= 100 µL Cal Mix to FV 0.5 mL $MeCl_2$ + 12.5 µL Internal Standard
2.5 µg/mL= 80 µL Cal Mix to FV 0.5 mL $MeCl_2$ + 12.5 µL Internal Standard
CCV 10 µg/mL= 200 µL Cal Mix to FV 0.5 mL $MeCl_2$ + 12.5 µL Internal Standard
CCB 0.5mL $MeCl_2$ + 12.5 µL Internal Standard

Part F: PCP Initial Calibration Verification

Stock Standards

2,4,6-tribromophenol (SS)	100 µg/mL
Acid SpikeMix (containing PCP)	200 µg/mL
Internal Standard Mix	40 µg/mL

Working Standards

ICV 15 µg/mL 75 µL Acid Spike + 150 µL SS to FV 1.0 mL $MeCl_2$ + 25 µL Internal Standard
ICB 1 mL $MeCl_2$ + 25 µL Internal Standard

Appendix 8. Reporting Limits

Analyte	Tissue (ng/g)	Sediment (ng/g)	Water (µg/L)
Acenaphthene	**50**	**100**	**N/R**
Acenapthylene	**50**	**100**	**N/R**
Anthracene	**50**	**100**	**N/R**
Fluorene	**50**	**100**	**N/R**
Naphthalene	**50**	**100**	**N/R**
Phenanthrene	**50**	**100**	**N/R**
Benzo(a)anthracene	**50**	**100**	**N/R**
Benzo(a)pyrene	**50**	**100**	**N/R**
Benzo(g,h,i)perylene	**50**	**100**	**N/R**
Benzo(b)fluoranthene	**50**	**100**	**N/R**

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Benzo(k)fluoranthene	**50**	**100**	**N/R**
Chrysene	**50**	**100**	**N/R**
Dibenzo(a,h)anthracene	**50**	**100**	**N/R**
Fluoranthene	**50**	**100**	**N/R**
Indeno(1,2,3-cd)pyrene	**50**	**100**	**N/R**
Pyrene	**50**	**100**	**N/R**
1,4-dichlrorbenzene	**3900**	**100**	**N/R**

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TM-001

January 26, 2018

Analysis of Trace Metals and Mercury in Salt Water and Tissue – E&E Region 2

This document describes the actions necessary to adapt ESI SOP's QA-1369 R10 "Determination of Trace Metals by ICP-MS", QA-1365 R7 "Mercury by Cold Vapour AF", and QA-1371 R5 "Mercury by AF Sed or Tissue " to meet the requirement of EPA Region 2.

Salt Water

A QC batch for salt water samples will consist of 1 to 20 samples. Each batch will contain a Procedural Blank (PB), Laboratory Control Sample (LCS), Matrix Spike (MS), Matrix Spike Duplicate (MSD), Matrix Spike Triplicate (MST), and Standard Reference Material (SRM).

The PB, and LCS for Trace Metals analysis will be made of 2% NaCl. For Mercury analysis they will be made in MilliQ water. The SRM for Trace Metals analysis will be QC3163. The SRM for Mercury Analysis will be made in 2% NaCL spiked with a certified secondary sourced Mercury Standard at a concentration of 2500 ng/L. When analyzing elutriates, sample QC will be performed on a site-water sample.

The Acceptance Criteria for Procedural Blanks is no analyte shall be detected at > RL, LCS samples is 70 – 130 % (recovery), Matrix Spikes is 70 – 130 % (recovery) and 30% (RSD), and SRM is 70 – 130%. All other QC acceptance criteria will be as defined in the relevant ESI SOP.

Results shall be reported in ug/L to 3 significant figures.

The Reporting Limit (RL) and Spike Levels are as follows:

Analyte	RL (ug/L)	Spike (ug/L)
Arsenic	NR	NR
Cadmium	0.2	50.0
Chromium	0.3	1400
Copper	0.4	5.0
Lead	0.2	250
Mercury	0.01	3.0
Nickel	0.8	100
Silver	0.2	4.0
Zinc	2	120

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Tissues

A QC batch for tissue samples will consist of 1 to 20 samples. Each batch will contain a Procedural Blank (PB), Laboratory Control Sample (LCS), Matrix Spike (MS), Matrix Spike Duplicate (MSD), and Matrix Spike Triplicate (MST). The analysis of a Standard Reference Material (SRM) is not required.

The PB and LCS for Trace Metals and Mercury analysis will be made in a digestion vessel containing 5 mL of MilliQ. The MS, MSD, and MST will be performed on pre-test tissues.

The acceptance Criteria for Procedural Blanks is no analyte shall be detected at > RL, LCS samples is 70 – 130 % (recovery), Matrix Spikes is 70 – 130 % (recovery) and 30% (RSD). All other QC acceptance criteria will be as defined in the relevant ESI SOP.

Results shall be reported as ug/g and ug/g dry weight to 3 significant figures.

The Reporting Limit (RL) and Spike Levels are as follows:

Analyte	RL (ug/g wet weight)	Spike (ug/g wet weight)
Arsenic	0.02	16.0
Cadmium	0.01	0.50
Chromium	0.02	16.0
Copper	0.01	15.0
Lead	0.01	2.0
Mercury	0.01	0.30
Nickel	0.8	6.5
Silver	0.2	2.0
Zinc	2	2000

The Spike Level for tissues samples is a nominal value that is determined by sample weight.

Session QC

An instrument session consists of 1 or more QC batches. Immediately following the calibration an ICV/ICB check shall be performed. The acceptance criteria for the ICV is 90 – 110%. Acceptance criteria for the ICB is no analyte present at > RL. A CCV/CCB shall be performed at the middle and end of end of each batch or 1 per every 10 analyses, whichever is greater. Acceptance criteria for the CCB is no analyte present at > RL. Acceptance criteria for the CCV is 90 – 110%.

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ORIGINAL

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Enthalpy Analytical – Wilmington, NC (Ultratrace)
Standard Operating Procedure

EPA Methods 23, 0023A, 8290A, 1613B & TO-9A for the Analysis of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans

Enthalpy Ultratrace SOP #	EU045	Revision #	1
Author	B. Vining	Date Authored	01/03/2017
Revised by	B. Vining	Date Revised	7/12/17

Pages: 41

Technical Director/designee Approval: Approval Date: 31 JUL 17

Quality Manager/designee Review: Review Date: 08 August 17

Effective Date: 08 August 17

1.0 Scope and Application:

This method is applicable to the determination of polychlorinated dibenzo-p-dioxins (PCDD's) and polychlorinated dibenzofurans (PCDF's), incorporating special requirements of EPA Methods 23, 0023A, 1613, 8290, and TO-9A in all applicable matrices.

2.0 Summary of Method:

This procedure uses matrix-specific extraction, analyte-specific cleanup, and high resolution capillary column gas chromatography/high-resolution mass spectrometry (HRGC/HRMS) analytical techniques.

The PCDD's and PCDF's are extracted from the sample, separated by high-resolution gas chromatography (HRGC), and measured by high-resolution mass spectrometry (HRMS). The underlying technology is isotope-dilution high-resolution gas chromatography coupled with high-resolution mass spectrometry.

3.0 Definitions:

3.1 Initial Calibration (ICAL) – Standardization of the analytical instrumentation using varying concentrations of reference materials to establish the instruments calibration range.

3.2 Laboratory Control Sample (LCS) and Ongoing Precision Recovery (OPR) – Quality Control samples prepared and analyzed with the samples to ensure the reliability and the accuracy (the bias and the precision). The samples are sometimes required by client for bias and the precision determinations instead of the BCS Spike.

3.3 Batch Control Spike (BCS) – A QC sample specifically introduced for comprehensive stable isotope-dilution mass spectrometric methodologies \to ensure the reliability and the accuracy (the bias and the precision) of the determinations.

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3.3 Batch Control Spike (BCS) – A QC sample specifically introduced for comprehensive stable isotope-dilution mass spectrometric methodologies \to ensure the reliability and the accuracy (the bias and the precision) of the determinations.

3.4 Method Blank (MB) – A QC sample prepared and analyzed with each batch of samples to assess the contamination present on the sample processing. The method blank must be subjected to the same extraction and cleanup procedures as the field samples.

3.5 Performance Evaluation (PE) or Proficiency Tests – Samples purchased from an approved supplier as required by the environmental testing accreditation standard to determine the performance capabilities of individual laboratories.

3.6 Alternate Standards (AS) – Standards added to the sample extracts before the fractionation step in order to help monitor selective losses of specific isomers/congeners to ensure that no distortion of the congener profiles occurs during the fractionation step. AS response factors are determined relative to the JS.

3.7 Cleanup Standards (CS) – Standards added to the sample extracts prior to the fractionation step. The role of the CS is to offer specific feedback to the laboratory regarding the fractionation step's efficiency, independent of the extraction step. CS recovery values are computed using a response factor relative to the JS.

3.8 Extraction Standards (ES) – Standards introduced (for all sample types) into the sample matrix prior to the extraction step (variously named internal standards, surrogates, and/or labeled internal standards in promulgated methods). Ideally, the ES should be introduced inside the sample before any step capable of modifying the sample's composition. The role of the ES is to monitor the extraction-fractionation efficiencies as well as to qualitatively and quantitatively characterize the target analytes. ES recovery values are determined relative to the Injection Standards (i.e. their response factors are determined relative to the corresponding JS). In addition, ES are used to establish the relative response factors for other labeled standards, which are used to monitor (independent of the extraction/fractionation) particular steps such as the sampling and storage conditions of stack flue gases and ambient air samples (SS), the field concentration of the air sampling component solvent rinses (FS), and the laboratory's ability to transfer the concentrated air sampling equipment rinses inside the extraction vessel (TS).

3.9 Surrogate Standard or Sampling Standard (SS) – Standards (variously named field standards or field surrogates in promulgated methods) introduced inside the sampling media before the sampling event. SS recovery values are computed in such a manner as to correct for losses occurring during the extraction-fractionation-concentration steps so that feedback on performance is specific to the sampling event.

3.10 Transfer Standard (TS) – Standard used to distinguish a loss of FS during the concentration step, versus a non-quantitative transfer step by the laboratory during the transfer of the concentrated rinses inside the extraction vessel. TS response factors are determined relative to the ES in order to correct for losses that occur during the extraction-fractionation steps

3.11 Field Concentration Standard (FS) – Although primarily introduced for PCDD/F flue gas air sampling trains, the concept of the FS can be expanded to other target analytes wherever appropriate and feasible, or other sample types, depending upon the availability of labeled standards. FS are fortified along with adequate "keepers" inside

containers where solvent rinses of the stack sampling equipment are collected, following a sampling event. The role of the FS is to monitor losses that might occur during field concentration (or wherever applicable, the laboratory concentration) of the rinses prior to shipment to the laboratory. In such circumstances, the response factor of the FS is determined relative to the ES. Concentrating rinses in the field reduces the risks, liabilities, complications, and costs normally associated with the shipment of dangerous goods. These may also be used to assess the extraction of compounds from the particulate matter found on modified method 5 air sampling trains.

3.12 A_x – Native Standards that are spiked into QC samples only

3.13 Injection Standards – JS – Standards (variously named internal standards or recovery standards in promulgated methods) used during the preparation of the final extract just before the injection of an aliquot of the final extract onto the GC column. JS are used to establish the relative response factors for a number of the labeled standards used throughout the procedure (e.g. ES, CS, AS) and to determine their recovery values up until the point of the JS' addition. An extract previously analyzed may require an additional cleanup step prior to a reanalysis. Since the JS is already present before this additional cleanup step, and unless a different JS is introduced before the analysis, the recovery values measured on the "cleaned" extract contain no valuable information regarding the additional cleanup step. Furthermore, for practical reasons, the JS addition is sometimes conducted prior to the final transfer and/or for a concentration step. This protocol recognizes that these practices are acceptable as long as the performance-based criteria are met. In other words, should significant evaporative losses occur the laboratory will experience difficulties meeting the performance-based criteria

3.14 Performance Based Measurement System (PBMS) – A quality control system using performance based criteria to quantify fitness for purpose and continuous improvement.

4.0 Safety:

4.1 Personal Protection Equipment (PPE) such as disposable plastic gloves, apron or lab coat, and safety glasses at a minimum must be warn for sample handling.

4.2 All samples should be considered hazardous. All personnel working in the laboratory are required to follow the Enthalpy Analytical, LLC Safety and Health Plan.

4.3 The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high boiling alcohols.

4.4 Please refer to Appendix A for more detailed information about the toxicity related to dioxin/furans.

5.0 Equipment and Supplies:

5.1 High-resolution gas chromatograph/high-resolution mass spectrometer (HRGC/HRMS) – Waters Autospec NT or equivalent with Agilent 6890 or equivalent GC system.

5.2 Autosampler – Agilent 7673 or equivalent

5.3 GC Injector Port – 1-µL injections can be used on the 60-m DB-5 column. The use of a moving needle injection port is also acceptable.

5.4 Autosampler glass vials

5.5 GC/MS interface – Graphite/Vespel™, or equivalent, ferrules are recommended.

5.6 Data System – MassLynx™

5.7 GC Columns:

- 60-m DB-5MS (J&W Scientific) fused-silica capillary column, or equivalent
- 30-m DB-225 (J&W Scientific) fused-silica capillary column , or equivalent

6.0 Reagents and Standards:

6.1 Helium for GC/MS carrier gas

NOTE: Hydrogen is not to be used. Using hydrogen as a carrier gas during the GC analysis of PCDD/Fs can result in 20% destruction of PCDD/Fs (i.e., reduction reactions) inside the ion source of the mass spectrometer.

6.2 Standard Solutions:

6.2.1 Purchased as solutions or mixtures with certification of analysis as to their purity, concentration, and authenticity.

6.2.2 When not being used, standards are stored in the dark at room temperature in amber screw-capped vials with fluoropolymer lined caps. Mark the vial at the level of the solution so that solvent loss by evaporation can be detected.

6.2.3 If solvent loss becomes evident replace the solution. The solution must fill at least half the volume of the vial, so transferring solutions to a smaller vial will be necessary as the solution is consumed. Alternatively, aliquoting the solution into a larger number of "one-use" vials may be prudent.

6.3 Stock Solutions:

6.3.1 Primary stock solutions of native PCDD/Fs are purchased from Cambridge Isotope Laboratories (CIL, Woburn, MA - EDF-5008 or equivalent).

6.3.2 Second source solutions are purchased from Wellington Laboratories (Guelph, ON – NK-ST-B4 or equivalent).

6.3.3 Solutions of native compounds are only to be used as purchased mixtures or dilutions thereof. It is strictly forbidden to create native analyte solutions from individual native analyte solutions.

6.3.4 Isotopically labeled standards may be purchased individually or in mixes (e.g., Cambridge Isotope Laboratories EDF-5041-20).

6.3.5 Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Reference standards that can be used to determine the accuracy of calibration standards are available from CIL and may be available from other vendors

6.4 Initial Calibration Solutions – See Table 1 for composition of the individual solutions.

6.5 Sample Fortification Solution - Table 2 gives the amounts of each congener to be spiked into samples.

6.5.1 The concentration of the solutions used to spike these amounts must be such that the spike volume needed to spike these amounts is not less than 20 µL and no more than 500 µL.

6.5.2 The solutions must be prepared in nonane or toluene.

6.6 GC Retention Time Window Defining Solution and Isomer Specificity Test Standard:

6.6.1 Used to define the beginning and ending retention times for the dioxin and furan isomers and to demonstrate isomer specificity of the GC columns employed for determination of 2,3,7,8-TCDD and 2,3,7,8-TCDF.

6.6.2 The standard must contain the compounds listed in Table 3 (CIL EDF-4006, or equivalent), at a minimum.

6.6.3 It is not necessary to monitor the window-defining compounds if only 2,3,7,8-TCDD and 2,3,7,8-TCDF are to be determined. In this case, an isomer-specificity test standard containing the most closely eluted isomers listed in Table 3 (CIL EDF-4033, or equivalent) may be used.

6.7 A QC Check Sample should be obtained from a source independent of the calibration standards. Ideally, this check sample would be a certified reference material containing the CDDs/CDFs in known concentrations in a sample matrix similar to the matrix under test.

7.0 Sample Preservation, Storage, and Handling:

7.1 Samples are stored per method requirements [cold (at 4ºC ± 2°C) or ambient conditions] and placed in a secure storage unit prior to and after analysis. Storage location is recorded on an internal chain-of custody.

7.2 Samples are retained for one month after a report is sent unless we are specifically requested by the clients to either return the samples or store them for a longer period of time.

7.3 Method 23 and 0023A samples extract archives (after split but before cleanup) are stored at room temperature in labeled glass vials with a screw top.

7.3.1 NOTE: This portion of the sample is retained for backup of the air sample extract in case any problems occur during the rest of the preparation/analysis of the samples. One doesn't get a second chance with air samples, unlike a solid sample).

7.3.2 Disposal of this extract archive is done at least 30 days after analysis is complete and final report delivered to the client.

7.4 The final extracts are labeled and stored in glass Autosampler vials and placed in a plastic storage box after analysis and stored at room temperature in the HRGC/HRMS area for at least 6 months.

7.5 Please refer to Enthalpy Ultratrace's corresponding sample preparation and extraction SOP EU046 for more information on sample preservation, storage and handling.

8.0 Calibration:

8.1 Initial Calibration:

8.1.1 Initial calibration is required before any samples are analyzed for PCDDs and PCDFs and must meet the acceptance criteria. Initial calibration may also be required if any routine calibration does not meet the required criteria listed in this SOP.

8.1.2 Initial calibration serves different purposes in this SOP than in non-isotope-dilution assays and differs as well from the EPA published methods. Initial calibration establishes; 1) the linearity of the instrument over the calibration range and 2) a set of reference RRFs against which continuing calibrations are evaluated.

8.1.3 At a minimum, all calibration solutions CS1 to CS5 listed in Table 1 must be used for the initial calibration.

8.1.4 Tune the instrument with PFK. The recommended mass spectrometer tuning conditions are based on the groups of monitored ions shown in Table 4. By using a PFK molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). By using peak matching conditions and the aforementioned PFK reference peak, verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors (Table 4).

8.1.5 Inject 1µL of the GC column performance check solution and acquire SIM mass spectral data as described later in this SOP. The total cycle time must be < 1 second. The laboratory must not perform any further analysis until it is demonstrated and documented that the criteria listed in Section 10.10 were met.

8.1.6 By using the same GC and MS conditions that produced acceptable results with the column performance check solution, analyze a 1-µL portion of each of the calibration solutions once with the following mass spectrometer operating parameters.

8.1.6.1 The ratio of integrated ion current for the ions appearing in Table 4 must be within the indicated control limits for all unlabeled calibration standards in Table 4.

8.1.6.2 The ratio of integrated ion current for the ions belonging to the carbon-labeled internal and recovery standards (Table 4) must be within the control limits stipulated in Table 4.

8.1.6.3 For each selected ion current profile (SICP) and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. Measurement of S/N is required for any GC peak that has an apparent S/N of less than 5:1.

8.1.7 The relative response factors to be used for the determination of the concentration of total isomers in a homologous series are calculated as follows:

8.1.7.1 For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 7; TCDD, PeCDD, HpCDD, and TCDF), the RRF used will be the same as the RRF determined in section 11.2.

8.1.7.2 For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 7), the RRF used for those homologous series will be the mean of the RRFs calculated for all individual 2,3,7,8-substituted in that homologous series.

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution pattern are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

8.1.8 The following initial calibration criteria must be met before sample analyses are performed.

8.1.8.1 The percent relative standard deviations for the mean response factors from the 17 unlabeled standards must not exceed ± 10 percent, and those for the labeled reference compounds must not exceed ± 20 percent.

8.1.8.2 The S/N for the GC signals present in every SICP (including the ones for the labeled standards) must be >10.

8.1.8.3 The ion abundance ratios (Table 4) must be within the specified control limits.

8.1.8.4 The RRF for each unlabeled target analyte must be within 20% of the historical mean RRF for all initial calibrations performed for this analysis on the instrument in question.

8.1.8.5 The RRF for each labeled standard must be within 30% of the historical mean RRF for all initial calibrations performed for this analysis on the instrument in question.

8.1.9 If the criteria for acceptable calibration listed in section 8.1.8 are met, the analyte-specific RRF can then be considered independent of the analyte quantity. The mean RRFs will be used for all calculations and accepted until new RRF values are calculated from a new set of injections of the calibration solutions.

8.1.10 Initial calibration verification (ICV) – A CS_3 prepared with unlabeled target analytes from a source different than that used for the ICAL must be analyzed and evaluated as part of the initial calibration process. The ICV is evaluated using the routine calibration criteria (Section 8.2.6). An ICV that fails to meet these criteria requires investigation and corrective action as necessary. The initial calibration cannot be accepted without a satisfactory ICV.

8.2 Continuing (Routine) Calibration Verification (Concal):

8.2.1 Routine calibration checks must be performed at the beginning of a 12-hour period, after successful mass resolution and GC resolution performance checks. A routine calibration is also required at the end of a 12-hour period. Inject 1µL of the concentration calibration solution CS_3 standard (Table 1). Using the same HRGC/HRMS conditions as used in section 8.1, determine and document an acceptable calibration as provided in Section 8.2.6.

8.2.2 The m/z abundance ratios for all PCDD/Fs shall be within the limits in Table 4; otherwise, the mass spectrometer shall be adjusted until the m/z abundance ratios fall within the limits specified, and the verification test shall be repeated. If the adjustment alters the resolution of the mass spectrometer, the resolution shall be verified prior to repeat of the verification test.

8.2.3 The peaks representing each PCDD/F and labeled compounds in the CS_3 standard must be present with S/N of at least 10; otherwise, the mass spectrometer shall be adjusted and the verification test repeated.

8.2.4 Compute the response factors of each unlabeled and labeled PCDD/F. Compare the RRF for each compound with the criteria given in Section 8.2.6

8.2.5 If all compounds meet the acceptance criteria, calibration has been verified and analysis of standards and sample extracts may proceed. If, however, any compound fails its respective limit, see Section 12 for appropriate action.

NOTE: It is acceptable to achieve both the GC resolution check and continuing calibration using a single injection, as long as the standard solution used contains NIST traceable concentrations for all calibrated parameters and the relevant close eluting isomers. The GC resolution check may also be combined with the batch control spike.

8.2.6 The following routine calibration criteria must be met before further analysis is performed.

8.2.6.1 The measured RRFs for the unlabeled standards obtained during the routine calibration runs must be within ± 20 percent of the mean values established during the initial calibration.

8.2.6.2 The measured RRFs for the labeled standards obtained during the routine calibration runs must be within ± 30 percent of the mean values established during the initial calibration.

8.2.6.3 The ion abundance ratios (Table 4) must be within the allowed control limits.

8.2.6.4 The relative percent difference (RPD) of the two routine calibrations performed before and after each 12-hour shift for each compound in the standard must meet certain criteria. For native (unlabeled) PCDD/Fs, the RPD must be no greater than 10%. For labeled compounds, the RPD must be no greater than 20%.

8.2.7 If any one of the criteria is not satisfied, repeat the routine calibration one more time, if possible, before analyzing any samples. If these criteria are still not satisfied (or a second injection could not be arranged), the entire routine calibration process must be reviewed. If no issues are found with the standard solution, corrective action for the instrumentation is needed.

8.3 Limit of Detection:

8.3.1 The laboratory will conduct one method detection limit (MDL) study per 40 CFR 136, Appendix B as required by Method 1613B. MDL studies will be presented as necessary in a statement of performance.

8.3.2 The detection limit is verified with each and every sample analyzed. Thus, once established, the MDL studies need not be repeated. The per-analyte and per-sample detection limit is established using the estimated detection limit (EDL).

8.3.3 The individual EDLs is tabulated on an annual basis to determine limits of detection and limits of quantitation. These are based on a procedure created by the American Chemical Society. This process and its underlying science and math are detailed in SOP EU027.

9.0 Procedure:

9.1 Interferences: Interferants coextracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalene, and polychlorinated alkyl dibenzofurans, that may be found at concentrations several orders of magnitude higher than that of the analytes of interest. PCDPEs can falsely appear to be PCDFs. These compounds are especially likely to be a problem with air samples, though they can occur in other samples. PCDPEs are removed from the sample extract by the florisil cleanup specified in this method.

9.2 Various organic molecules can be co-extracted with PCDD/Fs from samples and pass through the clean-up steps used in this procedure, showing up finally as non-specific interferences that affect the efficiency of ionization. These are known as quantitative interferences.

9.3 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

9.4 Selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Many materials used in this method require pre-cleaning.

9.5 In addition, analysts must avoid using PVC gloves, cosmetics, skin lotions, and any silicone based materials.

9.6 HRGC/HRMS Analysis:

9.6.1 Establish the operating conditions given used for either the ICAL acquisition or the last valid Concal.

9.6.2 At the beginning of each 12-hour shift during which analyses are performed, GC/MS system performance and calibration are verified for all PCDD/Fs and labeled compounds. For these tests, analysis of the CS3 calibration verification standard and the isomer specificity test standards, contained in the BCS, shall be used to verify all performance criteria. Only after all performance criteria are met may samples and quality control samples be analyzed.

9.6.3 Inject 1µL of the concentrated extract using on-column or splitless injection. The volume injected must be identical to the volume used for calibration.

9.6.4 Start the GC column initial isothermal hold upon injection. Start MS data collection after the solvent peak elutes. Stop data collection after the OCDD and OCDF have eluted. If only 2,3,7,8-TCDD and 2,3,7,8-TCDF are to be determined, you may stop data collection after elution of these compounds. Return the column to the initial temperature for analysis of the next extract or standard.

9.6.5 If an extract is to be reanalyzed and evaporation has occurred, do not add more JS. Rather, bring the extract back to its previous volume (e.g., 19µL) with pure tetradecane only.

9.6.6 MS Resolution – A static resolving power of at least 10,000 (10% valley definition) must be demonstrated at the appropriate m/z's before any analysis is performed. Static resolving power checks should be performed at the beginning and at the end of each 12-hour shift. However, at a minimum, the resolving power should be determined after

the completion of analysis, with the resolving power demonstrated to have held constant for the full duration of the analysis.

9.6.7 GC Resolution – The valley height between 2,3,7,8-TCDD and the other tetra-dioxin isomers at m/z 319.8965 shall not exceed 25%, and the valley height between 2,3,7,8-TCDF and the other tetra-furan isomers at m/z 303.9016 shall not exceed 40%. However, If the RRF for the compound meets routine calibration verification acceptance criteria. Corrective action must still be taken, even if data are accepted, to correct the resolution issue.

9.6.8 Sample Dilution:

9.6.8.1 If the SICP area at either quantitation m/z for any compound exceeds the calibration range of the system, the extract may be diluted to alleviate the problem.

9.6.8.2 The smallest dilution capable of bringing the signal to an unsaturated level should be used. If the dilution would cause any chromatographic peak associated with an ES compound to have a S/N below 10:1, re-extraction is indicated.

9.6.8.3 The size of the subsample taken for re-extraction should be based on the estimated actual concentration of the sample, with the goal being to place it in the upper 75% of the calibrated range of the system, in terms of concentration.

9.6.8.4 If a smaller sample size will not be representative of the entire sample, spike a larger amount of ES into the sample such that the final extract can be diluted to eliminate any saturated peaks in the SICPs.

10.0 Quality Control:

10.1 Performance evaluation samples – where available, performance evaluation (PE) samples containing known amounts of unlabeled 2,3,7,8-substituted PCDDs/PCDFs or other PCDD/PCDF congeners may be analyzed alongside routine field samples.

10.2 Duplicate samples may be analyzed to assess sample inhomogeneity. The special nature of isotope dilution does not lend to imprecision so long as samples are homogenous.

10.3 Method blanks should be prepared at a frequency of at least 5%, that is, one method blank for each group of up to 20 samples prepared at the same time, by the same procedures.

10.3.1 When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

10.3.2 The laboratory should not subtract the method blank results from those of any associated samples.

10.3.3 Ideally, the method blank should be free of hits for any native PCDD/Fs. When the customer does not specify use of the LCS/OPR, the ES recovery values are used to demonstrate adequate recovery of the analytes from a clean quality control matrix.

10.3.4 Compounds detected in the method blank will cause a B qualifier to be applied to those compounds detected in samples when the amount (NOT concentration) in the sample is not at least 10 times that in the method blank.

10.3.5 Compounds detected in the method blank also impact the measurement uncertainty of the same compounds in each sample, allowing the importance of the contamination to be placed in context.

10.3.6 The recovery of the ES should fall within the range 40%-130%.

10.4 System Performance Criteria:

10.4.1 The laboratory must document that all applicable system performance criteria (described in this section) were met before the analyses of any sample. This SOP provides recommended GC conditions that can be used to satisfy the required criteria. Figure 2 provides a typical 12-hr analysis sequence, whereby the response factors and mass spectrometer resolving power checks must be performed at the beginning and the end of each 12-hr period of operation. A GC column performance check is only required at the beginning of each 12-hr period during which samples are analyzed.

10.5 GC Column Performance:

10.5.1 Inject 1µL of an aliquot of the column performance check solution and acquire selected ion monitoring (SIM) data as described in this SOP, within a total cycle time of 1 second.

10.5.2 The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of 25 percent (Figure 3):

$$\text{Valley percent} = (x/y) \times (100\%)$$

Where:
x = measured as in Figure 3 from the 2,3,7,8-TCDD closest eluting isomer
y = the peak height of 2,3,7,8-TCDD

10.5.3 It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions described in this protocol. Their retention times are used to determine the five homologue retention time windows that are used for qualitative and quantitative purposes. All peaks (including labeled standards) should be labeled and identified on the chromatograms.

10.5.4 The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in

the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds. Allowable tolerance on the daily verification with the GC performance check solution should be better than 10 seconds for the absolute retention times of all the components of the mixture.

10.5.5 Particular caution should be exercised for the switching time between the last tetra-chlorinated congener (i.e., 1,2,8,9-TCDD) and the first penta-chlorinated congener (i.e., 1,3,4,6,8-PeCDF), as these two compounds elute very closely.

10.6 Mass Spectrometer Performance:

10.6.1 The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Static resolving power checks should be performed at the beginning and at the end of each 12-hr period of operation. However, it is recommended at a mimimum that a check of the static resolution be made and documented before and after each analysis. Corrective action must be implemented whenever the resolving power does not meet the requirement. Static resolving power demonstrated for a duration of greater than 12 hours indicates sufficient resolving power for the full duration.

10.6.2 Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass drift correction is mandatory. Select a lock-mass ion from the reference compound (PFK) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 4 offers some suggestions for the lock-mass ions.

10.6.3 However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in an increase in downtime for source cleaning.

10.6.4 Documentation of the instrument resolving power must then be accomplished by recording the the reference substance peaks in the mass range of each mass descriptor (see Figure 4). The minimum resolving power of 10,000 must be demonstrated on all ions for which there is no attributable cause for the poor resolution. The format of the peak profile representation (Figure 4) must allow manual determination of the resolution.

10.6.5 Please refer to SOP EU051 for a detailed explanation of the entire quality control system, as it is non-traditional, interlocking, and complex (i.e. PBMS/PBCA).

10.7 Batch Control Spike:

10.7.1 Interpretation of BCS results is a learned skill that takes some time to develop. The BCS results are calculated as response factors because, in certain cases, the BCS results may indicate that RRFs from the BCS are to be used instead of the average RRFs from the bracketing routine calibrations.

10.7.2 When the customer does not specify use of the LCS/OPR, the BCS provides assurance that native PCDD/F concentrations and labeled standard recovery values are accurate. The BCS is designed to detect and compensate for spiking errors. The LCS/OPR cannot definitively detect spiking errors, and it cannot compensate for them when detected. The LCS/OPR is intended to show sufficient recovery (accuracy) for all compounds in the assay.

10.7.3 Because the labeled standards behave identically to the unlabeled target analytes, the recovery values of the labeled standards are exactly the same as the recovery values of the unlabeled target analytes. Therefore, the BCS and method blank results, combined, perform the demonstration of measurement system accuracy and recovery that the LCS/OPR was intended to do but is incapable of doing in all cases. The BCS/LMB combination, by contrast, improves on the frequency with which the performance information is correct.

NOTE: No quality control system currently in use is 100% reliable in indicating inaccuracies. We can only hope to approach perfection not to achieve it. The BCS/LMB combination does a better job than the LCS/OPR system, but it can still be fooled. The best safeguard against data inaccuracy is human understanding of the technology and vigilance on the part of such individuals.

10.7.4 Every native PCDD/F and every labeled compound in the BCS has its RRF calculated in the same manner as a Concal.

10.7.5 The percent difference (PD) of each compound in the BCS is calculated relative to the average RRF of the routine calibrations bracketing the analytical sequence containing the BCS using the formula:

$$PD = \frac{(RRF_{BCS} - \overline{RRF_{CS3}})}{\overline{RRF_{CS3}}} \times 100\%$$

Where,
RRF_{BCS} = the response factor for the compound in the BCS, and
RRF_{CS3} = the average response factor for the compound in the two CS3 routine calibration injections.

10.7.6 The PD for each native PCDD/F must be no greater than 20%. The PD for each labeled compound must be no greater than 30%.

10.7.7 Spiking problems do not necessarily create failures of the BCS. Data reviewers should pay close attention to patterns in the BCS results that suggest systematic errors, even when the BCS meets the above acceptance criteria. When all compounds in a particular spiking solution (Table 2) have PDs greater than half of the maximum considered acceptable, the possibility of a spiking error should be considered. If evaluation of the data suggests a spiking error, the BCS should be treated as a failure (see Section 12.4.8), even if the criteria in Section 12.4.6 are met.

10.7.8 BCS failures can arise from a number of sources. The most common source is a spiking problem. The following steps will help determine the source of the failure. Note that no SOP can address all possible failures. The principles outlined below provide the framework for determining the correct course of action when a failure not addressed below arises.

10.7.8.1 First, determine if the failure applies to only a few compounds or to all compounds in one or more spiking solutions.

10.7.8.2 If the failure applies to only a few compounds and/or is distributed apparently randomly about the various spiking solutions, a spiking error is not indicated.

10.7.8.3 Compare the BCS result to the initial calibration verification (ICV) data. If the compounds having the highest PDs in the ICV also have the highest PDs in the BCS, the failure is likely due to a combination of instrument issues and inherent differences in the routine calibration and BCS spiking solutions. This situation will usually be accompanied by noticeable (but acceptable) excursions by the same compounds in the CS_3 injections. In this case, instrument maintenance is indicated. If the problem persists once the CS_3 injections shows excellent agreement with the ICAL, the BCS spiking solution in question should be discarded and a fresh one prepared.

10.7.8.4 Multiple failures with both positive and negative PDs suggest instrument problems. The instrument issues should be corrected. When the BCS is no longer showing this behavior, the instrument can be judged to have been successfully restored to normal performance.

10.7.8.5 When the BCS failure is not due to a spiking failure and does not appear due to a solution preparation issue, the data should be rejected, unless the failing compounds do not significantly affect the results being reported (typically defined by examining the data). The issue causing the BCS to fail must still be corrected, even if the data are accepted.

10.7.8.6 When the BCS failure is determined to be due to a solution preparation problem, the BCS RFs are to be used for quantifying the samples and determining labeled standard recovery values. The

errant spiking solution must still be re-prepared, even though the data are accepted.

10.7.8.7 If the BCS failure is determined to be due to a spiking error, the appropriate action will depend on which spiking solution(s) were improperly spiked.

10.7.8.8 Begin by comparing the CS and SS RFs. Differences in these two sets of RFs are highly informative for determining the cause of a BCS failure.

10.7.8.9 Examine Table 6 to see the patterns of PD behavior that accompany typical spiking errors. These errors can be of two types, A and B.

- A Type A error is one in which the spiking error is pervasive throughout the entire batch of samples. In this case, the BCS RFs are used for quantifying PCDD/Fs and determining labeled standard recovery values, unless the A_x was miss-spiked. In this latter case, the integrity of the BCS is indeterminate, and the associated data must be rejected.

- A Type B error occurs when it appears the miss-spiking event applied to only the BCS. Typically, this result is suggested when using the BCS RFs creates nonsensical data in the samples. For example, a case in which using BCS RFs results in a difference between CS and SS recovery values, even though no cleanup was performed for the sample, would indicate that the spiking error in question applied to only the BCS. In such cases, average CS_3 RFs are used.

10.8 Samples Ongoing Precision and Recovery:

10.8.1 For each PCDD/F and labeled compound, compare the concentration to the OPR limits given Method 1613-Table 6. If only 2,3,7,8-TCDD and 2,3,7,8-TCDF are to be determined, compare the concentration to the limits in Method 1613-Table 6a. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed.

10.8.2 If, however, any individual concentration falls outside of the range given, and the cause cannot be definitively determined, casting doubt on the accuracy of the data for that compound. Data may nonetheless be accepted if the data for that compound in all samples does not affect either the TEQ or total PCDD/Fs by more than 1%. Otherwise, correct the problem, re-prepare, extract, and clean up the sample batch and repeat the ongoing precision and recovery test.

11.0 Data Analysis and Calculations:

11.1 The formula below is to be used for determining the EDL.

$$EDL = \frac{2.5 \times Q_{ES} \times H_N \times A/H_{ES}}{A_{ES} \times Wt/Vol \times RRF}$$

Where,

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDD/Fs.

H_N = sum of the height of the noise level for each quantitation ion for the unlabeled PCDDs/PCDFs, measured as shown in Figure 3.

A/H_{ES} = Ratio of the area to the height for the labeled internal standard

A_{ES} = sum of the area for each quantitation ion (Table 4) for the labeled internal standard, measured as shown in Figure 3.

Wt./Vol = The weight or volume of the sample

Q_{ES} = The amount of ES spiked into the sample

RRF = relative response factor for the PCDD/F who EDL is being calculated

11.2 Calculate the 17 relative response factors (RRF) for unlabeled target analytes [$RRF_{(n)}$; n = 1 to 17] relative to their analogous internal standards and the 17 RRFs for the $^{13}C_{12}$-labeled internal standards [$RRF_{(IS)}$] relative to the three injection standards (Table 1) according to the following formulae (the formula for unlabeled target analytes is also used for FS, TS, and SS):

$$RRF_n = \frac{(A_n^1 + A_n^2) \times Q_{IS}}{(A_{IS}^1 + A_{IS}^2) \times Q_n}$$

$$RRF_{ES} = \frac{(A_{ES}^1 + A_{ES}^2) \times Q_{JS}}{(A_{JS}^1 + A_{JS}^2) \times Q_{ES}}$$

Where:

A_n^1 and A_n^2 = sum of the integrated ion abundances of the quantitation ions (Table 4) for unlabeled PCDDs/PCDFs,

A_{ES}^1 and A_{ES}^2 = sum of the integrated ion abundances of the quantitation ions (Table 4) for labeled PCDDs/PCDFs

A_{JS}^1 and A_{JS}^2 = sum of the integrated ion abundances of the quantitation ions (Table 4) for labeled PCDDs/PCDFs injection standards

Q_{ES} = quantity of the extraction standard injected (pg),

Q_{JS} = quantity of the injection standard injected (pg), and

Q_n = quantity of the unlabeled PCDD/PCDF analyte injected (pg)

Note: The formula for ES is also used for AS and CS.

Note: The RRF_n and RRF_{ES} are dimensionless quantities; the units used to express Q_{ES}, Q_{JS} and Q_n must be the same.

11.3 Calculate the average RRF values and their respective percent relative standard deviations (%RSD) for the calibration solutions:

$$\overline{RRF}_n = \frac{\sum_{j=1}^{N} RF_n}{N}$$

Where:

n represents a particular PCDD, j is the injection number (or calibration solution number; j = 1 to N) and N is the number of calibration standards.

11.4 Qualitative Determination: A PCDD/F or labeled compound is identified in a standard, blank, or sample when all of the criteria in below are met.

11.4.1 The signals for the two, exact m/z's in Table 4 must be present and must maximize within two seconds of one another.

11.4.2 The signal-to-noise ratio (S/N) for the GC peak at each exact m/z must be greater than or equal to 2.5.

11.4.3 The ratio of the integrated areas of the two, exact m/z's specified in Table 4 must be within the limits in Table 4, or within ±10% of the ratio in the most recently analyzed CS_3 standard (which could be from an ICAL).

11.4.4 If the ratio does not fall within these bounds but meets all other identification criteria, the compound is considered detected and will be reported with an estimated maximum possible concentration (EMPC). To calculate this concentration, adjust the area of the quantitation ion peak having the lower area of the two quantitation ion peaks. Raise the area until the ratio between the peaks equals the theoretical ratio. Then proceed with the calculation of concentration as normal using the adjusted peak area.

NOTE: Most EMPC detections are due to poor ion statistics accompanying the low S/N found near the detection limit. The limits set for the ratio of the peaks as an identification criterion have no special meaning in and of themselves. While the ratio of those peaks is well-defined by nature, the limits of statistical variance are not well-defined by humans. They are, in fact, arbitrary and selected because they “feel about right”. We cannot, therefore, say as a statement of fact that because a particular compound evinces a ratio of these peaks that is 15.1% different from the nominal ratio is not a PCDD/F while one that has a ratio that is 14.9% different from the nominal ratio is a PCDD/F.

NOTE: Due to software limitations and the possibility or irrational numbers, it may not be possible to adjust the area to exactly the number required to make the ratio exactly correct. Adjusting the area to the nearest whole number to the exact area required is sufficient

11.4.5 Confirmatory Analysis - When the concentration of any 2,3,7,8-chlorinated PCDD/F is significant (more than 25% of the toxic equivalency) and in question due to co-elution, a confirmatory analysis is indicated. A DB-225 column is recommended for confirmatory analyses.

11.5 Quantitative Determination:

11.5.1 Isotope Dilution Quantitation—By adding a known amount of a labeled compound to every sample prior to extraction, correction for recovery of the PCDD/F can be made because the PCDD/F and its labeled analog exhibit identical effects upon extraction and concentration and near-identical effects in gas chromatography.

11.5.2 Relative Response (RR) values are used in conjunction with the calibration data to determine concentrations directly, so long as labeled compound spiking levels are constant, using the following equation:

$$C_{Ax} = \frac{A_{Ax} \times Q_{ES}}{A_{ES} \times Wt./Vol. \times RF}$$

Where:

C_{Ax} = The concentration of the PCDD/F in the sample,

A_{Ax} = The summed area of the two peaks corresponding to the unlabeled, native target analyte,

Q_{ES} = The amount (in pg) of ES spiked into the sample,

A_{ES} = The summed area of the two peaks corresponding to the labeled standard corresponding to the native target analyte,

Wt./Vol. = the weight or volume of the sample,

RF = the average RF obtained from the CS_3 injections bracketing the analytical sequence.

11.5.3 Calculate a response factor for every labeled standard in the BCS. The CS standards must be calculated as both CS and SS in the BCS.

11.5.4 Any peaks representing non-2,3,7,8-substituted PCDD/Fs are quantitated using an average of the response factors from all of the labeled 2,3,7,8-isomers at the same level of chlorination with the same chemical backbone. For example, non-2,3,7,8-substituted hexa-chlorinated dioxins are quantified using the average response factor of the three 2,3,7,8-substituted hexa-chlorinated dioxins.

11.6 Labeled Compound Recovery:

11.6.1 Compute the recovery values of the labeled compounds using the average response factors determined from the routine calibrations bracketing the analytical sequence and the following equation:

$$\%Recovery = \frac{A_x \times Q_y \times 100\%}{A_y \times Q_x \times RF}$$

Where:

A_x = The summed area of the two peaks corresponding to the labeled compound whose recovery is being determined,

Q_y = The amount (in pg) of the reference labeled compound spiked into the sample (extract),

A_y = The summed area of the two peaks corresponding to the labeled standard against which the labeled compound whose recovery is being calculated is referenced,

Q_x = The amount (in pg) of the labeled compound whose recovery is being calculated spiked into the sample (extract),

RF = the average RF obtained from the CS_3 injections bracketing the analytical sequence.

11.6.2 In the above equation and the table below, x represents the labeled compound whose recovery is being calculated and y represents the labeled standard used as a reference for calculated the recovery. For example, FS recovery values (x) are calculated using the ES as the reference (y).

x	y
ES	JS
CS	JS
SS	ES
FS	ES
TS	ES
AS	JS

11.6.2.1 The CS and SS compounds are the same five labeled standards. When they have their RFs or recoveries determined relative to the JS, they are referred to as CS. When they have their RFs and recovery values determined relative to the ES, they are referred to as SS.

11.6.2.2 There is only one m/z monitored for the 37Cl-labeled standard.

11.6.3 The CS compounds should have recovery values calculated as both CS and SS, regardless of the sample type, as this information is invaluable in determining the cause of quality control problems.

11.7 Reporting units:

11.7.1 Aqueous samples — Report results in pg/L (parts-per-quadrillion).

11.7.2 Samples containing greater than 1% solids (soils, sediments, filter cake, compost) — Report results in pg/g based on the dry weight of the sample.

Report the percent solids so that the result may be converted to a wet weight basis.

11.7.3 Tissues — Report results in pg/g of wet tissue, not on the basis of the lipid content of the sample. Report the percent lipid content, so that the data user can calculate the concentration on a lipid basis if desired.

11.8 Reporting Levels:

11.8.1 The mean RRF from the routine calibration checks is used, rather than average ICAL RRFs, to quantify the sample results. This approach minimizes the measurement error (i.e. bias) otherwise knowingly introduced by using the ICAL. Results are reported to three significant figures for the PCDD/Fs and labeled compounds found in all standards, blanks, and samples.

11.8.2 All detected and EMPC-detected PCDD/Fs are reported. Customers may censor data as they see fit, but the laboratory will report any detections, including EMPCs.

11.8.3 Detections are determined by S/N alone, in conjunction with consideration of the EDL. If a concentration for a detected PCDD/F is calculated to be below its EDL, the compound is revised to be considered not-detected, as the uncertainty of the detection is considered to be too high in this scenario.

11.8.4 Compounds that are detected with an EMPC (due to peak area ratios) are reported as EMPCs and clearly indicated to have been detected as EMPCs, rather than a detection in which all identification criteria were met.

11.8.5 The total concentration of all isomers in an individual level of chlorination (i.e., total TCDD, total TCDF, total PeCDD, etc.) is reported by summing the concentrations of all isomers identified in that level of chlorination, including both 2,3,7,8-substituted and non-2,3,7,8-substituted isomers.

11.8.6 The toxic equivalency (TEQ) is also reported. The TEQ is reported with and without EMPCs included. Additionally, the TEQ is reported with non-detects treated as having a concentration of zero, a concentration of one-half the EDL, and a concentration equal to the EDL. The TEQ is calculated by multiplying the concentration for each 2,3,7,8-substituted PCDD/F by its toxic equivalency factor (TEF). There are multiple TEF schemes. By default, the two TEF schemes normally used are the International TEF (ITEF) scheme and the WHO-2005 TEF scheme.

12.0 Method Performance:

12.1 Refer to Performance Based Measurement System and Method Deviation SOP EU051 for measurement uncertainty information.

12.2 The laboratory, and each individual analyst, must demonstrate the capability to generate accurate and precise data using this method**.**

12.3 Traditional laboratory control samples (LCS/OPR) are used for this demonstration.

12.4 Each analyst will analyze four laboratory control samples. The average recovery for the four replicates for each native analyte must be between 70 to 130%, with an RSD of 20% or less. The mean recovery for each labeled standard must be within 40 to 130% (25 to 130% for OCDD and OCDF), with an RSD of 30% or less. Should the analyses fail to meet these criteria, the cause for failure will be investigated and the analyst will repeat the test once the cause has been addressed.

12.5 Recovery of labeled compounds from samples should be assessed and records should be maintained.

12.5.1 The recovery for each labeled standard should be within 40 to 130% (25 to 130% for OCDD and OCDF). Should any labeled standard fall outside these criteria, examine the chromatographic data for an understanding of why. High recovery values are usually the consequence of a quantitative interference. Low recovery values may indicate a data quality problem. If the chromatographic peaks associated with the poorly recovered labeled standard have a signal-to-noise ratio of at least 10:1, the data may be accepted, though the cause for the poor recovery should still be investigated. Signal-to-noise ratios below 10:1 indicate data should be rejected, unless the detection limit for the compound in question is adequate for the customer's purposes. QC Check Sample — Analyze the QC Check Sample periodically to assure the accuracy of calibration standards and the overall reliability of the analytical process. It is suggested that the QC Check Sample be analyzed at least quarterly.

12.5.2 Field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when the internal standard method is used.

12.6 Laboratory Performance Specification (LPS):

12.6.1 The LPS is determined annually by using the LODs determined during the annual LOD calculation as concentrations and calculating an ITEQ from them. The LPS otherwise remains constant, as it is a measure of historical performance. MDLs may be used instead of LODs until sufficient data are gathered to determine LODs.

12.6.2 For each sample, calculate the Basic TEQ by treating each compound's EDL as a concentration and using them to calculate the ITEQ.

12.6.3 Adjust the Basic TEQ for sample weight and volume, split factor, and dilution factor. The LODs are determined for specific weights and volumes. The Basic TEQ will be affected by sample size and must be normalized before comparison to the LPS. Splitting and diluting an extract increase the Basic TEQ artificially and must be adjusted for. Make the adjustment using this formula:

$$BasicTEQ_{adj.} = \frac{BasicTEQ \times W / V_{.}}{W / V_{nom} \times SF \times DF}$$

Where,

W/V = the weight or volume of the sample,

W/V_{nom} = the weight or volume used for LOD determination,

SF = split factor

DF = dilution factor

12.6.4 Compare the adjusted Basic TEQ to the LPS. If the adjusted Basic TEQ is no greater than the LPS, the test is successful. An adjusted Basic TEQ higher than the LPS indicates some type of analytical problem whose nature must be determined. A failed LPS does not necessarily indicate data must be rejected, but it absolutely must be investigated, the cause determined, the cause corrected.

12.7 Measurement Uncertainty:

12.7.1 Calculate $\%u_c$ for the TEQ as given in further detail in SOP EU051.

12.7.2 Compare the $\%u_c$ obtained for each sample to that specified by the customer or, if the customer did not specify an uncertainty limit, 20%.

12.7.3 If the $\%u_c$ exceeds the value determined in Section 12.7.1, determine if the sample is mostly non-detects or near-detection-limit concentrations. If so, the exceedance is due to the sample having very little PCDD/F content and does not, therefore, indicate a laboratory performance problem.

12.7.4 If the majority of sample concentrations are above the LODs, however, and $\%u_c$ exceedance indicates an analytical problem, the data should be rejected for that sample. Note that it is quite possible to accept all data in a batch save one sample based on the uncertainty of measurement.

12.8 Please refer to SOP EU051 for our PBMS insight to the quality control of data. This dual assessment approach results in situations wherein data may meet method/TNI criteria and be rejected and vice-versa. Of course, the ideal situation is that both method/TNI criteria and the PBCA show acceptable performance.

13.0 Pollution Prevention and Waste Management:

13.1 Waste must be disposed of in the appropriate laboratory waste streams.

13.2 The solvents used in this method pose little threat to the environment when managed properly. The solvent evaporation techniques used in this method are amenable to solvent recovery, and it is recommended that the laboratory recover solvents wherever feasible.

13.3 Standards should be prepared in volumes consistent with laboratory use to minimize disposal of standards.

13.4 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench

operations. Compliance is also required with any sewage discharge permits and regulations.

13.5 PCDD/Fs decompose above 800°C. Low-level waste such as absorbent paper, tissues, animal remains, and plastic gloves may be burned in an appropriate incinerator. Gross quantities (milligrams) should be packaged securely and disposed of through commercial or governmental channels that are capable of handling extremely toxic wastes.

14.0 References:

14.1 Y. Tondeur and W. F. Beckert, "Method 8290: Analytical Procedures and Quality Assurance for Multimedia Analysis of Polychlorinated Dibenzo-p-Dioxins and Dibenzofurans by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry," U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Las Vegas, NV.

14.2 Method 23 – Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources

14.3 Method 0023A – Sampling Method for Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources

14.4 Compendium Method TO-9A - Determination of Polychlorinated, Polybrominated and Brominated/Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in Ambient Air

14.5 Method 1613, Revision B, 1994: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS

14.6 Enthalpy Ultratrace SOPs EU046, Extraction-Preparation of Samples for PCDD-PCDFs

15.0 Tables, Diagrams, Flowcharts and Appendices:

- Table 1: Composition of the Seven Calibration Standard Solutions
- Table 2: Compositions of all Sample Fortification Solutions
- Table 3: Compounds that Comprise the Retention Time Window and GC Resolution Mixture for PCDD/Fs
- Table 4: Suggested Mass Descriptors for PCDD/F Analysis
- Table 5: Default Sample Quantities for Sample Preparation
- Table 6: BCS Failure Matrix
- Table 7: Tetra-Octa Dioxin/Furans Compounds and CAS Number
- Figure 1: Standards and Spike Flowchart
- Figure 2: Typical Analytical Sequence Flowchart
- Figure 3: Valley Determination for GC Resolution
- Figure 4: Resolution Check Example
- Figure 5: Peak Height and S/N Ratio Example

- Figure 6: Effect of Zero Threshold on Resolving Power
- Appendix A: Toxicity of Specific Dioxin/Furan Compounds
- Appendix B: HRGC/HRMS Equipment: Special Notations, Detail and Tips

Revision History:			
Revision #:	Date:	Author:	Comments:
1	6/6/2017	B. Vining	Revised in response to CAF-M1613-8290-J014
0	01/03/2017	B. Vining	New SOP

Table 1: Composition of the Seven Calibration Standard Solutions

Methods 23/0023A/8290/1613/TO-9A: ICAL (pg/mL)							
	CS0	CS1	CS2	CS3	CS4	CS5	CS6
Unlabeled Analytes							
2,3,7,8-TCDD	0.25	0.5	2	10	40	200	500
2,3,7,8-TCDF	0.25	0.5	2	10	40	200	500
1,2,3,7,8-PeCDD	1.25	2.5	10	50	200	1000	2500
1,2,3,7,8-PeCDF	1.25	2.5	10	50	200	1000	2500
2,3,4,7,8-PeCDF	1.25	2.5	10	50	200	1000	2500
1,2,3,4,7,8-HxCDD	1.25	2.5	10	50	200	1000	2500
1,2,3,6,7,8-HxCDD	1.25	2.5	10	50	200	1000	2500
1,2,3,7,8,9-HxCDD	1.25	2.5	10	50	200	1000	2500
1,2,3,4,7,8-HxCDF	1.25	2.5	10	50	200	1000	2500
1,2,3,6,7,8-HxCDF	1.25	2.5	10	50	200	1000	2500
1,2,3,7,8,9-HxCDF	1.25	2.5	10	50	200	1000	2500
2,3,4,6,7,8-HxCDF	1.25	2.5	10	50	200	1000	2500
1,2,3,4,6,7,8-HpCDD	1.25	2.5	10	50	200	1000	2500
1,2,3,4,6,7,8-HpCDF	1.25	2.5	10	50	200	1000	2500
1,2,3,4,7,8,9-HpCDF	1.25	2.5	10	50	200	1000	2500
OCDD	2.5	5	20	100	400	2000	5000
OCDF	2.5	5	20	100	400	2000	5000
Extraction Standards							
$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100	100	100
$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,7,8-PeCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,7,8-PeCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -2,3,4,7,8-PeCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,7,8-HxCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,6,7,8-HxCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,7,8,9-HxCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,7,8-HxCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,6,7,8-HxCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -2,3,4,6,7,8-HxCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,7,8,9-HxCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,6,7,8-HpCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,6,7,8-HpCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,7,8,9-HpCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -OCDD	200	200	200	200	200	200	200
$^{13}C_{12}$ -OCDF	200	200	200	200	200	200	200

	CS0	CS1	CS2	CS3	CS4	CS5	CS6
Cleanup or Sampling Standards							
$^{37}Cl_4$ -2,3,7,8-TCDD	-	0.5	2	10	40	200	-
$^{13}C_{12}$ -1,2,3,4,7-PeCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,6-PeCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,6,9-HxCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,6,8,9-HpCDF	100	100	100	100	100	100	100
Alternate Standards							
$^{13}C_{12}$ -1,3,6,8-TCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,3,6,8-TCDF	100	100	100	100	100	100	100
Field Concentration Standards							
$^{13}C_{12}$ -1,2,7,8-TCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,4,7,8-PeCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,6,8-HxCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,6,7,9-HpCDD	100	100	100	100	100	100	100
Transfer Standard							
$^{13}C_{12}$ -1,3,7,8-TCDD	100	100	100	100	100	100	100
Injection Standards							
$^{13}C_{12}$ -1,2,3,4-TCDD	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4-TCDF	100	100	100	100	100	100	100
$^{13}C_{12}$ -1,2,3,4,6,7-HxCDD	100	100	100	100	100	100	100

Table 2: Compositions of all Sample Fortification Solutions

Compositions of all sample fortification solutions used in this analytical protocol are such that the absolute amounts shown below are introduced inside the sample at the appropriate moment.

Compound	Amount Spiked (ng)	Amount Spiked_Air (ng)
Native (Unlabeled) Analytes		
2,3,7,8 -TCDD	0.2	0.4
1,2,3,7,8 -PeCDD	1	2
1,2,3,4,7,8 -HxCDD	1	2
1,2,3,6,7,8 -HxCDD	1	2
1,2,3,7,8,9 -HxCDD	1	2
1,2,3,4,6,7,8 -HpCDD	1	2
OCDD	2	4
2,3,7,8 -TCDF	0.2	0.4
1,2,3,7,8 -PeCDF	1	2
2,3,4,7,8 -PeCDF	1	2
1,2,3,4,7,8 -HxCDF	1	2
1,2,3,6,7,8 -HxCDF	1	2
2,3,4,6,7,8 -HxCDF	1	2
1,2,3,7,8,9 -HxCDF	1	2
1,2,3,4,6,7,8 -HpCDF	1	2
1,2,3,4,7,8,9 -HpCDF	1	2
OCDF	2	4
Compound	**Amount Spiked (ng)**	**Amount Spiked_Air (ng)**
Extraction Standards		
$^{13}C_{12}$-2,3,7,8 -TCDD	2	4
$^{13}C_{12}$-1,2,3,7,8 -PeCDD	2	4
$^{13}C_{12}$-1,2,3,4,7,8 -HxCDD	2	4
$^{13}C_{12}$-1,2,3,6,7,8 -HxCDD	2	4
$^{13}C_{12}$-1,2,3,7,8,9 -HxCDD	2	4
$^{13}C_{12}$-1,2,3,4,6,7,8 -HpCDD	2	4
$^{13}C_{12}$-OCDD	4	8
$^{13}C_{12}$-2,3,7,8 -TCDF	2	4
$^{13}C_{12}$-1,2,3,7,8 -PeCDF	2	4
$^{13}C_{12}$-2,3,4,7,8 -PeCDF	2	4
$^{13}C_{12}$-1,2,3,4,7,8 -HxCDF	2	4
$^{13}C_{12}$-1,2,3,6,7,8 -HxCDF	2	4
$^{13}C_{12}$-2,3,4,6,7,8 -HxCDF	2	4
$^{13}C_{12}$-1,2,3,7,8,9 -HxCDF	2	4
$^{13}C_{12}$-1,2,3,4,6,7,8 -HpCDF	2	4
$^{13}C_{12}$-1,2,3,4,7,8,9 -HpCDF	2	4
$^{13}C_{12}$-OCDF	4	8

Compound	Amount Spiked (ng)	Amount Spiked_Air (ng)
CS/SS - Cleanup or Sampling Standards		
$^{37}Cl_4$-2,3,7,8 -TCDD [SS or CS]	0.4	0.8
$^{13}C_{12}$-1,2,3,4,7 -PeCDD [SS or CS]	2	4
$^{13}C_{12}$-1,2,3,4,6 -PeCDF [SS or CS]	2	4
$^{13}C_{12}$-1,2,3,4,6,9 -HxCDF [SS or CS]	2	4
$^{13}C_{12}$-1,2,3,4,6,8,9 -HpCDF [SS or CS]	2	4
Compound	**Amount Spiked (ng)**	**Amount Spiked_Air (ng)**
AS - Alternate Standards		
$^{13}C_{12}$-1,3,6,8 -TCDD [CS] (BCS_3)	2	4
$^{13}C_{12}$-1,3,6,8 -TCDF [CS] (BCS_3)	2	4
Compound	**Amount Spiked (ng)**	**Amount Spiked_Air (ng)**
FS = Filter Extraction Standards (BCS_3)		
$^{13}C_{12}$-1,2,7,8 -TCDD	2	4
$^{13}C_{12}$-1,2,4,7,8 -PeCDD	2	4
$^{13}C_{12}$-1,2,3,4,6,8 -HxCDD	2	4
$^{13}C_{12}$-1,2,3,4,6,7,9 -HpCDD	2	4
TS = Transportation Rinse Concentration Standard (BCS_3)		
$^{13}C_{12}$-1,3,7,8 -TCDD	2	4
Compound	**Amount Spiked (ng)**	**Amount Spiked_Air (ng)**
JS - Injection Standards		
$^{13}C_{12}$-1,2,3,4 -TCDD	2	2
$^{13}C_{12}$-1,2,3,4 -TCDF	2	2
$^{13}C_{12}$-1,2,3,4,6,7 -HxCDD	2	2

Table 3: Compounds that Comprise the Retention Time Window and GC Resolution Mixture for PCDD/Fs

Compounds that comprise the retention time window and GC resolution mixture for PCDD/Fs. These are added to the BCS, but may also be analyzed as a separate standard. These standards are not quantitative.

Compound
RT Window/GC Resolution Standards
1,3,6,8 -TCDD
1,2,8,9-TCDD
1,2,3,9 -TCDD
1,2,3,7/1,2,3,8 -TCDD
1,2,4,7,9 -PeCDD
1,2,3,8,9 -PeCDD
1,2,4,6,7,9 -HxCDD
1,2,3,4,6,7-HxCDD
1,2,3,4,6,7,9-HpCDD
1,2,3,4,6,7,8 -HpCDD
1,3,6,8 -TCDF
1,2,8,9 -TCDF
2,3,4,7 -TCDF
1,2,3,9 -TCDF
1,3,4,6,8 -PeCDF
1,2,3,8,9 -PeCDF
1,2,3,4,6,8 -HxCDF
1,2,3,4,8,9 -HxCDF
1,2,3,4,6,7,8 -HpCDF
1,2,3,4,6,7,9 -HpCDF

Table 4: Suggested Mass Descriptors for PCDD/F Analysis

Function	1	Mass (Da)	Dwell (msec)	Inter-Channel Delay (msec)	Target Analyte	Ion-Abundance Ratio	Lower Limit	Upper Limit
Channel	1	303.9016	80	20	TCDF	0.77	0.65	0.89
Channel	2	305.8987	80	6	TCDF	0.77	0.65	0.89
Channel	3	315.9419	20	10	$^{13}C_{12}$-TCDF	0.77	0.65	0.89
Channel	4	316.9745	20	6	$^{13}C_{12}$-TCDF	0.77	0.65	0.89
Channel	5	316.9903	-	-	Lock Mass	N/A	-	-
Channel	6	316.9903	20	5	QC Check Ion	N/A	-	-
Channel	7	319.8965	80	10	TCDD	0.77	0.65	0.89
Channel	8	321.8936	80	6	TCDD	0.77	0.65	0.89
Channel	9	327.885	30	10	$^{37}Cl_4$-TCDD	N/A	-	-
Channel	10	331.9368	20	10	$^{13}C_{12}$-TCDD	0.77	0.65	0.89
Channel	11	333.9339	20	6	$^{13}C_{12}$-TCDD	0.77	0.65	0.89
Channel	12	339.8597	30	10	PeCDF	1.55	1.32	1.78
Channel	13	341.8568	30	6	PeCDF	1.55	1.32	1.78
Channel	14	375.8364	30	10	HxCDPE	N/A	-	-
Function	**2**	**Mass (Da)**	**Dwell (msec)**	**Inter-Channel Delay (msec)**	**Target Analyte**	**Ion-Abundance Ratio**	**Lower Limit**	**Upper Limit**
Channel	1	339.8597	80	20	PeCDF	1.55	1.32	1.78
Channel	2	341.8568	80	6	PeCDF	1.55	1.32	1.78
Channel	3	351.9	20	10	$^{13}C_{12}$-PeCDF	1.55	1.32	1.78
Channel	4	353.897	20	6	$^{13}C_{12}$-PeCDF	1.55	1.32	1.78
Channel	5	355.8546	80	10	PeCDD	1.55	1.32	1.78
Channel	6	357.8517	80	6	PeCDD	1.55	1.32	1.78
Channel	7	366.9884	-	-	Lock Mass	N/A	-	-
Channel	8	366.9884	20	5	QC Check Ion	N/A	-	-
Channel	9	367.8949	20	6	$^{13}C_{12}$-PeCDD	1.55	1.32	1.78
Channel	10	369.8919	20	6	$^{13}C_{12}$-PeCDD	1.55	1.32	1.78
Channel	11	409.7974	30	10	HpCDPE	N/A	-	-
Function	**3**	**Mass (Da)**	**Dwell (msec)**	**Inter-Channel Delay (msec)**	**Target Analyte**	**Ion-Abundance Ratio**	**Lower Limit**	**Upper Limit**
Channel	1	373.8207	80	20	HxCDF	1.24	1.05	1.43
Channel	2	375.8178	80	6	HxCDF	1.24	1.05	1.43
Channel	3	380.9855	-	-	Lock Mass	N/A	-	-
Channel	4	380.9855	20	5	QC Check Ion	N/A	-	-
Channel	5	383.8639	20	6	$^{13}C_{12}$-HxCDF	1.24	1.05	1.43
Channel	6	385.861	20	6	$^{13}C_{12}$-HxCDF	1.24	1.05	1.43
Channel	7	389.8156	80	10	HxCDD	1.24	1.05	1.43
Channel	8	391.8127	80	6	HxCDD	1.24	1.05	1.43
Channel	9	401.8559	20	10	$^{13}C_{12}$-HxCDD	1.24	1.05	1.43
Channel	10	403.853	20	6	$^{13}C_{12}$-HxCDD	1.24	1.05	1.43
Channel	11	445.7555	30	10	OCDPE	N/A	-	-
Function	**4**	**Mass (Da)**	**Dwell (msec)**	**Inter-Channel Delay (msec)**	**Target Analyte**	**Ion-Abundance Ratio**	**Lower Limit**	**Upper Limit**
Channel	1	407.7818	80	20	HpCDF	1.04	0.88	1.2
Channel	2	409.7788	80	6	HpCDF	1.04	0.88	1.2
Channel	3	417.8253	20	10	$^{13}C_{12}$-HpCDF	1.04	0.88	1.2
Channel	4	419.822	20	6	$^{13}C_{12}$-HpCDF	1.04	0.88	1.2
Channel	5	423.7767	80	10	HpCDD	1.04	0.88	1.2
Channel	6	425.7737	80	6	HpCDD	1.04	0.88	1.2
Channel	7	430.9836	-	-	Lock Mass	N/A	-	-
Channel	8	430.9836	20	5	QC Check Ion	N/A	-	-
Channel	9	435.8169	20	10	$^{13}C_{12}$-HpCDD	1.04	0.88	1.2
Channel	10	437.814	20	6	$^{13}C_{12}$-HpCDD	1.04	0.88	1.2
Channel	11	479.7165	30	10	NCDPE	N/A	-	-
Function	**5**	**Mass (Da)**	**Dwell (msec)**	**Inter-Channel Delay (msec)**	**Target Analyte**	**Ion-Abundance Ratio**	**Lower Limit**	**Upper Limit**
Channel	1	441.7428	50	20	OCDF	0.89	0.76	1.02
Channel	2	443.7398	50	6	OCDF	0.89	0.76	1.02
Channel	3	446.7402	20	10	alternate ion	-	0	0
Channel	4	448.7373	20	6	alternate ion	-	0	0
Channel	5	453.783	20	10	$^{13}C_{12}$-OCDF	0.89	0.76	1.02
Channel	6	454.9614	20	6	$^{13}C_{12}$-OCDF	0.89	0.76	1.02
Channel	7	454.9842	-	-	Lock Mass	N/A	-	-
Channel	8	454.9842	20	5	QC Check Ion	N/A	-	-
Channel	9	457.7377	50	6	OCDD	0.89	0.76	1.02
Channel	10	457.7771	20	6	OCDD	0.89	0.76	1.02
Channel	11	462.7352	20	10	alternate ion	-	0	0
Channel	12	464.7322	20	6	alternate ion	-	0	0
Channel	13	469.778	20	10	$^{13}C_{12}$-OCDD	0.89	0.76	1.02
Channel	14	471.775	20	6	$^{13}C_{12}$-OCDD	0.89	0.76	1.02
Channel	15	513.6775	30	10	DCDPE	N/A	-	-

Table 5: Default Sample Quantities for Sample Preparation

Sample Matrix	Example	%Solids	Phase	Quantity Extracted
Single-phase				
Aqueous	Non-Potable Water	<1	—3	1000 mL
Groundwater				
Treated wastewater				
Solid	Dry soil	>20	Solid	10 g
Compost				
Ash				
Organic	Waste solvent	<1	Organic	10 g
Waste oil				
Organic polymer				
Tissue	Fish	—	Organic	10 g
Human adipose				
Multi-phase				
Aqueous/Solid	Wet soil	1–30	Solid	10 g
Untreated effluent				
Digested municipal sludge				
Filter cake				
Paper pulp				
Organic/solid	Industrial sludge	1–100	Both	10 g
Oily waste				
Liquid/Liquid				
Aqueous/organic	In-process effluent	<1	Organic	10 g
Untreated effluent				
Drum waste				
Aqueous/organic/solid	Untreated effluent	>1	Organic & solid	10 g

Table 6: BCS Failure Matrix

Spiking Error	A_x	ES	CS	SS	AS	FS	TS	Type A Error Corrective Action
A_x low	L	-	-	-	-	-	-	Reject all data
A_x high	H	-	-	-	-	-	-	Reject all data
ES low	H	L	-	H	-	H	H	Use BCS RFs
ES high	L	H	-	L	-	L	L	Use BCS RFs
CS/SS high	-	-	H	H	-	-	-	Use BCS RFs
CS/SS low	-	-	L	L	-	-	-	Use BCS RFs
AS high	-	-	-	-	H	-	-	Use BCS RFs
AS low	-	-	-	-	L	-	-	Use BCS RFs
FS high	-	-	-	-	-	H	-	Use BCS RFs
FS low	-	-	-	-	-	L	-	Use BCS RFs
TS high	-	-	-	-	-	-	H	Use BCS RFs
TS low	-	-	-	-	-	-	L	Use BCS RFs
JS high	-	L	L	-	L	-	-	Use BCS RFs
JS low	-	H	H	-	H	-	-	Use BCS RFs

BCS Failure Matrix

- H indicates a positive PD greater than 10%.
- L indicates a negative PD greater than 10%.

Table 7: Tetra-Octa Dioxin/Furans Compounds and CAS Number

The following compounds have been determined by this method, but all 136 tetra-octa-chlorinated PCDD/Fs may be analyzed using this method:

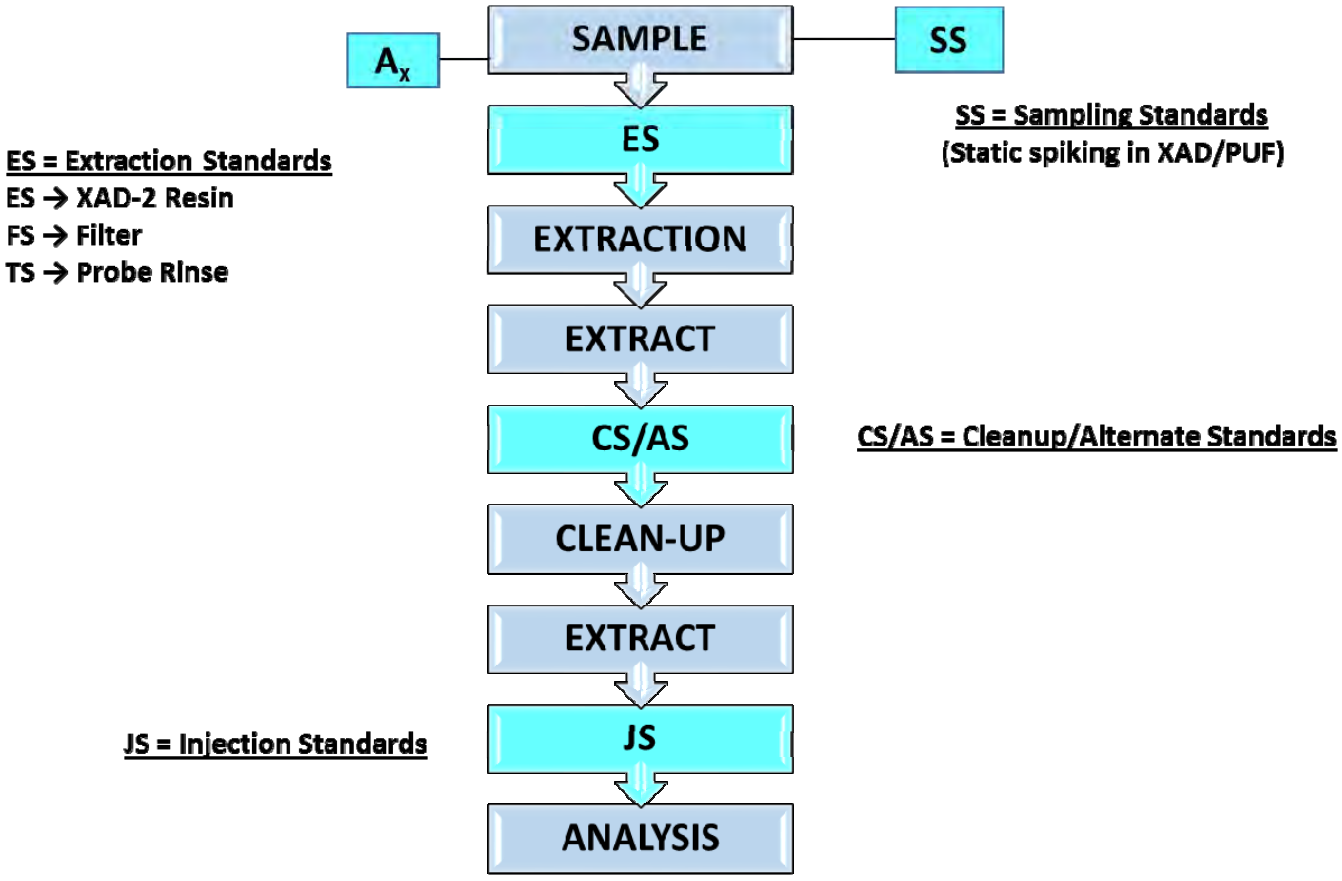
Compound	CAS Registry No.[a]
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6
1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	40321-76-4
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	57653-85-7
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	19408-74-3
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	35822-46-9
1,2,3,4,5,6,7,8-Octachlorodibenzo-p-dioxin (OCDD)	3268-87-9
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7
1,2,3,4,5,6,7,8-Octachlorodibenzofuran (OCDF)	39001-02-0
Tetrachlorodibenzo-p-dioxin (TCDD)	41903-57-5
Pentachlorodibenzo-p-dioxin (PeCDD)	36088-22-9
Hexachlorodibenzo-p-dioxin (HxCDD)	34465-46-8
Heptachlorodibenzo-p-dioxin (HpCDD)	41903-57-5
Tetrachlorodibenzofuran (TCDF)	36088-22-9
Pentachlorodibenzofuran (PeCDF)	34465-46-8
Hexachlorodibenzofuran (HxCDF)	41903-57-5
Heptachlorodibenzofuran (HpCDF)	36088-22-9

[a] Chemical Abstract Service Registry Number

Note: The method may also be used to determine mono-trichlorinated PCDD/Fs, should this request be made.

Figure 1: Standards and Spike Flowchart

The different types of standards used in this method and the standards against which they are referenced.



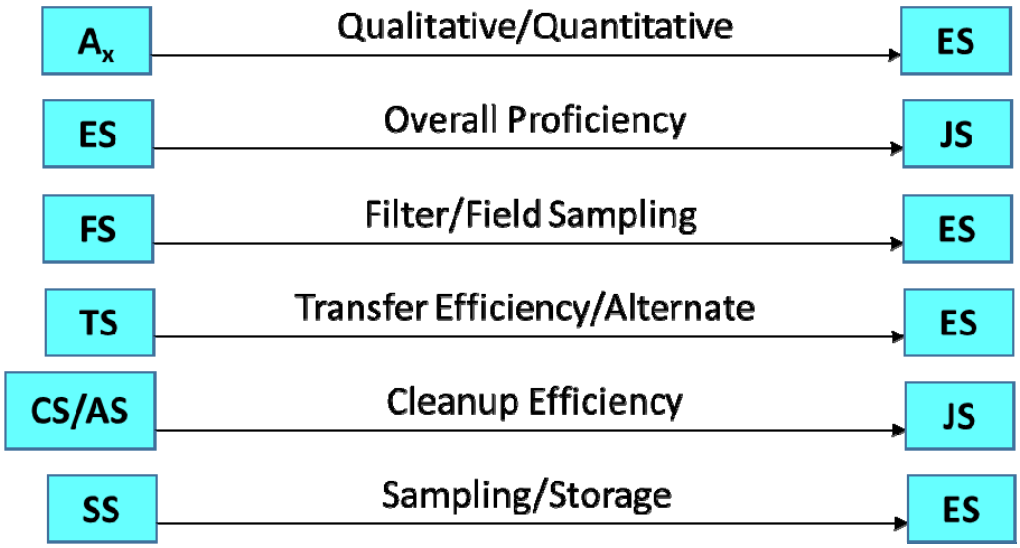


Figure 2: Typical Analytical Sequence Flowchart

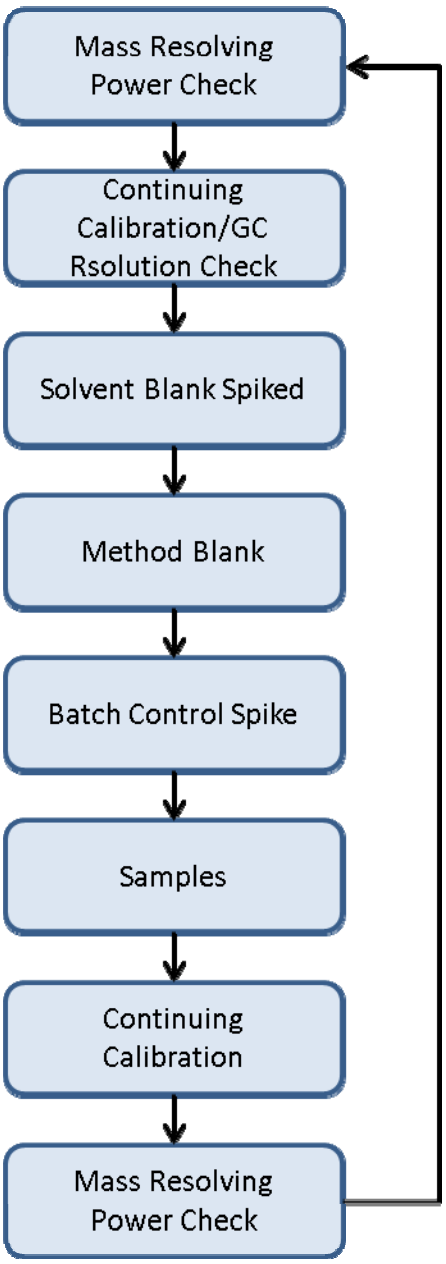


Figure 3: Valley Determination for GC Resolution

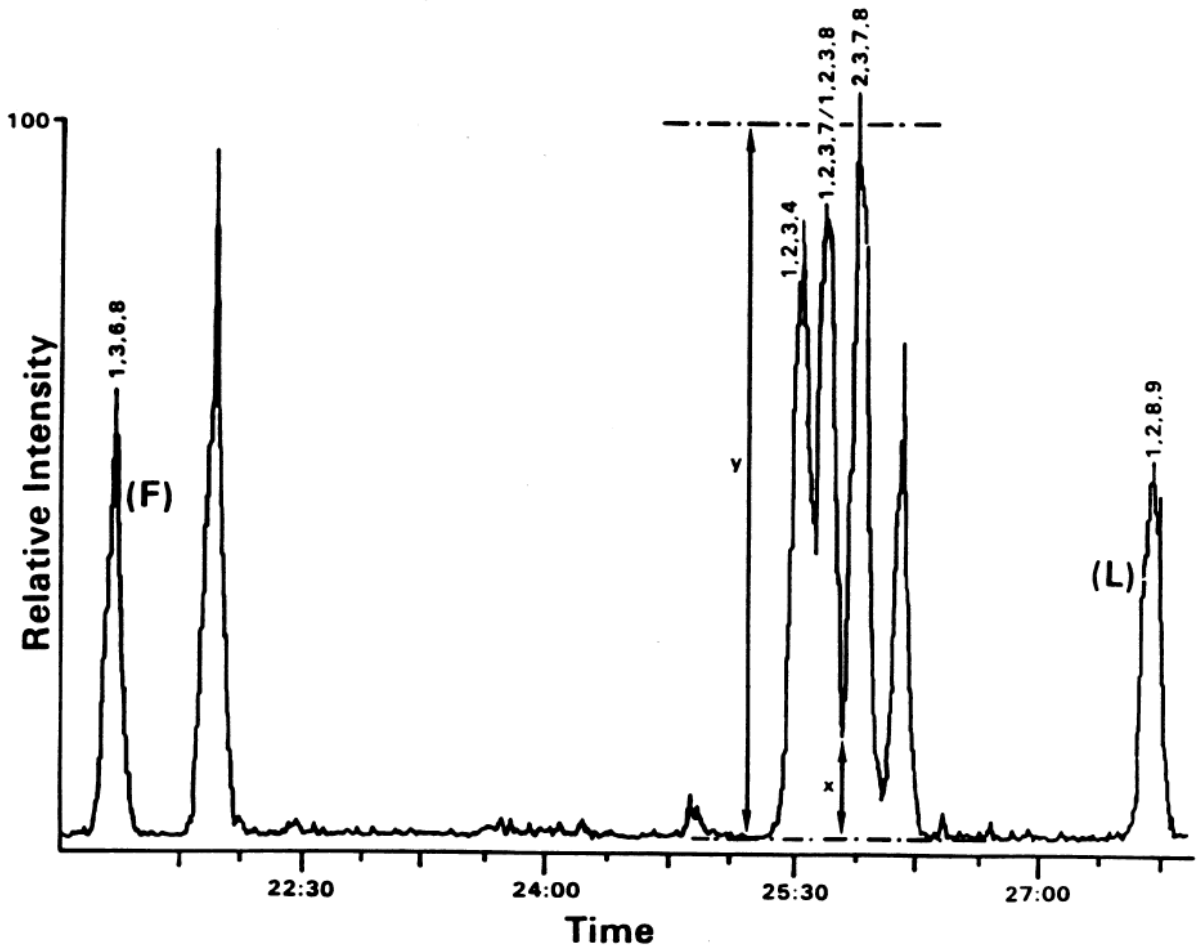
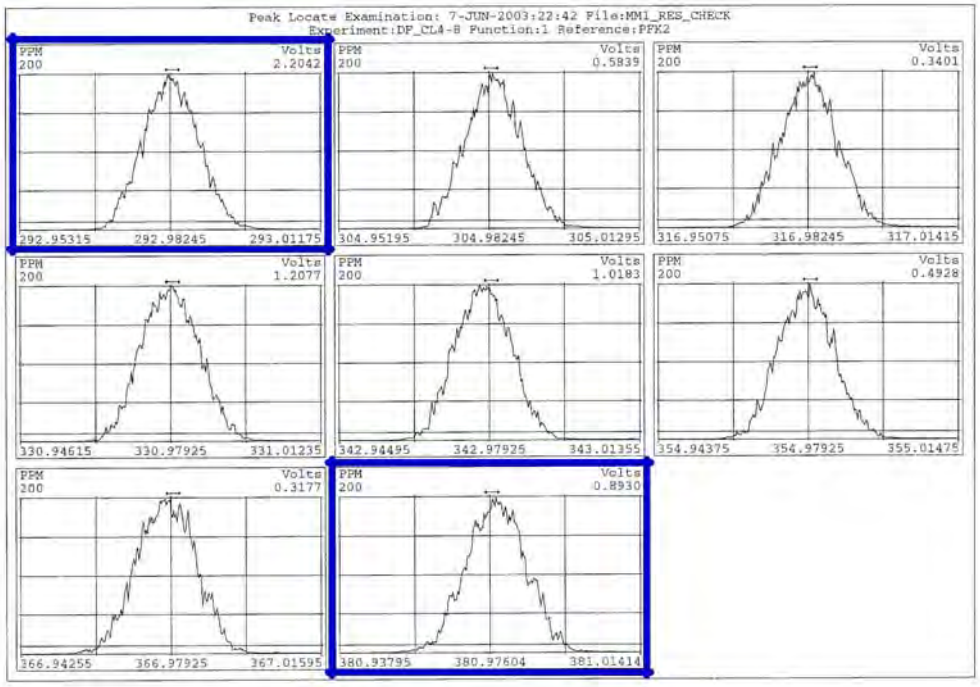
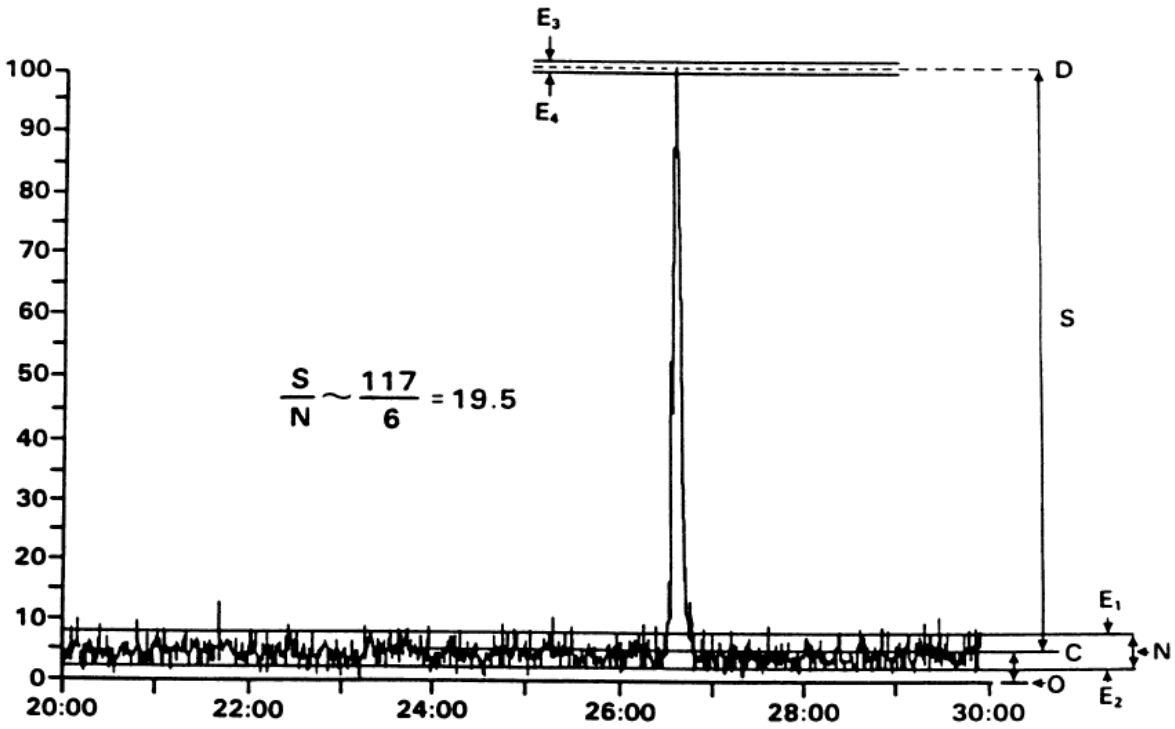


Figure 4: Resolution Check Example



Note: Illustration of the documentation of the mass resolution of 100 ppm (10,000 resolving power) for one of the four mass descriptors used in this SOP. A resolving power of 10,000 is achieved at the low mass m/z 293 (reference compound PFK) and the high-mass m/z 381 (reference compound PFK) when the peak width at 5 percent of the peak height is 100 ppm in mass. The window displayed is 200 ppm wide. By using all ions, the laboratory is able to assess and document the performance of the detector when operating at high resolving power. The peak shapes for all ions displayed within each of the mass descriptors are a good indicator of a properly tuned and well maintained instrument.

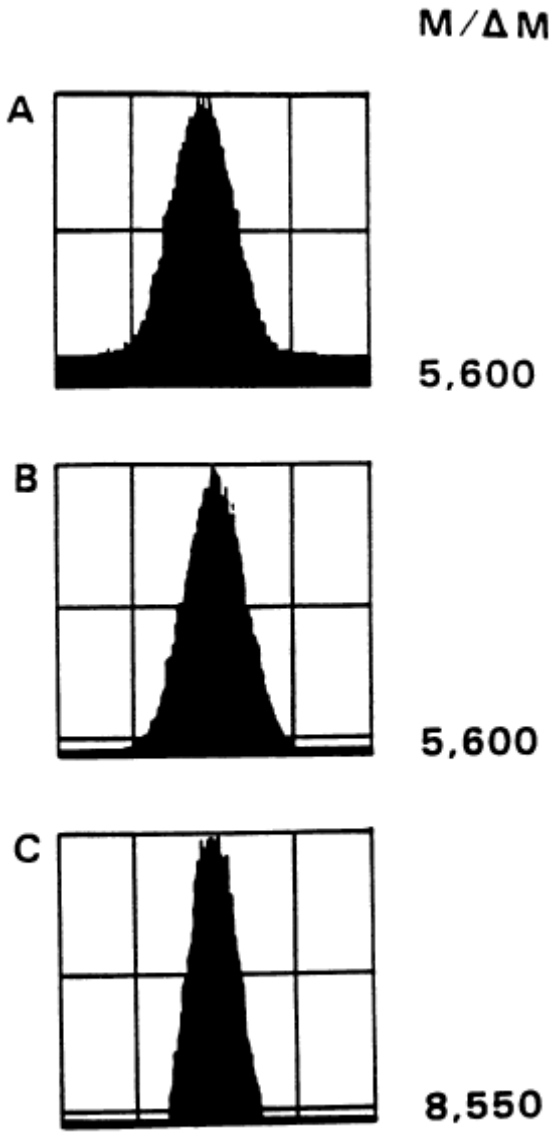
Figure 5: Peak Height and S/N Ratio Example



The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

Figure 6: Effect of Zero Threshold on Resolving Power



In this example, the true resolving power is 5,600.
A) The zero was set too high; no effect is observed upon the measurement of the resolving power.
B) The zero was adjusted properly.
C) The zero was set too low; this results in overestimating the actual resolving power because the peak-to-peak noise cannot be measured accurately.

Appendix A: Toxicity of Specific Dioxin/Furan Compounds

Because of the extreme toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCDDs or PCDFs. It is the responsibility of the laboratory personnel to ensure that safe handling procedures are employed.

The 2,3,7,8-TCDD isomer has been found to be acnegenic, carcinogenic, and teratogenic in laboratory animal studies. Other PCDDs and PCDFs containing chlorine atoms in positions 2,3,7,8 are known to have toxicities comparable to that of 2,3,7,8-TCDD. The analyst should note that finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. It is recommended that such samples be processed in a confined environment, such as a hood or a glove box. Laboratory personnel handling these types of samples should wear masks to prevent inhalation of dust.

The following precautions for safe handling of 2,3,7,8-TCDD in the laboratory were issued by Dow Chemical U.S.A. (revised 11/78) and were amended for use in conjunction with this method. The following statements on safe handling are as complete as possible on the basis of available toxicological information. The precautions for safe handling and use are necessarily general in nature since detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual use. Assistance in evaluating the health hazards of particular plant conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. The 2,3,7,8-TCDD isomer is extremely toxic to certain kinds of laboratory animals. However, it has been handled for years without injury in analytical and biological laboratories. Many techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8-TCDD.

Appendix B: HRGC/HRMS Equipment: Special Notations, Detail and Tips

GC injection port -- The use of splitless injection techniques is recommended. On-column 1µL injections can be used on the 60-m DB-5 column. The use of a moving needle injection port is also acceptable.

- NOTE: Use of an electronic pressure controller (EPC) is highly recommended to ensure higher reproducibility in gas flow and higher retention time stability

GC/MS interface -- The GC/MS interface components should withstand 350°C. The interface must be handled so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel™, or equivalent, ferrules are recommended.

Mass spectrometer -- The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley).

MassLynx™ Data system -- A dedicated data system is employed to control the rapid multiple-ion monitoring process and to acquire the data. Quantitation data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantitation's are reported based upon computer-generated peaks.

- NOTE: The detector ADC zero setting must allow peak-to-peak measurement of the noise on the base line of every monitored channel and allow for good estimation of the instrument resolving power. The effect of different zero settings on the measured resolving power is shown in Figure 6. Under no circumstances should be ADC zero setting be manipulated to make the resolving power appear to be better than it is.

GC Columns:

- 60-m DB-5MS (J&W Scientific) or equivalent fused-silica capillary column. To have an isomer-specific determination of 2,3,7,8-TCDD and to allow the quantitation of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, use of this column is recommended. With regard to 2,3,7,8-TCDF in actual samples, the peak height of the close-eluting isomer with shorter retention time than 2,3,7,8-TCDF shall not be more than 10 times the size of the 2,3,7,8-TCDF isomer. If it is so, the laboratory has the option to analyze a BCS (passing the acceptable criteria) with a similar ratio between the two isomers or analyze the sample extract using a DB-225 (or equivalent) column.
    - NOTE: The column is capable of achieving superior separation of 2,3,7,8-TCDD relative to the DB-5 column specified by most EPA published dioxin analysis methods (5-10 percent valley on DB-5MS vs. 15-25 percent on the DB-5). It is also capable of measuring the 2,3,7,8-TCDF congener in an isomer-specific mode (i.e., the role of the DB-225). The isomer-specificity demonstration is based on

"enfolded performance" (a concept discussed in an internally developed fitness-for-purpose PBMS version of M23) whereby the actual GC column performance is assessed in real time and in the presence of the corresponding interferences (i.e., close-eluting TCDD and TCDF isomers). It is worth noting that even, for example, if the valley between 2,3,7,8-TCDD and its closest eluting isomers is less than 25% as per M23, a 10% valley leading to a failed batch control spike (BCS; vide infra) requires column maintenance. Unlike the DB-5, the DB-5MS (or equivalent) is also capable of separating the three 2,3,7,8-substituted HxCDD isomers from non-2,3,7,8-HxCDD isomers (i.e., 1,2,3,4,6,7-HxCDD). This separation allows the laboratory to establish and document the GC column performance for components with higher retention times on a per-sample basis at the time of the sample analysis (i.e., 13C12-1,2,3,4,6,7-HxCDD can be used as an Injection Standard instead of a "precious" 2,3,7,8-substituted 13C12-1,2,3,7,8,9-HxCDD). The GC column performance for higher molecular weight target analytes is assessed and documented as part of the analyses of the BCS. Finally, the DB-5MS (or equivalent) column is now the column of choice adopted nearly universally in other countries.

- 30-m DB-225 (J&W Scientific) or equivalent fused-silica capillary column for confirmatory analysis when indicated.

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Enthalpy Analytical – Wilmington, NC (Ultratrace)
Standard Operating Procedure

Extraction-Preparation of Samples for PCDD-PCDFs

Enthalpy Ultratrace SOP #	EU046	Revision #	0
Author	Robin Appelle	Date Authored	01/03/2017
Revised by	N/A	Date Revised	N/A

Pages: 23

Technical Director/designee Approval: _____ Approval Date: 13 JUN 17

Quality Manager/designee Review: _____ Review Date: 13-June-17

Effective Date: 13 JUN 17

1.0 Scope and Application:

This SOP is applicable to the extraction/preparation of polychlorinated dibenzo-p-dioxins (PCDD's) and polychlorinated dibenzofurans (PCDF's), from all sample matrices incorporating special requirements of the different methods utilized (Methods 23, 0023A, TO-9A, 1613, 8290).

2.0 Summary of Method:

2.1 This procedure uses matrix-specific extraction, analyte-specific cleanup, transfer and spiking procedures to extract and finally prepare the sample for analysis by high resolution capillary column gas chromatography/high-resolution mass spectrometry (HRGC/HRMS).

2.2 The PCDD's and PCDF's are extracted from the sample, separated by high-resolution gas chromatography (HRGC), and measured by high-resolution mass spectrometry (HRMS). Refer to SOP EU045 for details of the analytical procedure.

3.0 Definitions:

3.1 CLL – Continuous Liquid-Liquid is a type of extraction technique utilized by the lab for water samples.

3.2 BCS – Batch Control Spike is a QC sample specifically introduced for comprehensive stable isotope-dilution mass spectrometric methodologies in order to ensure the reliability and the accuracy (the bias and the precision) of the determinations.

3.3 MB – Method Blank is a QC sample required with the analysis of any batch of samples, the method blank must be subjected to the same extraction and cleanup procedures as the actual samples.

3.4 SDS – Soxhlet Dean-Stark refers to an extraction technique utilized by the prep lab

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3.5 AS – Alternate Standards are added to the sample extract before the fractionation step in order to help monitor selective losses of specific isomers/congeners to ensure that no distortion of the congener profiles occurs during the fractionation step. AS response factors are determined relative to the JS.

3.6 CS – Cleanup Standards are added to the sample extract prior to the fractionation step. The role of the CS is to offer specific feedback to the laboratory regarding the fractionation step's efficiency, independent of the extraction step. CS recoveries are computed using a response factor relative to the JS.

3.7 ES – Extraction Standards are introduced (for all sample types) into the sample matrix prior to the extraction step (variously named internal standards, surrogates, and/or labeled internal standards in promulgated methods). Ideally, the ES should be introduced inside the sample before any step capable of modifying the sample's composition. The role of the ES is to monitor the extraction-fractionation efficiencies as well as to qualitatively and quantitatively characterize the target analytes. ES recoveries are determined relative to the Injection Standards (i.e. their response factors are determined relative to the corresponding JS). In addition, ES are used to establish the relative response factors for other labeled standards, which are used to monitor (independent of the extraction/fractionation) particular steps such as the sampling and storage conditions of stack flue gases and ambient air samples (SS), the field concentration of the air sampling component solvent rinses (FS), and the laboratory's ability to transfer the concentrated air sampling equipment rinses inside the extraction vessel (TS).

3.8 SS – Surrogate Standard or Sampling Standard (variously named field standards or field surrogates in promulgated methods) are introduced inside the sampling media before the sampling event. SS recoveries are computed in such a manner as to correct for losses occurring during the extraction-fractionation-concentration steps so that feedback on performance is specific to the sampling event.

3.9 TS – Transfer Standard used to distinguish a loss of FS during the concentration step, versus a non-quantitative transfer step by the laboratory during the transfer of the concentrated rinses inside the extraction vessel. TS response factors are determined relative to the ES in order to correct for losses that occur during the extraction-fractionation steps.

3.10 FS – Field Concentration Standard – Although primarily introduced for PCDD/F flue gas air sampling trains, the concept of the FS can be expanded to other target analytes wherever appropriate and feasible, or other sample types, depending upon the availability of labeled standards. FS are fortified along with adequate "keepers" inside containers where solvent rinses of the stack sampling equipment are collected, following a sampling event. The role of the FS is to monitor losses that might occur during field concentration (or wherever applicable, the laboratory concentration) of the rinses prior to shipment to the laboratory. In such circumstances, the response factor of the FS is determined relative to the ES. Concentrating rinses in the field reduces the risks, liabilities, complications, and costs normally associated with the shipment of dangerous goods. These may also be used to assess the extraction of

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compounds from the particulate matter found on modified method 5 air sampling trains.

3.11 Ax – Native Standards that are spiked into QC samples only.

3.12 JS – Injection Standards (variously named internal standards or recovery standards in promulgated methods) are used during the preparation of the final extract just before the injection of an aliquot of the final extract onto the GC column. JS are used to establish the relative response factors for a number of the labeled standards used throughout the procedure (e.g. ES, CS, AS) and to determine their recoveries up until the point of the JS addition. An extract previously analyzed may require an additional cleanup step prior to a reanalysis. Since the JS is already present before this additional cleanup step, and unless a different JS is introduced before the analysis, the recoveries measured on the "cleaned" extract contain no valuable information regarding the additional cleanup step. Furthermore, for practical reasons, the JS addition is sometimes conducted prior to the final transfer and/or for a concentration step. This protocol recognizes that these practices are acceptable as long as the performance-based criteria are met. In other words, should significant evaporative losses occur the laboratory will experience difficulties meeting the performance-based criteria

3.13 FH/BH – Front Half/Back Half, for stack gas samples where the client request the front half and the back half of the sample train are separated and the fractions are extracted and analyzed as such. The client sample ID will have -FH or -BH appended to it to differentiate the samples (The front half is the front half rinses and filter, the back half is the back half rinses and the XAD).

4.0 Safety:

4.1 Personal Protection Equipment (PPE) such as safety glasses, disposable Nitrile gloves, and apron or lab coat, at a minimum, should be worn at all time when handling samples.

4.2 All samples should be considered hazardous. All personnel working in the laboratory are required to follow the Enthalpy Analytical, Inc. Safety and Health Plan.

4.3 Laboratory Cleanliness:

4.3.1 Personal hygiene: Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift).

4.3.2 Decontamination of personnel: Apply a mild soap with plenty of scrubbing action.

4.3.3 Wipe tests: A useful method for determining cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper, extract the filter paper and analyze the extract.

4.3.4 The results and decision-making processes are based on the presence of 2,3,7,8-substituted PCDDs/PCDFs.

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5.0 Equipment and Supplies:

5.1 Soxhlet Extraction Apparatus Glassware: Dean-stark, thimble holder, thimble with extra coarse frit, 500mL round bottom flask, double jacketed condenser

5.2 Heating Mantles with temperature controllers

5.3 Chiller

5.4 Continuous Liquid-Liquid (CLL) Extraction apparatus

5.5 Stainless Steel Forceps – Fisher Scientific brand or equivalent

5.6 Cleanup/Drying Columns (glass/disposable, various sizes) for Acid/Base cleanup, florisil cleanup and carbon cleanup – Pyrex or equivalent

5.7 125mL Round bottom flasks

5.8 Glass Beakers (various sizes)

5.9 Glass Jars – 4 ounce

5.10 Aluminum Foil

5.11 Pasteur Pipets and bulbs

5.12 Kim Wipes

5.13 Nitrile Gloves

5.14 Glass vials with lids (for air split/archive)

5.15 HRGC/HRMS Glass vials with screw top lids

5.16 Color tape and tape dispenser

5.17 Label maker and labels (various sizes) – Dymo or equivalent

5.18 PTFE Solvent squeeze bottles

5.19 PTFE Boiling ships

5.20 18” Glass Stir rod – Custom made by Prism Research Glass

5.21 Calibrated Pipettor for spiking and disposable tips

5.22 Solvent Bottle top pumps (calibrated and adjustable volume capability)

5.23 Roto-Vap with temperature controlled water bath

6.0 Reagents and Standards:

6.1 Glasswool

6.2 Carbon Ax-21

6.3 XAD-2 Resin

6.4 PUF

6.5 Solvents (Pesticide, ACS or HPLC grade): Toluene, Hexane, Dichloromethane, Acetone, Tetradecane

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6.6 Silica Gel

6.6.1 Acid Treated Silica Gel

6.6.2 Base Treated Silica Gel

6.7 Silver Nitrate

6.8 Sodium Sulfate

6.9 Diatomaceous earth – Hyromatrix™ , Fisher , Catalog # NC0149934

6.10 Florisil

6.11 Nitrogen

6.12 Refer to SOP EU045 for detailed information on spiking standards/solutions

7.0 Sample Preservation, Storage, and Handling:

7.1 Air Samples:

7.1.1 XAD Traps, filters, PUFs and impinger and DI Catch water fractions must be stored cold at 4ºC ± 2°C.

7.1.2 Solvent rinses need to be stored at room temperature.

7.2 Water, Soil, Sediment, and Other Matrices (excluding biological tissue or serum):

7.2.1 Samples should be collected in amber glass jars. If the lab provides the sampling jars, the jars must be pre-cleaned amber glass jars.

7.2.2 Samples are to arrive at temperatures between at 4ºC ± 2°C. Notify clients if samples arrive outside these limits.

7.3 All samples (except solvents from air samples) must be stored in the dark, extracted within 30 days of collection and analyzed within 45 days of extraction or per method requirements.

7.4 Fish and adipose tissue samples should be stored at < 10°C in the dark, extracted within 30 days and analyzed within 45 days of collection.

7.5 Some unusual matrices (chemicals, printed circuit boards, etc.) may not require refrigeration.

7.6 All samples are retained for 90 days unless we are specifically requested by the clients to either return the samples or store them for a longer period of time.

7.6.1 Method 23 and 0023A samples extract archives (after split but before cleanup) are stored at room temperature in labeled, precleaned, glass vials with screw top.

7.6.2 Retain the archived portion of the sample in order to have a backup of the air sample extract to go back to if any problems occur during the rest of the preparation/analysis of the samples.

7.6.3 Disposal of this extract archive is done 90 days after analysis is complete and final report delivered to the client.

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7.7 The final extracts are labeled and stored in Autosampler glass vials with screw top lids and placed in a plastic storage box after analysis and stored at room temperature in the HRGC/HRMS area.

8.0 Procedure:

8.1 Refer to SOP EU038 for sorbent module prep and spiking, filter prep and creation of MB and BCS_3 per sorbent module order before shipping out into the field for use.

8.2 Sample conditioning - Sample conditioning involves modifying the physical form of the sample so that the PCDD/Fs can be extracted efficiently. In general, the samples must be in a liquid form or in the form of finely divided solids in order for efficient extraction to take place.

8.2.1 Sample conditioning must be kept to the minimum necessary to effect a successful analysis.

8.2.2 Sample conditioning necessarily changes the nature of the sample, making it unavoidably less representative of the sample submitted to the laboratory.

8.2.3 The analyzed sample is defined as the material spiked with labeled standards for analysis.

8.2.4 The goal of sample conditioning is to carry out the modifications necessary while keeping the material to be spiked as close to what the customer submitted as possible. Once the material is spiked properly, as many modifications as needed may be performed.

8.3 For samples known or expected to contain high levels of the PCDD/Fs, the smallest sample size representative of the entire sample should be used.

8.4 For all samples, the blank and, if the customer specifies its use, the IPR/OPR must be processed through the same steps as the sample to check for contamination and losses in the preparation processes.

8.5 Spiking:

8.5.1 The spiking step is a decisive step; it is through the spiking step that this SOP's entire set of objectives are accomplished, which are to qualitatively and quantitatively characterize what is in the sample.

8.5.2 The spiking step is not independent. For example, it is necessary to understand that data effectiveness (i.e. the reporting step) depends upon the accuracy of the delivery of the spike compounds and their integration inside the matrix. Similarly, a change in the spiking profile (e.g. in the amount spiked at the beginning of the process) impacts the analysis and the calibration of the measurement system.

8.5.3 In the context of isotope dilution, the importance of the spiking step is overriding. The goal is to introduce the spiked compounds so that they mimic, as closely as possible, endogenous compounds across the entire subsample undergoing extraction.

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8.5.4 Because of the key relationships between spiking and other steps, it is necessary to design the spiking profile accordingly. For example, the sample size, the final extract volume, the acceptable extraction standard recoveries, and the calibration curve should be considered carefully when settling on a particular spiking profile. If the sample is only analyzed for PCDD/Fs, and if the sample is suspected to contain pentachlorophenol, the laboratory should adjust the pH to less than 2 with sulfuric acid prior to spiking with acetone, if acetone is used to help overcome the water barrier during integration of the spikes.

8.5.5 Each matrix type may require a different approach to spiking to ensure the proper integration and dispersal of spiked compounds. In addition, spiking the sample correctly can be challenging for multi-component samples and those of unusual size or complexity. The loop closes with the timing of the spike (i.e. when the spike is carried-out) since this step defines the sample.

8.5.6 The spiking of the labeled extraction standards must occur prior to steps that could result in a change of the sample's composition, and definitely before sample extraction. For example, spiking of the ES should take place before drying or the addition of surface-enhancing agents (i.e. the sample matrix is the one that should be spiked). In a similar manner, the addition of the CS should take place prior to any fractionation step.

8.5.7 In addition to careful selection of the labeled standards, there are three interdependent aspects to consider when spiking a sample to ensure that it is done in a manner consistent with isotope dilution.

8.5.8 The location where spike compounds are applied is especially challenging for multi-component, multi-matrix, and voluminous samples.

8.5.9 Integration of the spiked compounds should be carried out in a manner such that the spiked compounds mimic the endogenous compounds.

8.5.10 Dispersal relates to the need to spike the labeled standards in such a way that the ratio of endogenous and ES compounds remains constant throughout the sample.

8.5.11 There are numerous sources of errors associated with spiking. A number of them, if systematized, can be compensated for through judicious use of controls such as the BCS. For example, the preparation of spiking solutions, storage conditions and usage, as well as the delivery of the labeled standards are sources of systematic errors. The laboratory should align its spiking procedures with the BCS's purpose in order to take advantage of the BCS.

8.5.12 The spiking volume depends upon the technique followed by the chemist as well as the delivery (i.e. the delivery device, its calibration, and the delivery volume setup). If consistency is maintained, systematic errors associated with the spiking volume can be self-corrected with the BCS.

8.5.13 The spiking solution's integrity refers to the quality of the solution (i.e. its preparation, the concentration of labeled compounds, the means of storage,

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and its usage). If consistency is maintained, systematic errors associated with the spiking solution can be self-corrected with the BCS.

8.6 Aqueous Samples

8.6.1 Because PCDD/Fs may be bound to suspended particles, the preparation of aqueous samples is dependent on the solids content of the sample.

8.6.2 Aqueous samples visibly absent particles are conditioned per Section 8.11 and extracted directly using the separatory funnel, CLL or SPE techniques described in this SOP.

8.6.3 Aqueous samples containing visible particles and containing less than one percent suspended solids are conditioned using the procedure in Section 8.13.

8.6.4 After conditioning, the sample is filtered using the SPE technique. After filtration, the particles and filter are extracted using the SDS procedures described in this SOP. Alternatively, the unfiltered sample can be extracted via CLL.

8.6.5 For aqueous samples containing one percent solids or greater, a sample aliquot sufficient to provide 10 g of dry solids is used, as described in Section 8.15.

8.7 Solid samples are prepared and extracted via the SDS, using the procedures described in this SOP.

8.8 Procedures for grinding, homogenization, and blending of various sample phases are given in Sections 8.16 thru 8.18.

8.9 Tissue samples: Conditioning procedures for fish and other tissues are given in Section 8.17 and 8.18.

8.10 Determination of Percent Suspended Solids:

NOTE: This aliquot is used for determining the solids content of the sample only, not for determination of PCDDFs.

8.10.1 Aqueous liquids and multi-phase samples consisting of mainly an aqueous phase:

- Weigh a 0.45 µm filter to three significant figures.
- Filter 10.0 ± 0.02 mL of well-mixed sample through the filter.
- Dry the filter a minimum of 2 hours at 110 ±5°C
- Calculate percent solids as follows:

$$\%solids = \frac{W_D - W_F}{W_S} \times 100\%$$

Where:
W_D = weight of sample and filter after drying
W_F = weight of the filter
W_S = Weight of the sample before drying

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8.10.2 Non-aqueous liquids, solids, semi-solid samples, and multi-phase samples in which the main phase is not aqueous (does not apply to tissue samples):

- Weigh 2 to 5g of sample to three significant figures in a weigh boat.
- Dry a minimum of 12 hours at 110 ±5°C.
- Calculate percent solids as follows:

$$\%solids = \frac{W_D}{W_S} \times 100\%$$

Where:
W_D = weight of sample after drying
W_S = Weight of the sample before drying

8.11 Conditioning of Aqueous Samples containing less than 1% Suspended Solids:

8.11.1 Aqueous samples visibly absent particles are conditioned per the procedure below and extracted directly using micro-steam distillation, CLL or separatory funnel.

8.11.2 Aqueous samples containing visible particles and less than one percent suspended solids are prepared using the procedure below and extracted using CLL or the SPE technique.

8.12 Conditioning of samples with no visible particulate matter:

8.12.1 Weigh the sample container with sample and record the weight. Tare the sample container.

8.12.2 Pour the sample into the extraction vessel. Re-weigh the empty sample container and record weight.

8.12.3 For each sample batch of not more than 20 samples, place one (or two, if the customer specifies the LCS/OPR) 1.0 L aliquot(s) of reagent water in clean extraction vessel(s).

8.12.4 Spike 2 ng (multiplied by any split factor) of the ES spiking solution into the sample or QC bottle.

8.12.5 Spike 0.2 ng (nominal, multiplied by any split factor) of A_x into the BCS_3 or, if the LCS/OPR was specified by the customer.

8.12.6 Extract the samples using micro-steam distillation, CLL or separatory funnel.

8.13 Conditioning of samples with less than 1% solids containing visible particulate matter:

8.13.1 Place a precleaned 1.25 L amber bottle on the balance and tare.

8.13.2 Pour the sample into the amber bottle. Record the weight.

8.13.3 Spike 2 ng (multiplied by any split factor) of the ES spiking solution into the sample or QC bottle.

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8.13.4 Spike 0.2 ng (nominal, multiplied by any split factor) of A_x into the BCS or, if the LCS/OPR was specified by the customer.

8.13.5 Add approximately 0.1 g of CarboPak to each sample. Alternatively, carbon pellets (approximately 8 to12 pellets) can be used on samples that are for 2,3,7,8 TCDD/F only.

8.13.6 Shake the sample for two hours.

8.13.7 Assemble a Buchner funnel on top of a clean filtration flask. Begin with a large pore (1 µm) filter. Apply vacuum to the flask, and pour the entire contents of the sample bottle through a glass-fiber filter in the Buchner funnel, swirling the sample remaining in the bottle to suspend any particles.

8.13.8 Remove the filter and place in a clean thimble. Add glass wool to the Buchner funnel and wet with reagent water. Wipe the funnel and add the glass wool to the thimble.

8.13.9 Repeat steps 8.13.7 and 8.13.8 with a 0.7 µm and a 0.45 µm filter.

8.13.10 Rinse the amber bottle with approximately 5 mL of toluene and add to the thimble.

8.13.11 Extract the filters containing the particles using the SDS procedure. Alternatively, the unfiltered sample can be extracted via CLL.

8.14 Conditioning of Samples Containing Greater Than 1% Solids:

8.14.1 Add approximately 3 to 5 g of Hydromatrix™ to a clean thimble.

8.14.2 Weigh a well-mixed aliquot of each sample (of the same matrix type) sufficient to provide 10 g of dry solids (based on the solids determination in section of this SOP) into the thimble.

8.14.3 For each sample or sample batch of not more than 20 samples, weigh one, or two (if the LCS/OPR was specified by the customer) aliquot(s) of the appropriate reference matrix of approximately the largest amount used for submitted samples in the batch into clean thimble(s). One aliquot will serve as the method blank, and the second will serve as the LCS/OPR, if required.

8.14.4 Add 2 Pasteur pipets of 5% sulfuric acid to the sample. Homogenize the sample as best as possible by stirring or other suitable means.

8.14.5 Spike 2 ng (multiplied by any split factor) of the ES spiking solution into the sample.

8.14.6 Spike 0.2 ng (multiplied by any split factor) of the A_x spiking solution into the BCS or, LCS/OPR.

8.14.7 Mix the sample thoroughly. Add additional Hydromartix™ if needed.

8.14.8 Extract the sample and QC aliquots using the SDS procedure.

8.15 Conditioning of Multiphase Samples:

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8.15.1 Using the percent solids determined, determine the volume of sample that will provide 10 g of solids, up to 1 L of sample.

8.15.2 Spike 2 ng (multiplied by any split factor) of the ES spiking solution into the sample.

8.15.3 Filter the determined amount of sample through Whatman GF/D glass-fiber filter paper. Pressure filter the blank and OPR aliquots through GF/D papers also. If necessary to separate the phases and/or settle the solids, centrifuge these aliquots prior to filtration.

8.15.4 Discard any aqueous phase (if present). Remove any non-aqueous liquid present and reserve the maximum amount filtered from the sample or 10 g, whichever is less, for combination with the solid phase.

8.15.5 Extract the particles and filter in the sample and QC aliquots directly using the SDS procedure.

8.16 Homogenization - Homogenize and/or blend samples, blanks, and, when specified by the customer, OPR aliquots as best as possible.

8.16.1 Samples containing a mix of water and solids are particularly difficult to homogenize, especially when particle sizes range significantly or water exceeds 40% of the sample weight. Stirring thoroughly or continuously can aid in the homogenization.

8.17 Conditioning of Fish and Other Tissues - Prior to processing tissue samples, the laboratory must determine the exact tissue to be analyzed. Common requests for analysis of fish tissue include whole fish-skin on, whole fish-skin removed, edible fish fillets (filleted in the field or by the laboratory), specific organs, and other portions. Once the appropriate tissue has been determined, the sample must be homogenized.

8.18 Homogenization of Tissue:

8.18.1 If the laboratory must dissect the whole fish to obtain the appropriate tissue for analysis, the unused tissues may be rapidly refrozen and stored in a clean glass jar for subsequent use.

8.18.2 Each analysis requires 5 to 25 g of tissue (wet weight), depending on the client's need. Therefore, the laboratory should homogenize at least 100 g of tissue (if available) to allow for re-extraction. One 5 to 25 g portion should be used for the initial extraction. Two additional aliquots should be frozen at <10°C in case of re-extraction.

8.18.3 Homogenize the sample in a tissue homogenizer or grind in a meat grinder. Cut tissue too large to feed into the grinder into smaller pieces. The final homogenization should be carried out with a hand blender to achieve a paste-like consistency.

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8.18.4 Transfer approximately 5 to 25 g (wet weight) of homogenized tissue to a clean, tared, thimble containing approximately 3 to 5 g of Hydromatrix™. Record the weight to the nearest 10 mg.

8.18.5 Weigh two additional aliquots into clean and tared jars (with lids) and record weight. Freeze immediately.

8.19 QC aliquots

8.19.1 For each sample batch of not more than 20 samples, weigh one (or two if the LCS/OPR was specified by the customer) aliquot(s) of the appropriate reference matrix of approximately the largest amount used for submitted samples in the batch into (a) thimble(s). One aliquot will serve as the method blank, and the second will serve as the LCS/OPR, if its use was specified by the customer.

8.19.2 Spike 2 ng (multiplied by any split factor) of the ES spiking solution into the samples, blank, and BCS or LCS/OPR.

8.19.3 Spike 0.2 ng (multiplied by any split factor) of the A_x spiking solution into the BCS or, if specified by the customer, the LCS/OPR.

8.19.4 Extract the aliquots using the procedures described in this SOP.

8.20 Stack Emissions/Flue Gas Samples:

8.20.1 Concentrate the acetone and/or methylene chloride rinses to approximately five mL in a 500 mL round bottom. Add the toluene rinse to the round bottom. If needed, add additional 'fresh' toluene to the round bottom to reach volume required for extraction.

8.20.2 For Method 0023A samples, the front half and back half fraction will be extracted, analyzed and reported as separated samples. FH = Filter and FH rinses, BH = XAD trap contents and BH rinses.

8.20.3 Open the container holding the filter (usually a glass petri dish). Transfer the filter with care, especially when it is wet, to the inner side of the glass, Soxhlet thimble, with the side of the filter having any particulate matter facing the inside of the thimble.

8.20.4 If the customer specified EPA Method 0023A analysis, the filter may be placed at the bottom of the thimble, crumpled up.

8.20.5 Unpack the XAD-2 resin from the trap into the same thimble the filter is in (unless the customer specified analysis by EPA Method 0023A). Using forced air from a bulb, rinse with the trap with methanol/toluene directly into the thimble.

8.20.6 Spike 2 ng (multiplied by any split factor) of the ES spiking solution atop the XAD-2 resin, the blank's XAD-2 resin, and the BCS, or, if specified by the customer, the LCS/OPR XAD-2 resin.

8.20.7 Extract the sample(s) using Soxhlet Dean-Stark extraction technique.

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8.21 Ambient Air Samples:

8.21.1 If a filter is submitted as part of the sample, open the container holding the filter (usually a glass petri dish). Transfer the filter with care, especially when it is wet, to the inner side of the Soxhlet thimble, with the side of the filter having any particulate matter facing the inside of the thimble.

8.21.2 Transfer the PUF plug into the same thimble the filter is in using forceps.

8.21.3 Spike 2 ng (multiplied by any split factor) of the ES spiking solution atop the PUF plug, the blank's PUF plug, and the BCS, or, if specified by the customer, the LCS/OPR PUF.

8.21.4 Extract the sample(s) using Soxhlet Dean-Stark extraction technique.

8.22 Extraction, Concentration, Solvent Exchange (Sections 8.25 thru 8.33):

8.23 Separatory funnel extraction:

8.23.1 Weigh the sample container with sample and record the weight. Tare the balance. Pour the sample or filtrate into a 2 L separatory funnel. Reweigh empty sample container and record weight.

8.23.2 Spike the sample with appropriate standards.

8.23.3 Add 30 mL methylene chloride to the separatory funnel and shake for 60 seconds, venting periodically. Alternatively, the same procedure can be done using hexane.

8.23.4 Allow the organic layer to separate from the aqueous phase for several minutes.

8.23.5 If an emulsion forms and is more than one-third the volume of the solvent layer, employ mechanical techniques to complete the phase separation (see note below).

8.23.6 Drain the methylene chloride extract through an anhydrous sodium sulfate plug.

8.23.7 Repeat the steps in sections 8.23.3 to 8.23.6 twice. After the third extraction, rinse the sodium sulfate plug twice with 20 mL of methylene chloride.

8.23.8 Concentrate the extract using one of the macro-concentration procedures.

- NOTE: If an emulsion forms, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, use of phase separation paper, centrifugation, use of an ultrasonic bath with ice, addition of NaCl, or other physical methods. Alternatively, solid-phase or other extraction techniques may be used to prevent emulsion formation. Any alternative technique is acceptable so long as the QC requirements of this SOP are met.

8.24 Micro-steam distillation extraction of aqueous samples:

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8.24.1 Weigh the sample container with sample and tare, record weight. Pour each sample into a 2 L or 3 L round-bottom flask. Reweigh empty sample container and record weight.

8.24.2 Add approximately 4 mL of reagent water to the micro-steam distillation apparatus.

8.24.3 Add approximately 4 mL of isooctane to the micro-steam distillation apparatus.

8.24.4 Turn on the heating mantle and bring the water to a boil. The condensers should be cold and running at all times.

8.24.5 Allow the water to boil for two hours, then turn off the heating mantle.

8.24.6 Decant the isooctane into a labeled vial. Rinse the micro-steam distillation apparatus twice with approximately 5mL of hexane and add to labeled vial.

8.24.7 Cleanup the extract using one of the techniques in Sections 8.35 thru 8.38.

8.25 SDS Extraction of Solids:

8.25.1 Add 3 to 5 g of Hydromatrix™ to a clean thimble.

8.25.2 Place the wet sample, filter, and any non-aqueous liquid set aside into the thimble, spike as needed, and manually mix it with Hydromatrix™ with a clean metal spatula, carefully breaking up any large lumps of sample, until a free-flowing solid is created.

8.25.3 Transfer the extraction thimble to a clean thimble holder and attach to a 500 mL round bottom flask containing approximately 400 mL of toluene.

8.25.4 Place a Dean Stark apparatus on the thimble holder and connect the condenser. Turn on the heating mantle. Set the controller box to 60 to 80.

8.25.5 Once the toluene has begun to boil, observe the apparatus to ensure that three or four cycles of evaporation and siphoning are taking place each hour. Remove copious amounts of water found in the Dean Stark arm and discard it at about one hour and as needed thereafter. Frequently check the apparatus for foaming during the first two hours of extraction. If foaming occurs, reduce the reflux rate until foaming subsides. Check for leaks and replace any cracked or leaking glassware.

8.25.6 Continue the extraction for a total of at least four hours (longer extraction times may be used at the lab's convenience).

8.25.7 After extraction is complete, reduce the volume of toluene in the system to about 30 to 50 mL by draining solvent from the Dean-Stark arm and/or thimble holder. Turn off the heating mantle and allow the system to cool.

8.25.8 Remove the thimble holder from the round-bottom flask.

8.25.9 Concentrate the extract using one of the macro-concentration procedures.

8.26 SDS Extraction of Tissue:

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8.26.1 Proceed as in Section 8.27; however, optionally, before adding the tissue and Hydromatrix™ to the thimble, place approximately 50g of acid-impregnated silica in the bottom of the thimble. If acid silica is not used, and acid partition step may be necessary after extraction.

8.26.2 NOTE: This procedure should not be used if co-extracting other analytes. In such cases, use the procedure given in Section 8.28 as given.

8.26.3 Concentrate the extract using one of the macro-concentration procedures.

8.27 Percent lipid determination:

8.27.1 Percent lipids determinations in tissue are highly technique-dependent. We are aware of no rational method that can yield method-independent percent lipids results. Thus, consistency is the goal more than accuracy. The solvent system used for lipids determination will dictate the consistency of the results.

8.27.2 Add 5g of homogenized tissue to a 40 mL VOA vial.

8.27.3 Add 25 mL of methylene chloride to the VOA vial and shake vigorously for 60 seconds. Let sit overnight.

8.27.4 Decant the methylene chloride over a sodium sulfate plug into a 4-oz. jar.

8.27.5 Add 20 mL of a 50/50 mix of acetone and methylene chloride. Shake vigorously for 60 seconds. Decant liquid over the sodium sulfate plug. Repeat two more times. Rinse sodium sulfate plug with 20 mL of the 50/50 mix.

8.27.6 Place jar in a hood and allow the extract to dry for 1 to 2 days.

8.27.7 Weigh a weigh boat on an analytical balance and record and record the weight. It is not necessary to determine the lipid content of the blank, IPR, or LCS/OPR aliquots.

8.27.8 Quantitatively transfer extract from the jar to the weigh boat and allow to dry in a hood for several hours. Weigh the weigh boat containing the extract on an analytical balance and record the weight.

8.27.9 Calculate the lipid content to three significant figures as follows:

$$\%lipids = \frac{W_L - W_B}{W_S} \times 100\%$$

Where:
W_L is the weight of the lipids plus the weight boat in which it was determined
W_B is the weight of the weigh boat in which the weight of the lipids was determined
W_S is the weight of the sample extracted

8.28 Acid and/or Base Partitioning:

8.28.1 Especially oily extracts, or those with very high lipid content, may require treatment with acid or base before moving on to other cleanup techniques. Alternatively, the extracts may be cleaned up using gel permeation chromatography (GPC), which is especially useful in the case of extracts that

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remain oily after acid/base partitioning. Some extractions of chemical materials may consist only of acid/base partitioning.

8.28.2 NOTE: If PAHs are also to be determined, no acid or base partitioning can be done, as these processes will destroy the PAHs.

8.28.3 If not already done, spike 2 ng (multiplied by any split factor) of the AS and CS spiking solutions into the sample extracts, blank, and LCS/OPR (if specified by the customer) in a separatory funnel or other suitable glassware, unless the sample is a stack emissions or ambient air sample.

8.28.4 For EPA Method 23, 0023A or TO-9A analyses, spike only AS (no CS).

8.28.5 If performing base partitioning, partition the extract against 30 mL of potassium hydroxide solution. Shake for two minutes with periodic venting into a hood. Remove and discard the aqueous layer. Repeat the base washing until no color is visible in the aqueous layer, to a maximum of four washings. Minimize contact time between the extract and the base to prevent degradation of the PCDD/Fs. Partition the extract against 50 mL of sodium chloride solution. Discard the aqueous layer.

8.28.6 If performing acid partitioning, partition the extract against approximately 10 mL of sulfuric acid. Shake for two minutes with periodic venting into a hood. Allow the layers to separate. If an emulsion forms, CAREFULLY add small amounts of reagent water until two distinct layers form.

8.28.7 Transfer the organic layer to a new vessel. Repeat the acid washing until the sample is clean enough to run through the normal clean up columns, to a maximum of four washings. Minimize contact time between the extract and the acid to prevent degradation of the PCDD/Fs. Note: samples containing extremely large amounts of oils or lipids may require being partitioned with much greater quantities of acid.

8.28.8 Pour each extract through an anhydrous sodium sulfate plug into a round bottom flask. Re-concentrate the sample and QC aliquots and clean up the samples and QC aliquots using procedures described in this SOP.

8.29 Macro-Concentration-Extracts in toluene are concentrated using a rotary evaporator or a heating mantle; extracts in methylene chloride or hexane are concentrated using a rotary evaporator, heating mantle, or Kuderna-Danish apparatus. Alternatively, extracts can be allowed to evaporate slowly in a laboratory hood, when concerns about more volatile components are significant

8.29.1 Rotary evaporation-Concentrate the extracts in separate round-bottom flasks.

8.29.2 Assemble the rotary evaporator according to manufacturer's instructions, and warm the water bath to 45°C. On a daily basis, clean the condenser by rinsing with copious amounts of acetone and toluene. Between samples, rinse the roto-vap shaft with several mL of acetone.

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8.29.3 Attach a roto-vap trap and then the round-bottom flask containing the sample extract to the rotary evaporator. Slowly apply vacuum to the system, and begin rotating the sample flask.

8.29.4 Lower the flask into the water bath, and adjust the speed of rotation and the temperature as required to complete concentration. At the proper rate of concentration, the flow of solvent into the receiving flask will be steady, but no bumping or visible boiling of the extract will occur.

8.29.5 NOTE: If the rate of concentration is too fast, analyte loss may occur.

8.29.6 When the liquid in the concentration flask has reached an apparent volume of approximately 2 mL (about quarter size), turn off the vacuum, remove the flask from the water bath and stop the rotation. Slowly and carefully admit air into the system. Be sure not to open the valve so quickly that the sample is blown out of the flask. Solvent exchange if needed.

8.29.7 Proceed to Section 8.33 for preparation for back-extraction or micro-concentration and solvent exchange.

8.30 Heating mantle-Concentrate the extracts in separate round-bottom flasks:

8.30.1 Add one or two clean boiling chips to the round-bottom flask, and attach a three-ball macro Snyder column. Prewet the column by adding approximately 1 mL of solvent through the top. Place the round-bottom flask in a heating mantle, and apply heat as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood.

8.30.2 When the liquid has reached an apparent volume of approximately 10 mL, remove the round-bottom flask from the heating mantle and allow the solvent to drain and cool for at least 10 minutes.

8.30.3 Remove the Snyder column and rinse the glass joint into the receiver with small portions of solvent.

8.30.4 Proceed to Section 8.33 for preparation for back-extraction or micro-concentration and solvent exchange.

8.31 Kuderna-Danish (K-D) Concentration:

8.31.1 Concentrate the extracts in separate 500 mL K-D flasks equipped with 10 mL concentrator tubes. The K-D technique is used for solvents such as methylene chloride and hexane. Toluene is difficult to concentrate using the K-D technique unless a water bath fed by a steam generator is used.

8.31.2 Add one to two clean boiling chips to the receiver. Attach a three-ball macro Snyder column. Prewet the column by adding approximately 1 mL of solvent through the top. Place the K-D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam.

8.31.3 Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-20 minutes. At the proper rate of

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distillation, the balls of the column will actively chatter but the chambers will not flood.

8.31.4 When the liquid has reached an apparent volume of 1 mL, remove the K-D apparatus from the bath and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of solvent. A 5 mL syringe is recommended for this operation.

8.31.5 Remove the three-ball Snyder column, add a fresh boiling chip, and attach a two-ball micro Snyder column to the concentrator tube. Prewet the column by adding approximately 0.5 mL of solvent through the top. Place the apparatus in the hot water bath.

8.31.6 Adjust the vertical position and the water temperature as required to complete the concentration in 5 to10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood.

8.31.7 When the liquid reaches an apparent volume of 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes.

8.31.8 Proceed to Section 8.33 for preparation for back-extraction or micro-concentration and solvent exchange.

8.32 Preparation for back-extraction or micro-concentration and solvent exchange:

8.32.1 For back-extraction, transfer the extract to a 250 mL separatory funnel. Rinse the concentration vessel with small portions of hexane, adjust the hexane volume in the separatory funnel to 10 to 20 mL, and proceed to back-extraction.

8.32.2 For determination of the weight of residue in the extract, or for clean-up procedures other than back-extraction, transfer the extract to a blowdown vial using two to three rinses of solvent. Proceed with micro-concentration and solvent exchange in Section 8.33.

8.33 Micro-Concentration and Solvent Exchange:

8.33.1 Extracts to be subjected to GPC or HPLC cleanup are exchanged into methylene chloride. Extracts to be cleaned up using silica gel, alumina, carbon, and/or Florisil are solvent exchanged into hexane.

8.33.2 Transfer the vial containing the sample extract to a nitrogen blowdown device (e.g. Turbovap) or a rotary evaporator. If using a nitrogen blowdown device, adjust the flow of nitrogen so that the surface of the solvent is just visibly disturbed. NOTE: A large vortex in the solvent may cause analyte loss.

8.33.3 Lower the vial or round bottom into a 45°C water bath and continue concentrating. If the extract is to be concentrated to dryness for weight determination, blow dry until a constant weight is obtained.

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8.33.4 If the extract is to be concentrated for injection into the GC/MS or the solvent is to be exchanged for extract cleanup, proceed as follows:

8.33.5 When the volume of the liquid is approximately 3 mL, add 30 mL of the desired solvent (methylene chloride for GPC and HPLC, or hexane for the other cleanups) and continue concentration to approximately 3mL. Repeat the addition of solvent and concentrate once more.

8.33.6 If the extract is to be cleaned up by GPC, adjust the volume of the extract to 6.0 mL with methylene chloride. If the extract is to be cleaned up by HPLC, further concentrate the extract to 5mL. Proceed with GPC or HPLC cleanup.

8.33.7 If the extract is to be cleaned up by column chromatography (alumina, silica gel, CarboPak/Celite, or Florisil), bring the final volume to 3.0 mL with hexane. Proceed with column cleanups.

8.33.8 If the extract is to be concentrated for injection into the GC/MS, spike 2 ng of the JS to the sample extract. Quantitatively transfer the extract to a properly labeled 1.5 mL GC/MS vial for final concentration, rinsing the larger vial with tetradecane twice and adding the rinses to the GC/MS vial. Reduce the volume to 20 µL of tetradecane.

8.33.9 Seal the vial. Store in the dark at room temperature until ready for GC/MS analysis. If GC/MS analysis will not be performed on the same day, store the vial at <7°C.

8.34 Extract Cleanup:

8.34.1 Acid-Base Silica Cleanup Column

- Build the acid/base column in the following order:
  - 1 g silica gel
  - 2 g base silica
  - 1 g silica gel
  - 15 g acid silica
  - 1 g silica gel
  - 2 g 5% silver nitrate/silica
  - 1 g sodium sulfate

8.34.2 Prepare an 8% DCM/Hexane mixture. The volume should be at least 50 mL times the number of samples (including QC samples) to be cleaned up.

8.34.3 Pre-elute (condition) the columns with 90 mL of hexane.

8.34.4 Review paperwork for projects to be cleaned up.

8.34.5 Verify type of projects for the run(s) and double check for any special instructions and confirm that all samples for clean-up have been spiked and are ready for cleanup procedure.

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8.34.6 Prep round bottoms for dioxin Fast Turns and four ounce jars for normal dioxin turn-around times.

8.34.7 Prep dioxin jars or round bottoms with 20 µL of tetradecane unless otherwise stated in the project instructions.

8.35 Florisil columns:

8.35.1 Load each small column with 2.00 g (+/- .04) florisil; these columns can be prepared during the last 12 to 15 minutes of the pre-elution step for greater efficiency.

8.35.2 Florisil columns should only be prepped and conditioned immediately prior to use. Florisil is sensitive to water and moisture in the atmosphere can deactivate it.

8.35.3 To condition the Florisil columns, pre-elute them with 20 mL of dichloromethane, followed by 20 mL of hexane.

8.35.4 Connect the acid/base and Florisil columns together (acid/base column on top).

8.35.5 Quantitatively transfer each sample onto a column.

8.35.6 Perform the first elution (Fraction "A") with 85 mL of hexane. Collect this elution in a waste jar.

8.35.7 Disconnect the acid/base and florisil columns. Set the acid/base columns aside.

8.35.8 Elute the florisil column with 20 mL of 8% DCM/hexane. Collect this elution (Fraction "B") in the same container as Fraction A.

8.35.9 Change the container beneath each column to the container that will be used for the PCDD/Fs. Elute the florisil column with 100 mL of dichloromethane. This elution is Fraction C.

8.36 Carbon Cleanup

- Plug one end of a glass tube with glass wool.
- Add 1 g of Na_2SO_4
- Add 0.25 g of 5 % AX-21 carbon on silica gel
- Rinse with toluene.
- Add 1 g of Na_2SO_4
- Rinse with toluene
- Plug the other end with glass wool and pack with a glass stir rod.
- Rinse with toluene
- Spike collection vessels with 20 µL of tetradecane.

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8.36.1 Set up waste jars under columns.

8.36.2 Pre-elute the column with 10 mL of hexane, making sure the 'clean' side of the glass wool is on top (clean in, clean out)

8.36.3 Load the sample and rinse with hexane.

8.36.4 Elute with 10 mL of hexane and allow to flow into waste.

8.36.5 Flip the column over and place a collection vessel under the column. (Clean side should be DOWN)

8.36.6 Elute with 50 mL of toluene into collection vessel. (If the column is inadvertently not flipped over before the toluene elution, flip it over and elute with 150 mL toluene).

8.37 Mini-acid silica cleanup:

8.37.1 Place a glass wool plug in a 10mL drying column or a long glass Pasteur pipette.

8.37.2 Add 2 g of acid-coated silica gel to the column (4 g may also be used) followed by 1 g of Na_2SO_4.

8.37.3 Position a waste collection vial under silica column.

8.37.4 Pre-elute the column with 20 mL of hexane.

8.37.5 Place a sample collection container beneath the column.

8.37.6 Quantitatively transfer the sample to the column.

8.37.7 Elute the column with 30 mL of hexane into sample collection vessel.

9.0 Quality Control and Preventative Maintenance:

9.1 The accuracy, precision, and integrity of the sample analysis is entirely dictated by successful spiking. Thus, refer to Section 8 for details on proper spiking and SOP EU045 to understand more about the batch control spike, which informs the laboratory about the spiking event. Refer to SOPs EU051 and EU045 for more details about Quality assurance, method deviation and performance based measurement system

9.2 The analysis for PCDDs/PCDFs in water samples is for much lower concentrations than in soil/sediment, fly ash, or chemical waste samples. Extreme care must be taken to prevent cross-contamination between soil/sediment, fly ash, chemical waste and water samples. Therefore, it is strongly recommended that separate glassware be reserved for analyzing water samples.

9.3 Glassware cleaning: A hot water wash with an appropriate detergent followed by tap water rinses before a final rinse with deionized water and a 4 hour stay inside a muffle furnace maintained at 400 °C.
NOTE: This organic solvent-less technique has been proven effective for the past 17 years and offers laboratory backgrounds that are the envy of the industry.

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9.4 It is recommended (but not required) that each piece of reusable glassware be numbered in such a fashion that the laboratory can associate all reusable with the processing of a particular sample. This will assist the laboratory in:

9.4.1 Tracking down possible sources of contamination for individual samples.

9.4.2 Identifying glassware associated with highly contaminated samples that may require extra cleaning.

9.4.3 Determining when glassware should be discarded.

10.0 Pollution Prevention and Waste Management:

10.1 Waste must be disposed of in the appropriate laboratory waste streams. Refer to SOP EU023 for Disposal of Laboratory Waste.

10.2 The solvents used in this method pose little threat to the environment when managed properly. The solvent evaporation techniques used in this method are amenable to solvent recovery, and it is recommended that the laboratory recover solvents wherever feasible.

10.3 Standards should be prepared in volumes consistent with laboratory use to minimize disposal of standards.

10.4 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance is also required with any sewage discharge permits and regulations.

10.5 PCDD/Fs decompose above 800°C. Low-level waste such as absorbent paper, tissues, animal remains, and plastic gloves may be burned in an appropriate incinerator. Gross quantities (milligrams) should be packaged securely and disposed of through commercial or governmental channels that are capable of handling extremely toxic wastes.

11.0 References:

11.1 Method 23 – Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources

11.2 Method 0023A – Sampling Method for Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources

11.3 Compendium Method TO-9A - Determination of Polychlorinated, Polybrominated and Brominated/Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in Ambient Air

11.4 Method 1613, Revision B, 1994: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS

11.5 Y. Tondeur and W. F. Beckert, "Method 8290: Analytical Procedures and Quality Assurance for Multimedia Analysis of Polychlorinated Dibenzo-p-Dioxins and Dibenzofurans by High-Resolution Gas Chromatography/High-Resolution Mass

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Spectrometry," U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Las Vegas, NV

12.0 Tables, Diagrams, and Flowcharts:

12.1 N/A

Revision History:			
Revision #:	Date:	Author:	Comments:
0	01/03/2017	R. Appelle	New SOP

TITLE: Conduct of Reference Toxicant Assays

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Prepared By	Date
1	Update and Review	K. A. Simon	07/1999
2	Update & Review	L. Hauthaway	10/1999
3	Review and Update	S. Dionne	06/2000
4	Review and Update	K. A. Simon	06/2006
5	Review and Update	R. A. McIsaac	12/2011
6	Review and Minor Edits	R. A. McIsaac	04/2013
7	Review and Update	L. Bordonaro	05/2016

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1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) provides guidance for conductance and use of data from reference toxicant assays. Reference toxicant assays are conducted to evaluate the validity of test organisms through the use of a known toxicant. They are run in association with assays conducted in the laboratory and follow the procedures, endpoints, and regulations of the associated standard assay.

2.0 Definitions

Reference Toxicant: A known compound used as a toxicant to determine the relative health of test organisms used in bioassays.

3.0 Applicable Documents and References

US EPA. 2002. *Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms*. Fourth Edition. EPA-821-R-02-013.

US EPA. 2002. *Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms*. Third Edition. EPA-821-R-02-014.

US EPA. 2002. *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*. Fifth Edition. EPA-821-R-02-012.

4.0 Materials and Apparatus

CETIS™ software
Appropriate test chambers and support equipment
Appropriate volumetric glassware
Electronic balance, 0.01 mg capacity
Laboratory water
Milli-Q® water
Reference toxicant material
Salts appropriate for method used
Test organisms of appropriate age

5.0 Methods and Procedures

Reference toxicant assays will be conducted on a monthly basis for all species for which assays are routinely run. For species that are not routinely tested on, reference toxicant assays will be conducted concurrent with a given assay.

5.1 Conduct for NPDES WET Programs

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For NPDES WET programs, reference toxicant study protocol used for each species and exposure period will be that currently in use in the laboratory. Reference toxicant assays will be conducted following all specified requirements for the conduct of the assay including general water quality measurements. Water qualities will only be measured on the laboratory control and highest surviving toxicant concentration.

5.2 Conduct for Soil and Sediment Assays

Reference toxicant studies for soil and sediment assays will be conducted following protocols identified by the specific test method. In general, reference toxicant assays for organisms used in soil and sediment assays will be conducted with the reference toxicant dissolved in water and the tests will be conducted in a liquid phase.

5.3 Conduct for Species Cultured at ESI

Reference toxicant assays for species cultured at ESI will be conducted on a regular basis. Reference toxicant evaluations for acute and chronic exposure assays will be conducted on a monthly basis. In cases where species/tests are conducted on an infrequent basis (an average of less than once a month), a reference toxicant assay will be conducted with each assay.

5.4 Stock solution preparation and mixing instructions may be found on the corresponding assay-specific reference toxicant bench sheets located in the following directory: P:\General Projects\FORMS\LABFORMS\Reference Toxicant Assay.

6.0 Quality Control Requirements

6.1 All acceptability criteria established for a particular assay shall apply to reference toxicant assays.

6.2 In cases where an assay fails to meet minimum acceptability criteria, the assay shall be repeated.

6.3 Acceptability

6.3.1 If the measured endpoint falls within ±2 standard deviations of the historic mean for the matrix, then the results of the assay shall be considered as acceptable.

6.3.2 If the endpoint falls outside ±2 standard deviations of the historic mean, then the results indicate that the test organism or procedures are suspect. In any such case the data will be reviewed in an attempt to determine the source of the variance. After the review the assay will be repeated.

7.0 Calculations and Reporting

7.1 Reference toxicant concentrations shall be reported as nominal values.

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7.2 Statistical analyses of assays will be conducted using CETIS™, Comprehensive Environmental Toxicity Information System, software. Data from the assays will be entered into an appropriate control chart. Control charts will document long-term mean value, and ±1 and ±2 standard deviations of the mean limits. Control charts will be updated on a regular basis. Results of reference toxicant tests for the appropriate species/test will be included in all final reports. At a minimum, reference toxicant data provided in a report will include reference toxicant, current value, number of tests conducted, historic mean, and ±2 standard deviations limits.

8.0 Corrective Actions

In cases where the measured endpoint for a species/test falls outside ±2 standard deviations of the historic mean for the species/test, the assay will be repeated. In addition, the Laboratory Manager will be notified immediately as to the failure of the reference toxicant assay. The Laboratory Manager will review the data from the reference toxicant test and determine, if possible, the reason for the failure. If the reason for the failure can be traced to a particular problem or event that does not impact the outcome of an assay in general, then other tests in progress at the time will be allowed to be completed and will be considered as acceptable. If the reason for the failure appears to be related to the overall health of test organisms or a problem that is considered to be related to overall laboratory performance that is likely to impact other tests, then the results of all tests will be inspected and a determination made as to their acceptability.

9.0 Health and Safety

As with any chemical compound, gloves and safety glasses must be worn when handling and preparing reference toxicant solutions. All safety precautions normally associated with the conduct of an assay and preservation of samples will be followed. Anyone performing these tests must be trained in the handling of those materials and the performance of those techniques. Before performing any procedure, the technician must be familiar with the Material Safety Data Sheet (MSDS) for all compounds and chemicals involved in the testing

10.0 Responsibilities

It is the Laboratory Manager's responsibility to ensure that reference toxicant evaluations are carried out as required by this SOP. It is also the Laboratory Manager's responsibility to schedule testing and to ensure results of the testing are entered into the appropriate control charts and made available for inclusion into reports.

11.0 Waste Management and Pollution Prevention

11.1 The solutions and reagents used in this method pose little threat to the environment when recycled and managed properly.

11.2 Solutions and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired materials to be disposed.

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11.3 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the biohazard and hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from bench operations. Compliance with all sewage discharge permits and regulations is also required.

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TITLE: Acute Toxicity of Sediments to the mysid shrimp, *Americamysis bahia* - New York District, Region II Testing

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	SOP Preparation	C. Sasso	01/2018

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TITLE: Acute Toxicity of Sediments to the mysid shrimp, *Americamysis bahia* - New York District, Region II Testing

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes methods for assessing the toxicity potential of marine and estuarine sediments to the mysid, *Americamysis bahia*. The assay is conducted using guidelines developed by ASTM, the US EPA and the US ACOE. Protocols are included for work in New York District, Region II.

The 10-day assay involves exposing organisms to solid phase sediment samples and overlying water in aerated 1 L glass test chambers in static mode. Test chambers are monitored on a daily basis for overlying water quality. At the end of the exposure period, mysids are recovered from the test vessels and counted. Survival data is used to determine toxicity potential. Natural marine sediment is used as the negative control in the test. The negative control provides a measure of the acceptability of the test by providing evidence of the health and relative quality of the test organisms, the suitability of the overlying water, test conditions and handling procedures. Typically a reference sediment is used for interpreting data obtained from the test sediments.

2.0 Definitions

Alternating Allocation: Addition to test chambers is ordered so that chambers are loaded in replicate order (replicate A, followed by replicate B, followed by replicate C, etc.). Within replicate groups the order is alternated between low to high and high to low sample numbers.

Overlying Water: The water placed over sediment in a test chamber during a test.

Reference Sediment: A whole sediment near an area of concern used to assess sediment conditions exclusive of material(s) of interest. The reference sediment may be used as an indicator of localized sediment conditions exclusive of the specific pollutant input of concern. Such sediment would be collected near the site of concern and would represent the background conditions resulting from any localized pollutant inputs as well as global pollutant input.

Reference Toxicity Test: A test conducted with reagent-grade reference chemical to assess the sensitivity of the test organisms. Deviations outside an established normal range may indicate a change in the sensitivity of the test organism population. Reference toxicity tests are most often are performed in the absence of sediment.

Pore Water: Water located in spaces between grains of sediment.

Sediment: Particulate material that usually lies below water; formulated particulate material that is intended to lie below water in a test.

Whole Sediment: Sediment and associated pore water which have had minimal manipulation. The term bulk sediment has been used synonymously with whole sediment.

3.0 Applicable Documents and References

ASTM. 2016. Annual Book of ASTM Standards. Volume 11.06. *Standard Guide for Measuring the*

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Toxicity of Sediment-Associated Contaminants with Marine and Estuarine Amphipods. E-1367-03 (2008). West Conshohocken, PA.

US EPA. 1994. *Methods for Assessing the Toxicity of Sediment-associated Contaminants with Estuarine and Marine Amphipods*. EPA 600/R-94/025.

US EPA. 2002. *Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms*. Third Edition. EPA-821-R-02-014.

US EPA, US Army Corps of Engineers. 1998. *Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual*. EPA-823/B-98/002.

US EPA Region 2, US Army Corps of Engineers, New York District. 2016. *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal*.

ASTM. 2002a. Standard Guide for Conducting Acute Toxicity Test with Fishes, Macroinvertebrates, and Amphibians In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05, E729-96. Philadelphia, PA., pp 135-156.

4.0 Materials and Apparatus

A. bahia, 1 - 5 days old (24-hour range in age)
Aeration system (compressor)
Airlines
Airline holders
Artemia nauplii, <24 hours post-hatch
Beakers, 1 L drilled and screened to facilitate water exchanges
Marine laboratory sediment, field collected
Disposable transfer pipettes
Incubator or water bath, capable of maintaining 20±2°C
Ambient laboratory lighting
Timer, to maintain photoperiod
Light Table
Marine salts, to adjust salinity if necessary
Natural sea water
Pill cups
Shallow dish
Sieves,200 µm for recovery, 1-2 mm for sample preparation
Temperature logger
Wash bottles
Water bath with water exchange system
Water quality meters

5.0 Methods and Procedures

5.1 Test Material

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5.1.1 Test substances will be clearly identified as to source, collection date, period of collection and description of collection methods. The laboratory will identify sample storage procedures and maintain receipt records. Records for sample storage, usage and disposal will be generated when necessary.

5.1.2 Test material is stored at 4±2°C in the original shipping container, typically bioasssay-grade cubitainers or appropriately sized HDPE buckets lined with food-grade or other chemically clean liners. Samples may be held for up to 8 weeks from date/time of collection.

5.1.3 Any overlying water in the sample container will be assumed to be pore water that has come out of t he sediment due to settling since collection. Prior to sub-sampling the water will be mixed back into the sample.

5.2 Test Species

Organisms are obtained from a commercial source which meets ESI quality assurance standards. Confirmation of species is provided by the supplier. Each lot of organisms obtained from a commercial supplier must be taxonomically verified. A citation of the taxonomic key used and the distinguishing characteristics must be documented during verification.

5.2.1 Healthy mysids from the same source, 1 - 5 days old, with an age range within batch of ≤24 hours are used in the test. Organisms should be held similar to testing conditions, with overlying water at the required temperature and salinity that will be used in the test.

5.2.2 Organisms must be held for minimum of 48 hours prior to testing and acclimated to testing conditions gradually during holding time. Overlying water should be renewed every 24 - 48 hours during the acclimation period. Temperature and salinity changes during holding should be <3°C and <3 ppt over any 24 hour period.

5.2.2.1 Overlying water during holding is the same as water used for testing.

5.2.3 Water quality measurements (temperature, pH, salinity, and dissolved oxygen) must be measured daily during acclimation. Water quality parameters should be recorded before and after renewal of holding water.

5.2.4 Mysids must be fed daily (approximately 100 *Artemia* nauplii per mysid) during acclimation. Note that mysids should not be overfed as this could cause unacceptable water quality conditions.

5.2.5 Pretest observation data concerning the source, handling procedures, disease treatment (if any), health, feeding, mean dry weight and mortality of test animals is recorded and reported as necessary. Note that if mortality exceeds 10% during holding period or 5% over the final 24 hours of holding, it is recommended that the lot of organisms be discarded.

5.3 Study Conduct

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Exposure conditions and monitoring requirements detailed in the following paragraphs are the minimum requirements specified by ESI for this assay. Note the client should be contacted should there be any concerns with assay ammonia levels.

5.3.1 The assay is conducted as a static non-renewal exposure, unless pore water ammonia levels exceeds limits described in step 5.3.10 (as directed by the client). Any water lost through evaporation is replaced by DI water. Static renewal assays will be conducted with overlying water renewal at a rate of one volume addition daily.

5.3.2 Sediment for the laboratory control treatment is marine sediment that has been processed to remove indigenous organisms. The sediment is press sieved through a .5 mm screen and placed it in a holding chamber, designed for flow through conditions with natural seawater, until time of use.

Each batch of marine sediment should be given an "A-number" and recorded in the General Inventory Receipt Logbook. The "A-number" is also recorded in the Laboratory Control Sediment Logbook and should be entered on applicable assay records.

5.3.3 The reference site and testing sediments are press sieved through a 2 mm screen to remove indigenous organisms and debris.

5.3.3.1 Final sorting and evaluations of reference site sediment should take place on a light table. If any organisms are observed, the reference sediment will be re-sieved down through a .5mm screen.

5.3.3.2 All sediment handling procedures, including any excluded material and test sediment information (smell, texture, etc.) is noted in a "Test Sediment Preparation Notes" log included in the raw data of the final report.

5.3.4 All treatments (including sample site sediment, laboratory control sediment and reference site sediment) consist of a minimum of 5 replicates with 20 mysids per replicate. Test chambers are arranged in columns by treatment and samples will be identified by a test sample number during testing.

5.3.5 A set of 11 surrogate chemistry chambers containing test sediment and water but no organisms will be loaded for each dredged material sample tested . These are included to allow for daily monitoring of overlying ammonia during test, even if initial total ammonia is <20 ppm in the pore water.

5.3.6 Approximately 175 mL of homogenized test sediment is placed into each test chamber. Test chambers are 1 liter beakers that have been drilled and screened to facilitate water exchange, should renewal be warranted. Sediment is smoothed to a uniform depth and should be consistent among all chambers.

5.3.7 Overlying water is immediately added to each test chamber in order to avoid drying of the sample. Overlying water is added carefully to minimize sediment disturbance. The volume of the overlying water is a minimum of approximately 625 mL. Overlying water is natural seawater obtained from the Hampton-Seabrook Estuary. While

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being held, the water is filtered to remove debris and maintained at a range of 30±2‰ either by the addition of de-ionized laboratory water to lower or addition of EDTA free commercial sea salts to raise the salinity. (See section 6.7 for more information on laboratory seawater.) The filtration process provides constant aeration for the large holding tanks.

5.3.7.1 Anytime new salt water is received into the building, from the beginning of the ammonia mitigation period through assay termination, a sample will be taken and submit for total ammonia testing.

5.3.8 Chambers are then allowed to settle overnight prior to overlying water ammonia extraction.

5.3.9 On the following day, the overlying water in ammonia surrogate chemistry chambers is pulled and checked using a color-metric aquaria test kit, as well as submit for chemical analysis. The overlying water in chambers are renewed twice, at least 3 hours apart from one another, each day during the ammonia mitigation phase of the assay. All color-metric values will be recorded on associated paperwork for the assay. Ammonia levels are confirmed to be below the upper threshold prior to test initiation.

Note that overlying water renewals only take place during the ammonia mitigation period. Once pore water ammonia levels decrease to within threshold limits, all renewals cease for testing.

5.3.10 All test chambers, including surrogate chemistry chambers, will be covered throughout testing to reduce any water loss due to evaporation.

5.3.11 Aeration is supplied to all test chambers at a rate of approximately 100 bubbles per minute. The tip of the air line will be kept above the sediment's surface to avoid sediment resuspension. The aeration will be constant to provide ≥40% saturation to each testing chamber.

5.3.12 Water temperature is maintained at 20±2°C. If any instances arise causing a greater than 20±2°C, temperature change, immediate steps will take place to mitigate the situation. A corrective action plan will be implemented to document the occurrence and to ensure it does not reoccur.

5.3.13 Photoperiod is set at 16 hours light : 8 hours dark. Light is provided by ambient laboratory illumination, LED bulbs.

5.3.14 Overlying water qualities are recorded in every beaker for the duration of the assay.

5.3.14.1 Water quality measurements are taken daily on the surrogate chemistry chamber being sampled from that day.

5.3.14.2 Daily water quality parameters measured in the overlying water are dissolved oxygen, pH, salinity, and temperature.

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5.3.14.3 Temperature is also recorded on an hourly basis in a separate test vessel by a data-logger.

5.3.15 Total ammonia levels in pore water must be below 20 ppm in all treatments prior to assay initiation. Organisms are added to test chambers after ammonia levels have been confirmed to be within acceptable limits.

After test initiation, ammonia in the surrogate chambers must be monitored to evaluate the potential for ammonia interference. Overlying water un-ionized ammonia concentrations must be measured and recorded daily. The not-to-exceed toxicity threshold ammonia concentration for mysids is established as the permissible concentrations of un-ionized ammonia in test chamber overlying water and is related to the pH of the water, as described in the following table:

Average 10-Day pH	Not to Exceed Threshold (ppm)
≥8.1	0.5
7.9 - 8.0	0.4
7.7 - 7.8	0.3
≤7.6	0.2

[1] Table adapted from Table 6-2 of *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal*, US EPA, US Army Corps of Engineers, New York District Region 2, 2016.

5.3.16 Adding Animals to the Test

Mysids representative of the general population are counted with the aid of a light table into pill cups containing seawater. Ten organisms are added to each pill cup. Since *A. bahia* have a tendency to stick to pipettes, extra care is needed when allocating organisms to cups.

Organisms are then transferred from the pill cup to the test chamber in a methodical process. One cup of organisms is added to each test chambers and then the process is repeated for a total of two pill cups (20 organisms) to each vessel. When test chambers are arranged in a block randomized position order, addition of organisms will be conducted in the position order. When test chambers are not arranged in a block randomized position order, addition of organisms will either be by alternating allocation or a similar method.

After each organism addition, the pill cup is checked to ensure that all mysids were successfully transferred to the test chamber. If organisms are observed in the cup, they are rinsed down into the test chamber using saltwater. The cup is checked again, and the process is repeated until all organisms have been transferred.

5.3.17 Organisms will be fed <24 hour old brine shrimp (*Artemia* nauplii) twice daily during

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the assay (~0.2 mL/day). Obvious mortalities will be recorded within the project's raw data.

5.4 Study Termination

5.4.1 On the final day of the assay, water qualities measurements are collected from each test replicate. Overlying water ammonia samples are collected for analysis from the remaining surrogate chambers.

5.4.2 The contents of each chamber are carefully decanted and washed through 200 µm sized screen. Care must be taken to minimize the potential for sediment disturbance/resuspension. This process is repeated several times to ensure all organisms are removed.

5.4.2.1 Organisms remaining on the screen are rinsed into a clear holding vessel to be examined and enumerated using a light board. Any mysid that shows signs of movement is considered alive and recorded on bench sheet recovery records for survival statistically analysis.

5.5 Calibration and standardization are not applicable components of this method and procedure.

6.0 Quality Control Requirements

6.1 Survival acceptance criteria in the laboratory control is ≥90%. Note that if mean reference and mean test organism survivals are <90%, then assay may need to be repeated. If <90% survival, the client should be contacted immediately as the affected test(s) may have to be repeated. The client may choose to contact the New York District. Any delays in contacting the client may lead to problems meeting holding times if a retest is required.

6.2 Survival acceptance criteria in the reference sediment is ≥90%. Note that if mean reference and mean test organism survivals are <90%, then assay may need to be repeated. If <90% survival, the client should be contacted immediately as the affected test(s) may have to be repeated. The client may choose to contact the New York District. Any delays in contacting the client may lead to problems meeting holding times if a retest is required.

6.3 A reference toxicant evaluation must be successfully conducted with each batch of organisms. The reference toxicant is a 96-hour 'water only' test.

6.3.1 The reference toxicant evaluated will be ammonia.

6.3.2 If ≥90% in controls, then the reference toxicant test will be considered valid. If <90%, assay may need to be restarted. The Laboratory Manager is notified for appropriate action.

6.3.3 Results must fall between ±2 standard deviations from the historical mean.

6.3.4 For more information see SOP QA-1114 "Conduct of Reference Toxicant Assays".

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6.4 It is crucial to refer to each project's scope of work before the beginning of testing to ensure that all individual requirements are satisfied.

6.5 It is imperative that all materials (i.e. data sheets) are reviewed on a daily basis by personnel performing the assay to ensure that all applicable data is available and accurate.

6.6 For the assay to be considered valid, conditions should meet those specified in Section 11.0: "Summary of Test Conditions".

6.7 Interferences

6.7.1 Living organisms present in the sample may compete with the larvae or may be predators, reducing overall survival. This impact is mitigated by sieving samples prior to testing.

6.7.2 Ammonia has been recognized as a potential confounding factor in sediment evaluations. The ammonia mitigation phase that takes place prior to test initiation ensures total ammonia levels are below 20mg/L in all sediment pore water.

6.8 The salinity and pH of natural seawater obtained from the Hampton-Seabrook Estuary and used for laboratory salt water are monitored regularly. On a quarterly basis, water quality analyses are performed to monitor the following parameters: total mercury, total metals, pesticides, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners.

6.8.1 Saltwater is also tested annually for all parameters listed under ASTM 2002a.

6.9 Detection limit is not an applicable aspect of the quality control requirements for this method and procedure.

7.0 Calculations and Reporting

7.1 Data Analysis

Survival data from each treatment will be subjected to the one way student unpaired T-test, or another appropriate statistical test, to determine if significant differences exist between treatments and the control. Statistical evaluations will be made with CETIS™ (Comprehensive Environmental Toxicity Information System) software using appropriate standard statistical models.

7.1.1 Prior to statistical analysis, the survival data will be reviewed to determine the presence of outliers. If outliers are found, an explanation must be sought. If a reasonable source for the deviation is found, the value may be excluded from further analysis. If no explanation is found, the analysis should be performed both with and without the questionable data point and both sets of results reported. All data sets are evaluated to determine sample variance homogeneity and normality. Those data sets meeting the criteria for normality and homogeneity are evaluated with

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parametric statistical models, while those that do not meet both criteria are evaluated using non-parametric models.

7.1.2 Endpoint to be evaluated is survival.

7.1.3 Statistical comparisons for each sample site will be made against the laboratory control treatment and each project reference site or as specified. Project reference sites may also be compared to one another.

7.2 Reporting

The final report will provide the following: cover page with title, name and address of the laboratory, unique identifier of the test report, date of issue, name and address of the client; introduction, materials, methods, sample receipt information, results of test, discussion of deviations, references, summary of reference toxicant tests. Copies of all bench sheets, raw data and statistical output will be provided in an appendix included with the report. Both the report and the data appendix will be paginated.

8.0 Corrective Actions

8.1 If survival fails to meet the minimum value specified by the protocol, the client will be notified and the test may be restarted.

8.2 In the event that an element of the assay falls outside acceptable limits, the deviation must be documented and corrective actions initiated by the analyst. The laboratory manager should be notified. The laboratory manager, using sound scientific practice, will determine if the study requires repeating or if the data is allowed to be accepted.

9.0 Health and Safety

9.1 As with all samples, gloves should be worn when handling sediment samples and chemicals. It is advisable to wear a lab coat to protect clothing. Full body coveralls are also available.

9.2 Assays and sample disposal will be conducted using procedures to minimize potential pollution of surface and ground waters, soils and sediments.

9.3 ESI is a fully licensed and approved waste generator and storage facility. ESI operates in full compliance with the State of New Hampshire Department of Environmental Services solid waste guidelines. Waste storage, transfer and disposal will be in compliance with State of New Hampshire Department of Environmental Services and US EPA waste disposal regulations.

9.4 Unused samples will be maintained in a secure location until disposal is approved by the client. Once a determination has been made that samples are no longer required by the project, they will be removed from their original container and transferred to a DOT approved container for transfer to an approved waste disposal facility.

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10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedures outlined in this SOP.

10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOP's, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification will be based upon a review of the person's demonstrated abilities.

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TITLE: Acute Toxicity of Sediments to the mysid shrimp, *Americamysis bahia* - New York District, Region II Testing

11.0 Summary of Test Conditions

(Unless otherwise specified by client)

1.	Test Mode:	Static non-renewal (initiate static renewal methods if un-ionized ammonia in overlying water exceeds levels in step 5.3.15).
2.	Test Duration:	10 Days
3.	Renewal Schedule:	None (1 volume exchange daily for static renewal assays only)
4.	Temperature:	20±2°C
5.	Photoperiod:	16 hour light/ 8 hour dark
6.	Light Source:	Ambient laboratory illumination
7.	Salinity:	30±2 ppt
8.	pH:	7.8 ± 0.5 (target)
9.	Test Chamber:	1 L beakers
10.	Solution Volume:	A minimum of ~625 mL overlying water
11.	Sediment Depth:	2 cm (~175 mL) Homogenized whole-sediment
12.	Organisms/Chamber:	20
13.	Replicates/Treatment:	5 minimum for endpoint; 11 for ammonia monitoring
14.	Organisms/Treatment:	100
15.	Age of Organisms:	1 - 5 days, 24-hour range in age
16.	Feeding Regime:	~0.2 mL/day; <24 hour old brine shrimp (*Artemia* nauplii) twice daily
17.	Overlying Water:	Natural Seawater
18.	Aeration:	Continuous aeration to maintain ≥40% saturation
19.	Endpoint:	Survival
20.	Acceptability:	Mean lab control survival of ≥90%, Mean reference site survival of ≥90%
21.	Support Chemistry:	Overlying water qualities: DO, pH, salinity and temperature on all replicates daily. Overlying water ammonia daily; hourly temperature readings in one surrogate vessel. Total ammonia analysis upon any salt water received.
22.	Reference Toxicant:	Ammonia, run concurrent with testing on organisms from same batch received for assay conduct
23.	Sample Holding Time:	≤ 8 weeks, held at 4±2°C

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TITLE: Acute Toxicity of Sediments to the Marine Amphipod, *Ampelisca abdita,* based on Region II Guidelines

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Creation, based on QA-1426	J. T. Provencher	01/2018

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TITLE: Acute Toxicity of Sediments to the Marine Amphipod, *Ampelisca abdita,* based on Region II Guidelines

1.0 Purpose and Applicability

This Standard Operating Procedure describes the method used for assessing the toxicity potential of marine and estuarine sediments to the amphipod, *Ampelisca abdita*. The assay is conducted using guidelines developed by ASTM, the US EPA and the US ACOE for New York District Region II.

The 10-day assay involves exposing organisms to solid phase sediment samples and overlying water in aerated 1L glass test chambers in a static non-renewal mode. If ammonia levels dictate, the test may be run with daily overlying water renewals of test water. Test chambers are monitored on a daily basis for overlying water quality, obvious mortality, formation of tubes or burrows, amphipod emergence from sediment and any physical or behavioral abnormalities.

At the end of the exposure period, amphipods are recovered from the test vessels and counted. Survival data is used to determine toxicity potential. Natural marine sediment is used as the negative control in the test. The negative control provides a measure of the acceptability of the test by providing evidence of the health and relative quality of the test organisms, the suitability of the overlying water, test conditions and handling procedures. A reference sediment is used for interpreting data obtained from the test sediments.

2.0 Definitions

Alternating Allocation: Addition to test chambers is ordered so that chambers are loaded in replicate order. (Replicate A, followed by replicate B, followed by replicate C, etc.) Within replicate groups the order is alternated between low to high and high to low sample numbers.

Overlying Water: The water placed over sediment in a test chamber during a test.

Reference Sediment: A whole sediment near an area of concern used to assess sediment conditions exclusive of material(s) of interest. The reference sediment may be used as an indicator of localized sediment conditions exclusive of the specific pollutant input of concern. Such sediment would be collected near the site of concern and would represent the background conditions resulting from any localized pollutant inputs as well as global pollutant input. This is the manner in which reference sediment is used in dredge material evaluations.

Reference Toxicity Test: A test conducted with reagent-grade reference chemical to assess the sensitivity of the test organisms. Deviations outside an established normal range may indicate a change in the sensitivity of the test organism population. Reference toxicity tests are most often are performed in the absence of sediment.

Pore Water: Water located in spaces between grains of sediment.

Sediment: Particulate material that usually lies below water; formulated particulate material that is intended to lie below water in a test.

Whole Sediment: Sediment and associated pore water which have had minimal manipulation. The term bulk sediment has been used synonymously with whole sediment.

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TITLE: Acute Toxicity of Sediments to the Marine Amphipod, *Ampelisca abdita,* based on Region II Guidelines

3.0 Applicable Documents and References

ASTM. 2014. Annual Book of ASTM Standards. Volume 11.06. *Standard Guide for Measuring the Toxicity of Sediment-Associated Contaminants with Marine and Estuarine Amphipods*. E-1367-03 (2008). West Conshohocken, PA.

US EPA. 1994. *Methods for Assessing the Toxicity of Sediment-associated Contaminants with Estuarine and Marine Amphipods*. EPA 600/R-94/025.

US EPA. 2001. *Methods for Assessing the Chronic Toxicity of Marine and Estuarine Sediment-associated Contaminants with the Amphipod, Leptocheirus plumulosus*. March 2001. EPA 600/R-01/020.

US EPA, US Army Corps of Engineers. 1998. *Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual*. EPA-823/B-98/002.

US EPA Region 2, US Army Corps of Engineers, New York District. 2016. *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal*.

ASTM. 2002a. Standard Guide for Conducting Acute Toxicity Test with Fishes, Macroinvertebrates, and Amphibians In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05, E729-96. Philadelphia, PA., pp 135-156.

4.0 Materials and Apparatus

A. abdita; subadults, 3-5 mm, retained on 0.7 mm sieve after passing through a 1.0 mm sieve
Aeration system
Beakers, 1 L, drilled and screened to facilitate water exchanges
Marine Laboratory Control Sediment (field collected)
Disposable transfer pipettes
Light Table
Marine salts (if necessary to adjust salinity of natural ocean seawater)
Natural sea water
Pill cups
Shallow dish
Sieves
Temperature logger
Wash bottles
Water bath with water exchange system capable of maintaining 20±2°C
Water quality meters
Ambient laboratory illumination
Test chamber coverings

5.0 Methods and Procedures

5.1 Test Material

5.1.1 Test substances will be clearly identified as to source, collection date, period of collection and description of collection methods. The laboratory will identify sample storage procedures and maintain receipt records. Records for sample storage,

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usage and disposal will be generated when necessary.

5.1.2 Test material is stored at 4±2°C in the original shipping container, typically bioasssay-grade cubitainers or appropriately sized HDPE buckets lined with food-grade or other chemically clean liners. Samples may be held for up to 8 weeks from date/time of collection.

5.1.3 Any overlying water in the sample container will be assumed to be pore water that has come out of t he sediment due to settling since collection. Prior to sub-sampling the water will be mixed back into the sample.

5.2 Test Species

Organisms are obtained from a commercial source which meets ESI quality assurance standards. Confirmation of species is provided by the supplier. Each lot of organisms obtained from a commercial supplier must be taxonomically verified. A citation of the taxonomic key used and the distinguishing characteristics must be documented during verification.

5.2.1 Healthy amphipods from the same source, sub-adult, 3-5 mm total length, and retained on 0.7 mm sieve but passing through a 1.0 mm sieve, are used in the test.

5.2.2 Amphipods should be held and maintained for a minimum of 48 hours in similar water and test conditions prior to assay initiation.

5.2.2.1 Holding should occur in sieved clean sediment for 24 hours prior to use for testing

5.2.2.2 Overlying water should be renewed every 24-48 hours during acclimation. Water quality parameter should be recorded before and after renewal of holding water.

5.2.2.3 Water temperature and salinity changes during holding should be <3°C and <3 ppt over any 24 hour period.

5.2.2.4 Water qualities (temperature, pH, salinity, and dissolved oxygen) must be measured daily during acclimation.

5.2.2.5 It is recommended that organisms be fed daily during acclimation. (Note that organisms are not fed during assay conduct.) Recommended food includes *Phaeodactylum tricornutum* (approximately 2.5 x 10^5 cells/mL) or *Isochrysis* species (concentrated). Any food source, including commercially prepared sources, that the laboratory has documented success is acceptable

5.2.2.6 If mortality exceeds 10% over the entire acclimation period and holding period, or 5% over the final 24 hours of acclimation, then it is recommended that the affected lot of organisms be discarded and the acclimation be started with a new batch of organisms.

5.2.3 Pretest observation data concerning the source, handling procedures, disease

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treatment (if any), health, feeding, mean dry weight and mortality of test animals is recorded and reported as necessary. Note that if mortality exceeds 10% during holding period or 5% over the final 24 hours of holding, it is recommended that the lot of organisms be discarded.

5.3 Study Conduct

Exposure conditions and monitoring requirements detailed in the following paragraphs are the minimum requirements specified by ESI for this assay. Note that the client should be contacted should there be any concerns with ammonia levels.

5.3.1 The assay is conducted as a static non-renewal exposure, unless pore water ammonia levels dictate once daily renewals (as directed by the client). Any water lost through evaporation is replaced by DI water. Static renewal assays will conducted with overlying water renewal at a rate of one volume addition daily.

5.3.2 Sediment for the laboratory control treatment is marine sediment that has been processed through a 0.5 mm screen and then placed it in a holding chamber, designed for flow through conditions with natural seawater, until time of use.

Each batch of marine sediment should be given an “A-number” and recorded in the General Inventory Receipt Logbook. The “A-number” is also recorded in the Laboratory Control Sediment Logbook and should be entered on applicable assay records.

5.3.3 The reference site sediment is press sieved through a 1 mm screen and testing sediments are press sieved through a 2 mm screen to remove indigenous organisms and debris.

5.3.3.1 Final sorting and evaluations of reference site sediment should take place on a light table. If any organisms are observed, the reference sediment will be re-sieved down through a .5mm screen.

5.3.3.2 All sediment handling procedures, including any excluded material and test sediment information (smell, texture, etc.) is noted in a “Test Sediment Preparation Notes” log included in the raw data of the final report.

5.3.4 All treatments (including sample site sediment, laboratory control sediment and reference site sediment) consist of a minimum of 5 replicates with 20 amphipods per replicate. Test chambers are arranged in columns by treatment and samples will be identified by a test sample number during testing.

5.3.5 A set of 11 surrogate chemistry chambers, containing test sediment and water but no organisms, for each dredged material sample tested. These are included to allow for daily monitoring of pore water ammonia during test, even if initial total ammonia is <20 ppm in the pore water.

Aeration stones attached to plastic tubing are placed in the pore water ammonia surrogate chambers. The stone must be completely submerged in the sediment. By attaching a syringe onto the open end of the tubing, a vacuum is created drawing up

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pore water through the aeration stone into the tubing. This procedure takes place once for each test sediment daily until assay termination.

5.3.7 Approximately 175 mL of homogenized test sediment is placed into each test chamber. Test chambers are 1 liter beakers that have been drilled and screened to facilitate water exchange, should renewal be warranted. Sediment is smoothed to a uniform depth and should be consistent among all chambers.

5.3.8 Overlying water is immediately added to each test chamber in order to avoid drying of the sample. Overlying water is added carefully to minimize sediment disturbance. The volume of the overlying water is a minimum of approximately 625 mL. Overlying water is natural seawater obtained from the Hampton-Seabrook Estuary. While being held, the water is filtered to remove debris and maintained at a range of 30±2‰ either by the addition of de-ionized laboratory water to lower or addition of EDTA free commercial sea salts to raise the salinity. (See section 6.7 for more information on laboratory seawater.)

5.3.8.1 This assay is run using 28±2‰ natural sea water.

5.3.9 Chambers are then allowed to settle overnight prior to overlying and pore water extractions

5.3.10 On the following day, pore water ammonia in the ammonia surrogate test chambers is pulled and checked using a color-metric aquaria test kit, as well as submit for chemical analysis. The overlying water in chambers are renewed twice, at least 3 hours apart from one another, each day during the ammonia mitigation phase of the assay. All color-metric values will be recorded on associated paperwork for the assay. Ammonia levels are confirmed to be below the upper threshold prior to test initiation.

Note that overlying water renewals only take place during the ammonia mitigation period. Once pore water ammonia levels decrease to within threshold limits, all renewals cease for testing.

5.3.11 All test chambers will be covered throughout testing to reduce any water loss due to evaporation.

5.3.12 Aeration is supplied to all test chambers at a rate of approximately 100 bubbles per minute. The tip of the air line will be kept above the sediment's surface to avoid sediment resuspension. The aeration will be constant to provide ≥90% saturation to each testing chamber.

5.3.13 Water temperature is maintained at 20±2°C. If any instances arise causing a greater than 20±2°C temperature change, immediate steps will take place to mitigate the situation. A corrective action plan will be implemented to document the occurrence and to ensure it does not reoccur.

5.3.14 Photoperiod is set for continuous light. Light is provided by ambient laboratory illumination, LED bulbs.

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5.3.15 Overlying water qualities are recorded in every test chamber daily. Water qualities are also recorded on individual surrogate chemistry replicates when sampling from that replicate takes place.

Daily water quality parameters measured in the overlying water are dissolved oxygen, pH, salinity, and temperature. Temperature is also recorded on an hourly basis in a separate test vessel by a data-logger.

5.3.16 Total ammonia levels in pore water must be below 20 ppm in all treatments prior to assay initiation. Organisms are added to test chambers after ammonia levels have been confirmed to be within acceptable limits.

After test initiation, ammonia in the surrogate chemistry chambers must be monitored daily to evaluate the potential for ammonia interference. The not-to-exceed toxicity threshold ammonia concentration for amphipods is 20 ppm total ammonia in pore water. Overlying water un-ionized ammonia concentrations must be measured and recorded daily.

5.3.17 Adding Animals to the Test

Amphipods representative of the general population are counted with the aid of a light table into pill cups containing seawater. Ten organisms are added to each pill cup. Since *A. abdita* have a tendency to stick to pipettes, extra care is needed when allocating organisms to cups.

Organisms are then transferred from the pill cup to the test chamber in a methodical process. One cup of organisms is added to each test chambers and then the process is repeated for a total of two pill cups (20 organisms) to each vessel. Addition of organisms will either be by alternating allocation or a similar method. When test chambers are arranged in a block randomized position order, addition of organisms will be conducted in the position order. When test chambers are not arranged in a block randomized position order, addition of organisms will either be by alternating allocation or a similar method.

After each organism addition, the pill cup is checked to ensure that all amphipods were successfully transferred to the test chamber. If organisms are observed in the cup, they are rinsed down into the test chamber using saltwater. The cup is checked again, and the process is repeated until all organisms have been transferred. Test chambers should be checked for amphipods that remain in the overlying water surface tension. Animals that appear healthy should be encouraged to descend using a gentle stream of water from a wash bottle or a pipette. Animals that appear to be dying should be replaced with animals from the original organism pool. Organisms that burrow, but then resurface, are not replaced.

5.3.18 Animals are not fed during the assay. Obvious abnormalities and mortalities will be recorded within the project's raw data.

5.4 Study Termination

5.4.1 On the final day of the assay, water qualities measurements are collected from each

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test replicate. Overlying and pore water ammonia samples are collected for analysis from the remaining surrogate chambers.

5.4.2 The contents of each vessel are individually washed through 0.5 mm sized screen and gently rinsed with water to remove sediment. The material remaining on the screen is examined over a light table to recover remaining amphipods. Note that *A. abdita* are tube-dwelling organisms. It is imperative that all sediment is examined very carefully. Sediment tubes and clumps must be gently broken apart to recover all surviving organisms. Any amphipod that shows signs of movement is considered alive and recorded.

5.5 Calibration and standardization are not applicable components of this method and procedure.

6.0 Quality Control Requirements

6.1 Survival acceptance criteria in the laboratory control is ≥90%. Survival in the reference control sediment is >80%. If survival in reference sediment exposures is <80%, the client should be contacted immediately as the affected test(s) may have to be repeated. The client may choose to contact the New York District. Any delays in contacting the client may lead to problems meeting holding times if a retest is required.

6.2 A reference toxicant evaluation must be successfully conducted with each batch of organisms. The reference toxicant is a 96-hour 'water only' test and the toxicant used will be ammonia (NH3). For more information see SOP QA-1114 "Conduct of Reference Toxicant Assays".

6.3 It is crucial to refer to each project's scope of work before the beginning of testing to ensure that all individual requirements are satisfied.

6.4 It is imperative that all materials (i.e. data sheets) are reviewed on a daily basis by personnel performing the assay to ensure that all applicable data is available and accurate.

6.5 For the assay to be considered valid, conditions should meet those specified in Section 11.0: "Summary of Test Conditions".

6.6 Interferences

6.6.1 Living organisms present in the sample may compete with the amphipods or may be predators, reducing overall survival. This impact is mitigated by sieving samples prior to testing.

6.6.2 Ammonia has been recognized as a potential confounding factor in sediment evaluations. Monitoring of ammonia levels daily must take place to ensure <20mg/L total ammonia is maintained.

6.7 The salinity and pH of natural seawater obtained from the Hampton-Seabrook Estuary and used for laboratory salt water are monitored regularly. On a quarterly basis, water quality analyses are performed to monitor the following parameters: total mercury, total metals, pesticides, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB)

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congeners.

6.7.1 Saltwater is also tested annually for all parameters listed under ASTM 2002a.

6.8 Detection limit is not an applicable aspect of the quality control requirements for this method and procedure.

7.0 Calculations and Reporting

7.1 Data Analysis

Survival data from each treatment will be subjected to the one way student unpaired T-test, or another appropriate statistical test, to determine if significant differences exist between treatments and the control. Statistical evaluations will be made with CETIS™ (Comprehensive Environmental Toxicity Information System) software using appropriate standard statistical models.

7.1.1 Prior to statistical analysis, the survival data will be reviewed to determine the presence of outliers. If outliers are found, an explanation must be sought. If a reasonable source for the deviation is found, the value may be excluded from further analysis. If no explanation is found, the analysis should be performed both with and without the questionable data point and both sets of results reported. All data sets are evaluated to determine sample variance homogeneity and normality. Those data sets meeting the criteria for normality and homogeneity are evaluated with parametric statistical models, while those that do not meet both criteria are evaluated using non-parametric models.

7.1.2 Endpoint to be evaluated is survival.

7.1.3 Statistical comparisons for each sample site will be made against the laboratory control treatment and each project reference site or as specified. Project reference sites may also be compared to one another.

7.2 Reporting

The final report will provide the following: cover page with title, name and address of the laboratory, unique identifier of the test report, date of issue, name and address of the client; introduction, materials, methods, sample receipt information, results of test, discussion of deviations, references, summary of reference toxicant tests. Copies of all bench sheets, raw data and statistical output will be provided in an appendix included with the report. Both the report and the data appendix will be paginated.

8.0 Corrective Actions

8.1 If survival fails to meet the minimum value specified by the protocol, the client will be notified and the test may be restarted.

8.2 In the event that an element of the assay falls outside acceptable limits, the deviation must

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be documented and corrective actions initiated by the analyst. The Laboratory Manager should be notified. The Laboratory Manager, using sound scientific practice, will determine if the study requires repeating or if the data is allowed to be accepted.

9.0 Health and Safety

9.1 As with all samples, gloves should be worn when handling sediment samples and chemicals. It is advisable to wear a lab coat to protect clothing. Full body coveralls are also available.

9.2 Assays and sample disposal will be conducted using procedures to minimize potential pollution of surface and ground waters, soils and sediments.

9.3 ESI is a fully licensed and approved waste generator and storage facility. ESI operates in full compliance with the State of New Hampshire Department of Environmental Services solid waste guidelines. Waste storage, transfer and disposal will be in compliance with State of New Hampshire Department of Environmental Services and US EPA waste disposal regulations.

9.4 Unused samples will be maintained in a secure location until disposal is approved by the client. Once a determination has been made that samples are no longer required by the project, they will be removed from their original container and transferred to a DOT approved container for transfer to an approved waste disposal facility.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedures outlined in this SOP.

10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOPs, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification will be based upon a review of the person's demonstrated abilities.

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TITLE: Acute Toxicity of Sediments to the Marine Amphipod, *Ampelisca abdita,* based on Region II Guidelines

11.0 Summary of Test Conditions
(Unless otherwise specified by client)

1.	Test Mode:	Static non-renewal (initiate static renewal methods if pore water ammonia concentration exceeds 20 ppm)
2.	Test Duration:	10 Days
3.	Renewal Schedule:	None (1 volume exchange daily for static renewal assays only)
4.	Temperature:	20±2°C
5.	Photoperiod:	Constant light
6.	Light Source:	Ambient laboratory illumination (no intensity required)
7.	Salinity:	28±2 ppt
8.	pH:	7.8 ± 0.5 (target)
9.	Test Chamber:	1 L beakers
10.	Solution Volume:	A minimum of ~625 mL overlying water
11.	Sediment Depth:	2 cm (~175 mL) Homogenized whole-sediment
12.	Organisms/Chamber:	20
13.	Replicates/Treatment:	5 minimum for endpoint; 11 for ammonia monitoring
14.	Organisms/Treatment:	100
15.	Age of Organisms:	Sub-adult (3-5 mm) retained on 0.7 mm sieve after passing through a 1 mm sieve
16.	Feeding Regime:	Must not be fed during 10-day bioassay
17.	Overlying Water:	Natural Seawater
18.	Aeration:	Continuous aeration to maintain ≥90% saturation
19.	Endpoint:	Survival
20.	Acceptability:	Mean lab control survival of ≥90%, ≥80% survival in reference sediment
21.	Support Chemistry:	Overlying water qualities: DO, pH, salinity and temperature on all replicates daily; overlying and pore water ammonia daily; hourly temperature readings in one surrogate vessel
22.	Sample Holding Time:	≤ 8 weeks, held at 4±2°C

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TITLE: US EPA Region II Marine Sediment Bioaccumulation Evaluation with the Bivalve Clam, *Macoma nasuta*

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Protocol Developed from QA-1469 / USEPA Region II RTM	K. Cram	01/2018

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TITLE: US EPA Region II Marine Sediment Bioaccumulation Evaluation with the Bivalve Clam, *Macoma nasuta*

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) summarizes details for conducting a 28-day bioaccumulation sediment evaluation using a marine bivalve clam, *Macoma nasuta*. The assay is conducted using guidelines developed by the US EPA and the Army Corp of Engineers (ACOE). This protocol is designed for use with samples from US EPA Region II and the New York District ACOE.

Testing involves using clams of similar age and size in a 28 day sediment exposure assay. The test is conducted using a flow-through system. At the end of the exposure, organisms are recovered from the treatments and counted to provide data for survival analysis. The organisms are allowed to depurate overnight and then they are processed or stored for bioaccumulation analysis.

Bioaccumulation tests are conducted to obtain information concerning the ability of organisms to accumulate materials from environmental sources. The results may also be an important consideration in hazard assessment.

2.0 Definitions

Overlying Water: The water placed over sediment in a test chamber during a test.

Reference Sediment: A whole sediment near an area of concern used to assess sediment conditions exclusive of material(s) of interest. The reference sediment may be used as an indicator of localized sediment conditions exclusive of the specific pollutant input of concern. Such sediment would be collected near the site of concern and would represent the background conditions resulting from any localized pollutant inputs as well as global pollutant input.

Sediment: Particulate material that usually lies below water; formulated particulate material that is intended to lie below water in a test.

Whole Sediment: Sediment and associated pore water which have had minimal manipulation. The term bulk sediment has been used synonymously with whole sediment.

3.0 Applicable Documents and References

ASTM. 2016. Annual Book of ASTM Standards. Volume 11.06. *Standard Guide for Determination of the Bioaccumulation of Sediment-Associated Contaminants by Benthic Invertebrates*. ASTM E 1688-10. West Conshohocken, PA.

US EPA, US Army Corps of Engineers. 1998. *Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual*. EPA-823-B-98-004. February 1998.

US EPA Region II, US Army Corps of Engineers New York District. 2016. Final Guidance for Performing Tests on Dredged Material Proposed for Ocean Dumping. April 2016.

EPA. 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Water to Freshwater

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and Marine Organisms, 5th ed. EPA-821-R-02-012. Office of Research and Development, Washington, D.C. 20460.

ASTM. 2002a. Standard Guide for Conducting Acute Toxicity Test with Fishes, Macroinvertebrates, and Amphibians In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05, E729-96. Philadelphia, PA., pp 135-156.

4.0 Materials and Apparatus

Flow-through water delivery system
Freezer (maintained at ≤-10°C)
Glass jars (for organism tissue storage after assay)
Glass pipettes (for aeration)
Plastic tubing (for aeration)
Large diameter plastic tubing (for siphoning)
Air compressor (for aeration)
Glass 10 gallon aquaria test vessels
Incubator (capable of maintaining 12-14°C, within ±2°C of target)
Ambient laboratory light
Timer (to provide 16 : 8 light : dark photoperiod)
Macoma nasuta: 2-4 years old; 28-45 mm shell length
Natural seawater
Marine salts (if needed to adjust seawater)
Plastic bags (for organism storage after assay)
Temperature logger
Tissue homogenizer
Metal spatula (for tissue preparation)
Ceramic knives (for tissue preparation)
De-ionized laboratory water
Water quality meters (capable of measuring DO, pH, salinity, specific conductivity and temperature)

5.0 Methods and Procedures

5.1 Test Material

5.1.1 Test substances will be clearly identified as to source, collection date, period of collection and description of collection methods. The laboratory will identify sample storage procedures and maintain receipt records. Records for sample storage, usage and disposal will be generated when necessary.

5.1.2 Test material is stored at 4±2°C in the original shipping container. Holding time is dependent upon contaminants of concern and specific project objectives. If the sediment being analyzed is for dredge material disposal permitting, the holding time < 8 weeks.

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5.1.3 Any overlying water in the sample container will be assumed to be pore water that has come out of the sediment due to settling since collection. Prior to sub-sampling the water will be mixed back into the sample.

5.2 Test Organisms

Organisms are obtained from a commercial source which meets ESI quality assurance standards. Confirmation of species is provided by the supplier. Each lot of organisms obtained from a commercial supplier must be taxonomically verified. A citation of the taxonomic key used and the distinguishing characteristics must be documented during verification.

5.2.1 Healthy organisms from the same source and of similar age and size are used in the test. *M. nasuta* used in testing are approximately 2-4 years old, with individual shell lengths of 28-45 mm.

5.2.2 Clams should be held and maintained for a minimum of 48 hours under flow-through in similar water and test conditions prior to assay initiation.

5.2.2.1 Holding should occur in clean sediment

5.2.2.2 Test organisms should experience at least two volume additions per day during the holding period.

5.2.2.3 Water temperature and salinity changes during holding should be <3°C and <3 ppt over any 24 hour period.

5.2.2.4 Water qualities (temperature, pH, salinity, and dissolved oxygen) must be measured daily during acclimation.

5.2.2.5 Feeding should occur daily while acclimating and never after. Flow-though should be temporarily stopped during feeding, to allow absorption by the clams.

5.2.2.6 If mortality exceeds 10% over the entire acclimation period and holding period, or 5% over the final 24 hours of acclimation, then it is recommended that the affected lot of organisms be discarded and the acclimation be started with a new batch of organisms.

5.2.3 Clams should be held in clean field collected marine laboratory sediment, as this provides necessary pressure against the clam shells. Clams not held in sediment for prolonged periods of time will die. Marine laboratory sediment also provides food for the clams, as clams should be fed daily while holding.

5.2.4 Pretest observation data concerning the source, handling procedures, disease treatment (if any), health, feeding and mortality of test animals will be recorded and reported as necessary.

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5.2.5 One set of clams, comprised of at least twice the number of organisms that will be added to each replicate, are reserved for analysis to determine background concentrations of target analytes. Organisms must be depurated overnight prior to freezing. Organisms must not be depurated longer than 24 hours.

5.3 Exposure Conditions

5.3.1 The test vessels are glass aquaria with a capacity of 38 L (10 gallons). Dimensions are approximately 50cm (l) x 25cm (w) x 30cm (h).

5.3.1.1 Prior to use, test vessels are cleaned by the use of laboratory grade detergent, dilute 10% HCl, full-strength pesticide-grade acetone, multiple tap water and de-ionized water rinses. This approach is based on EPA 2002 guidance.

5.3.2 Photoperiod is set at 16 hours light : 8 hours dark. Light is provided by ambient LED laboratory illumination.

5.3.3 Each treatment consists of a minimum of 5 replicates (with the exception of the laboratory control which consists of 3 replicates) and a minimum of 20 organisms per replicate. Organisms are randomly assigned to test vessels. Test vessel size and number of replicates, along with sediment and overlying water volumes, may be modified to provide adequate tissue for further analysis once the bioassay portion is completed.

5.3.3.1 For flow-through exposures, organisms can be added after an overnight settling period with the water flowing.

5.3.3.2 If any organisms fails to burrow within the first hour of being added to tanks, they should be replaced.

5.3.4 Sediments are sieved through a 2 mm sieve to remove large debris prior to use. Each vessel contains approximately 6 L of sediment and 20 - 30 L of overlying water. The sediment must have a depth of at least ≥5 cm. A sediment depth of 7-11 cm is recommended if sediment has high silt content. Sediment should be smoothed to a uniform depth and must be consistent among all test treatments.

5.3.5 Laboratory control sediment is collected from the Hampton Estuary, Hampton, New Hampshire.

5.3.6 Overlying water is natural seawater that is obtained from the Hampton-Seabrook estuary. Stored water is circulated to provide aeration and passed through multiple ≤50 µm filter to remove debris. Salinity of the overlying water should be 30±2 ‰.

If the seawater salinity is <28‰, then the water's salinity is increased with the addition of EDTA free commercially available marine salts. If the seawater salinity is >32‰, then the water's salinity is decreased with the addition of laboratory de-ionized water.

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5.3.7 Studies are conducted with constant flow-through water renewal. The flow rate should be set to at least 6 volume additions per 24 hour time period. Incoming water renewal occurs in a way that does not disturb the surface of the sediments or cause stress to the organisms.

5.3.8 Water temperature is maintained at 12 - 14°C. An automatic temperature logger is used to record temperature hourly.

5.3.9 Test chambers are not aerated unless DO concentrations fall below 60% saturation. If this occurs, aeration is supplied to all test chambers.

5.3.10 Clams are exposed to sediments for 28 days. The study design must include a laboratory control treatment and test sediment treatment(s). It may include reference control treatment(s).

5.3.11 Water qualities (dissolved oxygen, pH, salinity and temperature) are measured in each test chamber daily (target pH is 7.8±0.5). Mortality and sublethal symptoms are recorded daily during the test. The flow rate is monitored daily and increased/decreased to maintain a minimum of 6 volume replacements per 24 hour period.

5.3.12 Organisms are not fed during the assay.

5.4 Study Termination

5.4.1 Test aquaria replicates are systematically drained of overlying water by siphon and replicate vessels are hand searched for surviving organisms. Surviving clams are enumerated, survival numbers per replicate are recorded on recovery record bench logs.

5.4.2 Once all surviving clams are recovered, each test aquaria is rinsed out with clean seawater. The organisms are returned to their respective aquaria sediment free, flow-through conditions are re-initiated and the clams are allowed to depurate overnight.

5.4.3 After depuration, each test replicate is re-siphoned. Organisms are poured out of the test aquaria into a screen sieve, rinsed with de-ionized laboratory water and transferred to a labeled plastic bag. (Each test replicate has its own corresponding labeled plastic bag.) The organisms are then frozen until needed for tissue preparation.

5.4.4 Control organisms should be archived at the end of the test until after the tissue data has passed EPA's QA review.

5.5 Tissue Preparation

The clams are thawed just prior to preparation. Tissue is removed from the shell and the

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shells are discarded. Each test replicate's tissue is ground separately using a tissue homogenizer. Ground tissue is transferred to labeled glass jars and frozen until analysis.

5.6 Calibration and standardization are not applicable components of this method and procedure.

6.0 Quality Control Requirements

6.1 Acceptance Criteria

Survival in the laboratory control sediment after 28 days of exposure is ≥90%. Sufficient tissue mass has been generated for chemical analysis based on project specific objectives.

If control survival is <90%, the client should be contacted immediately as the affected test(s) may have to be repeated. The client may choose to contact the New York District. Any delays in contacting the client may lead to problems meeting holding times if a retest is required.

6.2 A reference toxicant evaluation must be successfully conducted with each batch of organisms. The reference toxicant is a 96-hour 'water only' test.

6.3.1 The reference toxicant evaluated will be copper (Cu).

6.3.2 If ≥90% in controls, then the reference toxicant test will be considered valid. If <90%, assay may need to be restarted. The Laboratory Manager is notified for appropriate action.

6.3.3 Results must fall between ±2 standard deviations from the historical mean.

6.3.4 For more information see SOP QA-1114 "Conduct of Reference Toxicant Assays".

6.3 It is crucial to refer to the client's project specific Statement Of Work (SOW) before the beginning of testing to ensure that all individual requirements are satisfied. It is also imperative that all materials (i.e. data sheets) are reviewed on a daily basis by personnel performing the assay to ensure that all applicable data is available and accurate.

6.4 The salinity and pH of natural seawater obtained from the Hampton-Seabrook Estuary and used for laboratory salt water are monitored regularly. On a quarterly basis, water quality analyses are performed to monitor the following parameters: total mercury, total metals, pesticides, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners.

6.4.1 Saltwater is also tested annually for all parameters listed under ASTM 2002a.

6.5 Detection limit is not an applicable aspect of the quality control requirements for this method and procedure.

7.0 Calculations and Reporting

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7.1 Data Analysis

Data from the assay may be subjected to statistical comparisons made against the laboratory or reference control sample using CETIS™ (Comprehensive Environmental Toxicity Information System) software or similar software.

7.2 The final report will provide the following: cover page with title, name and address of the laboratory, unique identifier of the test report, date of issue, name and address of the client; introduction, materials, methods, sample receipt information, results of test, discussion of deviations, and references. Copies of all bench sheets, raw data and statistical output will be provided in an appendix included with the report. Both the report and the data appendix will be paginated.

8.0 Corrective Actions

8.1 In the event that an element of the assay falls outside acceptable limits, the deviation must be documented and corrective actions initiated by the analyst. The laboratory manager must be notified. The laboratory manager, using sound scientific practice, will determine if the study requires repeating or if the data is allowed to be accepted.

8.2 If survival in the control sediment is less than 90%, then the following should be evaluated:

a) Is there adequate replication to obtain sufficient statistical power?
b) Is there adequate tissue for chemical analyses?
c) Is the system contaminated?
d) Is the control sediment contaminated?
e) Are there other quality control problems?

If answers to a) or b) are “no”, then the test may need to be rerun.
If answers to c) through e) are “yes”, then the test may need to be rerun.

If there is adequate tissue for chemical analysis, sufficient statistical power, no contamination and review does not reveal any quality control problems, then the test is still considered valid.

9.0 Health and Safety

9.1 As with all samples, gloves should be worn when handling sediment samples and chemicals. It is advisable to wear a lab coat to protect clothing. Full body coveralls are also available.

9.2 Assays and sample disposal will be conducted using procedures to minimize potential pollution of surface and ground waters, soils and sediments.

9.3 ESI is a fully licensed and approved hazardous waste generator and storage facility. ESI operates in full compliance with the State of New Hampshire Department of Environmental Services solid waste guidelines. Waste storage, transfer and disposal will be in compliance with State of New Hampshire Department of Environmental Services and US EPA waste

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disposal regulations.

9.4 Unused samples will be maintained in a secure location until disposal is approved by the client. Once a determination has been made that samples are no longer required by the project, they will be removed from their original container and transferred to a DOT approved container for transfer to an approved waste disposal facility.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedures outlined in this SOP.

10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOP's, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification will be based upon a review of the person's demonstrated abilities.

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TITLE: US EPA Region II Marine Sediment Bioaccumulation Evaluation with the Bivalve Clam, *Macoma nasuta*

11.0 Summary of Test Conditions

(Unless otherwise specified by client)

1.	Test Mode:	Flow-through (minimum 6 volume additions per day, 6 vol/day)
2.	Test Duration:	28 Days
3.	Temperature:	Daily mean 12-14°C, within ±2°C of target, with no readings beyond ±3°C of target
4.	Photoperiod:	16 hour light/ 8 hour dark
5.	Light Source:	Ambient laboratory light
6.	Light Intensity:	500 to 1000 lux
7.	Salinity:	30±2‰
8.	Test Chamber:	38 L (10 gallon) Aquaria
9.	Overlying Water Volume:	20 - 30 L overlying water
10.	Sediment Depth:	5 cm (minimum); ~6 L per vessel. 7-11 cm recommended if sediment has high silt content
11.	Organisms/Chamber:	20 (minimum)
12.	Replicates/Treatment:	5 each for sample sites and reference control, 3 for Laboratory Control
13.	Age of Organisms:	2-4 years old; 28-45 mm shell length
14.	Feeding Regime:	None
15.	Target pH:	7.8±0.5
16.	Overlying Water:	Natural Seawater
17.	Aeration:	Aerate if DO concentration falls below 60% saturation
18.	Effect Measured:	Survival and Bioaccumulation
19.	Acceptability:	Laboratory Control survival ≥90% and sufficient tissue mass for analysis
20.	Analysis:	Statistical comparison of survival and body burdens
21.	Water Quality:	Daily dissolved oxygen, pH, salinity and temperature in all chambers; Hourly temperature readings in a surrogate vessel. Ammonia analysis of each batch of natural salt water pumped into the system.
22.	Holding Time:	Dredge permit sample holding time is 8 weeks

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TITLE: US EPA Region II Marine Sediment Bioaccumulation Evaluation with the Polychaete, *Nereis virens*

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Protocol Developed from QA-1469 / USEPA Region II RTM	K. Cram	01/2018

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1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) summarizes details for conducting a 28-day bioaccumulation sediment evaluation using a marine polychaete worm, *Nereis virens*. The assay is conducted using guidelines developed by the US EPA and the Army Corp of Engineers (ACOE). This protocol is designed for use with samples from US EPA Region II and the New York District ACOE.

Testing involves using worms of similar age and size in a 28 day sediment exposure assay. The test is conducted using a flow-through system. At the end of the exposure, organisms are recovered from the treatments and counted to provide data for survival analysis. The organisms are allowed to depurate overnight and then they are processed or stored for bioaccumulation analysis.

Bioaccumulation tests are conducted to obtain information concerning the ability of organisms to accumulate materials from environmental sources. The results may also be an important consideration in hazard assessment.

2.0 Definitions

Overlying Water: The water placed over sediment in a test chamber during a test.

Reference Sediment: A whole sediment near an area of concern used to assess sediment conditions exclusive of material(s) of interest. The reference sediment may be used as an indicator of localized sediment conditions exclusive of the specific pollutant input of concern. Such sediment would be collected near the site of concern and would represent the background conditions resulting from any localized pollutant inputs as well as global pollutant input.

Sediment: Particulate material that usually lies below water; formulated particulate material that is intended to lie below water in a test.

Whole Sediment: Sediment and associated pore water which have had minimal manipulation. The term bulk sediment has been used synonymously with whole sediment.

3.0 Applicable Documents and References

ASTM. 2016. Annual Book of ASTM Standards. Volume 11.06. *Standard Guide for Determination of the Bioaccumulation of Sediment-Associated Contaminants by Benthic Invertebrates*. ASTM E 1688-10. West Conshohocken, PA.

US EPA, US Army Corps of Engineers. 1998. *Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual*. EPA-823-B-98-004. February 1998.

US EPA Region II, US Army Corps of Engineers New York District. 2016. Final Guidance for Performing Tests on Dredged Material Proposed for Ocean Dumping. April 2016.

Department of Defense (DoD), Department of Energy (DoE). 2017. *DoD Quality Systems Manual (QSM) for Environmental Laboratories*. Version 5.1.

EPA. 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Water to Freshwater

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and Marine Organisms, 5th ed. EPA-821-R-02-012. Office of Research and Development, Washington, D.C. 20460.

ASTM. 2002a. Standard Guide for Conducting Acute Toxicity Test with Fishes, Macroinvertebrates, and Amphibians In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05, E729-96. Philadelphia, PA., pp 135-156.

4.0 Materials and Apparatus

Flow-through water delivery system
Freezer (maintained at ≤-10°C)
Glass jars (for organism tissue storage after assay)
Glass pipettes (for aeration)
Plastic tubing (for aeration)
Large diameter plastic tubing (for siphoning)
Air compressor (for aeration)
Glass 10 gallon aquaria test vessels
Incubator (capable of maintaining 20°C, within ±2°C of target)
Ambient laboratory light
Timer (to provide 16 : 8 light : dark photoperiod)
Nereis virens: (Adult; 3-15 g/organism)
Natural seawater
Marine salts (if needed to adjust seawater)
Plastic bags (for organism storage after assay)
Temperature logger
Tissue homogenizer
De-ionized laboratory water
Plastic bags (quart or gallon zip style)
Water quality meters (capable of measuring DO, pH, salinity, specific conductivity and temperature)

5.0 Methods and Procedures

5.1 Test Material

5.1.1 Test substances will be clearly identified as to source, collection date, period of collection and description of collection methods. The laboratory will identify sample storage procedures and maintain receipt records. Records for sample storage, usage and disposition will be generated when necessary.

5.1.2 Test material is stored at 4±2°C in the original shipping container. Holding time is dependent upon contaminants of concern and specific project objectives. If the sediment being analyzed is for dredge material disposal permitting, the holding time < 8 weeks.

5.1.3 Any overlying water in the sample container will be assumed to be pore water that has come out of the sediment due to settling since collection. Prior to sub-sampling the water will be mixed back into the sample.

5.2 Test Organisms

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Organisms are obtained from a commercial source which meets ESI quality assurance standards. Confirmation of species is provided by the supplier. Each lot of organisms obtained from a commercial supplier must be taxonomically verified. A citation of the taxonomic key used and the distinguishing characteristics must be documented during verification.

5.2.1 Healthy organisms from the same source and of similar age and size are used in the test. *N. virens* used in testing are adults, weighing 3-15 grams per organism.

5.2.2 Worms should be held and maintained for a minimum of 48 hours under flow-through in similar water and test conditions prior to assay initiation.

5.2.2.1 Holding should occur in shallow clean sediment

5.2.2.2 Test organisms should experience at least two volume additions per day during the holding period.

5.2.2.3 Water temperature and salinity changes during holding should be <3°C and <3 ppt over any 24 hour period.

5.2.2.4 Water qualities (temperature, pH, salinity, and dissolved oxygen) must be measured daily during acclimation.

5.2.2.5 Feeding should occur daily while acclimating and never after. Flow-though should be temporarily stopped during feeding, to allow absorption by the worms.

5.2.2.6 If mortality exceeds 10% over the entire acclimation period and holding period, or 5% over the final 24 hours of acclimation, then it is recommended that the affected lot of organisms be discarded and the acclimation be started with a new batch of organisms.

5.2.3 Pretest observation data concerning the source, handling procedures, disease treatment (if any), health, feeding and mortality of test animals will be recorded and reported as necessary.

5.2.4 One set of worms, comprised of at least twice the number of organisms that will be added to each replicate, are reserved for analysis to determine background concentrations of target analytes.

5.2.4.1 Organisms must be depurated overnight prior to freezing.

5.2.4.2 Organisms must not be depurated longer than 24 hours.

5.2.4.3 Fecal material from worms must be siphoned twice during the 24-hour depuration period.

5.3 Exposure Conditions

5.3.1 The test vessels are glass aquaria with a capacity of 38 L (10 gallons). Dimensions

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are approximately 50cm (l) x 25cm (w) x 30cm (h).

5.3.1.1 Prior to use, test vessels are cleaned by the use of laboratory grade detergent, dilute 10% HCl, full-strength pesticide-grade acetone, multiple tap water and de-ionized water rinses. This approach is based on EPA 2002 guidance.

5.3.2 Photoperiod is set at 16 hours light : 8 hours dark. Light is provided by ambient LED laboratory illumination.

5.3.3 Each treatment consists of a minimum of 5 replicates (with the exception of the laboratory control which consists of 3 replicates) and a minimum of 20 organisms per replicate. Organisms are randomly assigned to test vessels. Test vessel size and number of replicates, along with sediment and overlying water volumes, may be modified to provide adequate tissue for further analysis once the bioassay portion is completed.

5.3.3.1 For flow-through exposures, organisms can be added after an overnight settling period with the water flowing.

5.3.3.2 If any organisms fails to burrow within the first hour of being added to tanks, they should be replaced.

5.3.4 Sediments are sieved through a 2 mm sieve to remove large debris prior to use.

5.3.4.1 Each vessel contains approximately 6 L of homogenized sediment and 20 - 30 L of overlying water.

5.3.4.2 The sediment must have a depth of at least ≥5 cm, but a sediment depth of 7-11 cm is recommended if sediment has high silt content.

5.3.4.3 Sediment should be smoothed to a uniform depth and must be consistent among all test treatments.

5.3.5 Laboratory control sediment is collected from the Hampton Estuary, Hampton, New Hampshire.

5.3.6 Overlying water is natural seawater that is obtained from the Hampton-Seabrook estuary. Stored water is circulated to provide aeration and passed through multiple ≤50 µm filter to remove debris. Salinity of the overlying water should be 30±2‰.

If the seawater salinity is <28‰, then the water's salinity is increased with the addition of EDTA free commercially available marine salts. If the seawater salinity is >32‰, then the water's salinity is decreased with the addition of laboratory de-ionized water.

5.3.7 Studies are conducted with constant flow-through water renewal. The flow rate should be set to at least 6 volume additions per 24 hour time period. Incoming water renewal occurs in a way that does not disturb the surface of the sediments or cause stress to the organisms.

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5.3.8 Water temperature is maintained at 20±2°C. An automatic temperature logger is used to record temperature hourly.

5.3.9 Test chambers are not aerated unless DO concentrations fall below 60% saturation. If this occurs, aeration is supplied to all test chambers.

5.3.10 Worms are exposed to sediments for 28 days. The study design must include a laboratory control treatment, reference control treatment and test sediment treatment(s).

5.3.11 Water qualities (dissolved oxygen, pH, salinity and temperature) are measured in each test chamber daily (target pH is 7.8±0.5). Mortality and sublethal symptoms are recorded daily during the test. The flow rate is monitored daily and increased/decreased to maintain a minimum of 6 volume replacements per 24 hour period.

5.3.12 Total ammonia levels are analyzed on every addition of natural sea water received into the system from pumping in.

5.3.13 Organisms are not fed during the assay.

5.4 Study Termination

5.4.1 Test aquaria replicates are systematically drained of overlying water by siphon and replicate vessels are hand searched for surviving organisms. Surviving worms are enumerated, survival numbers per replicate are recorded on recovery record bench logs.

5.4.2 Once all surviving worms are recovered, each test aquaria is rinsed out with clean seawater. The organisms are returned to their respective aquaria completely free of sediment, flow-through conditions are re-initiated and the worms are allowed to depurate overnight.

5.4.3 After depuration, each test replicate is re-siphoned. Organisms are poured out of the test aquaria into a screen sieve, rinsed with de-ionized laboratory water and transferred to a labeled plastic bag. (Each test replicate has its own corresponding labeled plastic bag.) The organisms are then frozen until needed for tissue preparation.

5.4.4 Control organisms should be archived at the end of the test until after the tissue data has passed EPA's QA review.

5.5 Tissue Preparation

The worms are thawed just prior to preparation. Each test replicate's tissue is ground separately using a tissue homogenizer. Ground tissue is transferred to labeled glass jars and frozen until analysis.

5.6 Calibration and standardization are not applicable components of this method and procedure.

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6.0 Quality Control Requirements

6.1 Acceptance Criteria

Survival in the laboratory control sediment after 28 days of exposure is ≥90%. Sufficient tissue mass has been generated for chemical analysis based on project specific objectives.

If control survival is <90%, the client should be contacted immediately as the affected test(s) may have to be repeated. The client may choose to contact the New York District. Any delays in contacting the client may lead to problems meeting holding times if a retest is required.

6.2 A reference toxicant evaluation must be successfully conducted with each batch of organisms. The reference toxicant is a 96-hour 'water only' test.

6.3.1 The reference toxicant evaluated will be copper (Cu).

6.3.2 If ≥90% in controls, then the reference toxicant test will be considered valid. If <90%, assay may need to be restarted. The Laboratory Manager is notified for appropriate action.

6.3.3 Results must fall between ±2 standard deviations from the historical mean.

6.3.4 For more information see SOP QA-1114 "Conduct of Reference Toxicant Assays".

6.3 It is crucial to refer to the client's project specific Statement Of Work (SOW) before the beginning of testing to ensure that all individual requirements are satisfied. It is also imperative that all materials (i.e. data sheets) are reviewed on a daily basis by personnel performing the assay to ensure that all applicable data is available and accurate.

6.4 The salinity and pH of natural seawater obtained from the Hampton-Seabrook Estuary and used for laboratory salt water are monitored regularly. On a quarterly basis, water quality analyses are performed to monitor the following parameters: total mercury, total metals, pesticides, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners.

6.4.1 Saltwater is also tested annually for all parameters listed under ASTM 2002a.

6.5 Detection limit is not an applicable aspect of the quality control requirements for this method and procedure.

7.0 Calculations and Reporting

7.1 Data Analysis

Data from the assay may be subjected to statistical comparisons made against the laboratory or reference control sample using CETIS™ (Comprehensive Environmental Toxicity Information System) software or similar software.

7.2 The final report will provide the following: cover page with title, name and address of the laboratory, unique identifier of the test report, date of issue, name and address of the client; introduction, materials, methods, sample receipt information, results of test, discussion of

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deviations, references. Copies of all bench sheets, raw data and statistical output will be provided in an appendix included with the report. Both the report and the data appendix will be paginated.

8.0 Corrective Actions

8.1 In the event that an element of the assay falls outside acceptable limits, the deviation must be documented and corrective actions initiated by the analyst. The laboratory manager must be notified. The laboratory manager, using sound scientific practice, will determine if the study requires repeating or if the data is allowed to be accepted.

8.2 If survival in the control sediment is less than 90%, then the following should be evaluated:

a) Is there adequate replication to obtain sufficient statistical power?
b) Is there adequate tissue for chemical analyses?
c) Is the system contaminated?
d) Is the control sediment contaminated?
e) Are there other quality control problems?

If answers to a) or b) are “no”, then the test may need to be rerun.
If answers to c) through e) are “yes”, then the test may need to be rerun.

If there is adequate tissue for chemical analysis, sufficient statistical power, no contamination and review does not reveal any quality control problems, then the test is still considered valid.

9.0 Health and Safety

9.1 As with all samples, gloves should be worn when handling sediment samples and chemicals. It is advisable to wear a lab coat to protect clothing. Full body coveralls are also available.

9.2 Assays and sample disposal will be conducted using procedures to minimize potential pollution of surface and ground waters, soils and sediments.

9.3 ESI is a fully licensed and approved hazardous waste generator and storage facility. ESI operates in full compliance with the State of New Hampshire Department of Environmental Services solid waste guidelines. Waste storage, transfer and disposal will be in compliance with State of New Hampshire Department of Environmental Services and US EPA waste disposal regulations.

9.4 Unused samples will be maintained in a secure location until disposal is approved by the client. Once a determination has been made that samples are no longer required by the project, they will be removed from their original container and transferred to a DOT approved container for transfer to an approved waste disposal facility.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible

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for following the procedures outlined in this SOP.

10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOP's, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification will be based upon a review of the person's demonstrated abilities.

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TITLE: US EPA Region II Marine Sediment Bioaccumulation Evaluation with the Polychaete, *Nereis virens*

11.0 Summary of Test Conditions
(Unless otherwise specified by client)

1.	Test Mode:	Flow-through (minimum 6 volume additions per day)
2.	Test Duration:	28 Days
3.	Temperature:	20±2 °C, with no readings beyond ±3 °C
4.	Photoperiod:	16 hour light/ 8 hour dark
5.	Light Source:	Ambient laboratory illumination
6.	Salinity:	30±2 ‰
7.	Test Chamber:	38 L (10 gallon) Aquaria
8.	Overlying Water Volume:	20 - 30 L overlying water
9.	Sediment Depth:	5 cm (minimum); ~6 L per vessel. 7-11cm recommended if sediment has high silt content
10.	Organisms/Chamber:	20 minimum
11.	Replicates/Treatment:	5 each for sample sites and reference control, 3 for Laboratory Control
12.	Age of Organisms:	Adult (3 - 15 grams / organism)
13.	Feeding Regime:	None
14.	pH:	7.8±0.5 (target)
15.	Overlying Water:	Natural Seawater
16.	Aeration:	Aerate if DO concentration falls below 60% saturation
17.	Effect Measured:	Survival and Bioaccumulation
18.	Acceptability:	Laboratory control survival ≥90% and sufficient tissue mass for analysis
19.	Analysis:	Statistical comparison of survival and body burdens
20.	Water Quality:	Daily dissolved oxygen, pH, salinity and temperature in all chambers; Hourly temperature readings in a surrogate vessel. Ammonia analysis of each batch of natural salt water pumped into the system.
21.	Holding Time:	Dredge permit sample holding time < 8 weeks

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TITLE: Acute Toxicity of Proposed Dredged Sediment Suspended Particulate Phase Solution to the Inland Silverside Minnow*, Menidia beryllina*, based on Region II Guidance

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Creation, based on QA-1451	J. T. Provencher	01/2018

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1.0 Purpose and Applicability

The purpose of this Standard Operating Procedure is to describe how to determine the 96 hour median lethal concentration (LC-50) of a dredged sediment elutriate solution to the silverside minnow, *Menidia beryllina*. This protocol is designed for use with samples from US EPA Region II and the New York District ACOE. Testing is conducted as a static exposure and is referred to as a suspended particulate phase (abbreviated as SPP) evaluation.

Fish, 9-14 days old, are exposed to a series of elutriate concentrations including a laboratory control and an untreated disposal site reference water control for a period of 96 hours. Survival and toxic symptoms are recorded at 1-2 hours, and at 24±2 hour intervals throughout the assay. Water quality data are collected daily on the static test treatments.

2.0 Definitions

Assay Review Checklist (ARC): A single document designed to ensure that the data package is complete on a daily basis and all data sheets are reviewed entirely.

Elutriate: Liquid solution recovered from a mix of sediment and water after a defined settling period and subsequent filtration or centrifugation to remove suspended material.

LC-50: Theoretical concentration of a toxicant or sample that results in 50% mortality of the test population.

NOEC: No Observed Effect Concentration; highest concentration which is not statistically different from the control.

Sediment: Particulate material that usually lies below water.

Suspended Particulate Phase (SPP): The liquid and material (sediment) remaining in suspension after a 1-hr settling period, unfiltered.

3.0 Applicable Documents and References

ASTM. 2017. Annual Book of ASTM Standards. Volume 11.06. *Standard Guide for Conducting Acute Toxicity on Test Materials with Fishes, Macroinvertebrates, and Amphibians* E729-96 (2014). West Conshohocken, PA.

US EPA. 2002. *Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms*. Fifth Edition. EPA-821-R-02-012.

US EPA, US ACE. 1991. *Evaluation of Dredged Material Proposed for Ocean Disposal - Testing Manual.* EPA-503/8-91/001, February 1991, 204 pages.

US EPA, US ACE. 1998. *Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual*. EPA-823-B-98-004, February 1998.

US EPA Region 2, US Army Corps of Engineers, New York District. 2016. *Guidance for Performing Tests*

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on Dredged Material Proposed for Ocean Disposal.

ASTM. 2002a. Standard Guide for Conducting Acute Toxicity Test with Fishes, Macroinvertebrates, and Amphibians In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05, E729-96. Philadelphia, PA., pp 135-156.

4.0 Materials and Apparatus

Analytical balance with sensitivity of 0.01g
Artemia nauplii, <24 hour old
250 mL glass beakers
Graduated cylinders
Incubator: 20 ± 2°C, 16:8 photoperiod
Large bore pipette
Light table
Marine salts
Menidia beryllina, 9-14 days old, 24-hour range in age
Plastic trays for beakers
Water quality meters

5.0 Methods and Procedures

5.1 Test Material

5.1.1 Test substances will be clearly identified as to source, collection date, period of collection and description of collection methods. The laboratory will identify sample storage procedures and maintain receipt records. Records for sample storage, usage and disposal will be generated when necessary.

5.1.2 Test material is stored at 4±2°C in the original shipping container. Samples may be held for up to 8 weeks and water may be held for up to 2 weeks from date/time of collection. As required, elutriate solutions will be prepared by EnviroSystems, Incorporated (ESI) from dredged sediments. If water is being used to create elutriates for chemical analysis, then water must be used within 7 days.

5.1.3 Any overlying water in the sediment sample container will be assumed to be pore water that has come out of the sediment due to settling since collection. Prior to sub-sampling the water will be mixed back into the sample.

5.2 Test Species

Organisms are obtained from a commercial source which meets ESI quality assurance standards. Confirmation of species is provided by the supplier. Each lot of organisms obtained from a commercial supplier must be taxonomically verified. A citation of the taxonomic key used and the distinguishing characteristics must be documented during verification.

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5.2.1 Healthy fish all of the same source and age, 9-14 days post-hatch and 24-hours range in age, are used in the test. Minnows received from a commercial supplier are assigned a unique lot ID and maintained until the start of the assay. Organism history data must accompany each batch of organisms. Organisms are held under static-renewal conditions at similar water quality, photoperiod and temperature as will be used during testing.

5.2.2 The recommended acclimation time for organisms is 48 hours and no less than 24 hours. Overlying water, natural clean sea water (SPP control/dilution water) should be renewed every 24 - 48 hours during the acclimation period.

5.2.2.1 Water temperature and salinity changes during holding should be <3°C and <3 ppt over any 24 hour period.

5.2.2.4 Water qualities (temperature, pH, salinity, and dissolved oxygen) must be measured daily during acclimation, both before and after any water renewals take place.

5.2.2.2 If mortality exceeds 10% over the entire acclimation period and holding period, or 5% over the final 24 hours of acclimation, then it is recommended that the affected lot of organisms be discarded and the acclimation be started with a new batch of organisms.

5.2.3 The organisms will be fed <24 hour old *Artemia* nauplii daily until the start of the test. It is very important not to overfeed, as there can be detrimental affects to water qualities and organism health.

5.2.4 Pretest observation data concerning the source, handling procedures, disease treatment (if any), health, feeding, mean dry weight and mortality of test animals is recorded and reported as necessary.

5.3 Exposure Conditions and Study Conduct

Exposure conditions and monitoring requirements detailed in the following paragraphs are the minimum requirements specified by ESI for this assay.

5.3.1 *M. beryllina* are exposed to elutriate solutions and control treatments for 96 hours under static non-renewal conditions.

5.3.1.1 Dilution water is natural seawater. It may be modified GP2, Forty Fathoms® or equivalent, artificial seawater prepared with Milli-Q® water or equivalent DI water, or clean natural seawater. Treatments include a laboratory control (0%), SPP solution (100%) and elutriate solution diluted to 50%, 10% and 1% concentrations.

5.3.2 The laboratory control water is natural seawater obtained from the Hampton-Seabrook estuary. Water is pumped through a ≤50 µm filter to remove debris and provide continuous aeration. Salinity of the water should be 30 ±2‰. If the seawater salinity is

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below 28 ppt, the sample is adjusted with EDTA free commercially available marine salts.

5.3.3 Water temperature is 20±2°C and salinity should be 30±2 ppt. Samples with salinity <28 ppt will be adjusted with EDTA free commercially available marine salts and noted on the bench record. Samples with salinity >32 ppt is typically run as it; however, a manager must be consulted.

5.3.4 Dissolved oxygen concentration should be maintained at ≥40% saturation. If dissolved oxygen (DO) is low, close the elutriate cubitainer allowing for some overlying air in the head space and vigorously shake the cubitainer for approximately 1 minute. Check the DO reading again and if the level remains low, then aerate the samples prior to mixing and the assay must be aerated.

5.3.5 Incubator temperatures are recorded daily in all incubators used for *M. beryllina* testing. Photoperiod in incubators is 16 hours light:8 hours dark.

5.3.6 Each treatment group will consist of 5 replicates with 20 organisms per replicate. Before adding solutions to test vessels or mixing dilutions, the salinity and DO of elutriates and natural seawater are checked. Dilution treatments are measured using graduated cylinders and mixed thoroughly. Each replicate contains 400 mL of solution in 600 mL glass beakers. Data sheets are clearly labeled and arranged in a logical manner.

5.3.7 Water qualities (salinity, pH, dissolved oxygen and temperature) are measured in each test replicate daily, and are monitored at 24±2 hour intervals throughout the assay. Ammonia is measured in the undiluted elutriate and control treatments upon assay initiation and termination.

5.3.8 *M. beryllina* are added to the test vessels with the aid of a light table. Fish are added gently, using an inverted glass pasteur pipette to prevent trauma to the organisms. Fish are randomly assigned to test vessels. Addition to test chambers is ordered so that all replicates are loaded in A, B, C, D and E order. Within replicate groups the order is alternated between low to high and high to low concentrations. This is referred to internally as “alternating allocation”.

5.3.9 Fish will be fed by adding approximately 0.2 mL of ≤24 hour old concentrated *Artemia* nauplii to each testing chamber after 48 hours of testing.

5.3.10 Mortality and any observed toxic symptoms, are recorded 1-2 hours after organisms are added. Survival is then recorded every 24±2 hours throughout the assay. Dead organisms should be counted and removed daily. The assay is terminated after 96 hours exposure.

5.3.10.1 In addition to counting the number of surviving organisms, any observed sub-lethal effects, such as behavioral or physical abnormalities should be recorded.

5.4 Calibration and standardization are not applicable components of this method and procedure.

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6.0 Quality Control Requirements

6.1 For the assay to be considered valid, conditions should meet those specified in Section 11.0 "Summary of Test Conditions".

6.2 Acceptance critera for survival in the laboratory control is ≥90%. If control survival is <90%, the client should be contacted immediately as the affected test(s) may have to be repeated. The client may choose to contact the New York District. Any delays in contacting the client may lead to problems meeting holding times if a retest is required.

6.3 A reference toxicant evaluation must be successfully conducted each month. The assay can run concurrent with a client assay. The reference toxicant used is ammonia (NH3). For more information see SOP QA-1114 “Conduct of Reference Toxicant Assays”.

6.4 It is crucial to refer to each project’s scope of work before the beginning of testing to ensure that all individual requirements are satisfied.

6.5 Data sheets should be reviewed on a daily basis by personnel performing the assay to ensure that all applicable data are available and accurate. The Assay Review Checklist (ARC) data sheet is a single document designed to ensure that the data package is complete on a daily basis and all data sheets are reviewed entirely.

6.6 During testing if the salinity exceeds 32ppt due to evaporation, DI water is added to the solution to bring the salinity closer to the initial value. This is noted on the bench record.

6.7 Occasionally, organisms occur naturally in client water samples. These organisms are removed, as they may be predatory or pathogenic and could affect test organism survival.

6.8 The salinity and pH of natural seawater obtained from the Hampton-Seabrook Estuary and used for laboratory salt water are monitored regularly. On a quarterly basis, water quality analyses are performed to monitor the following parameters: total mercury, total metals, pesticides, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners.

6.8.1 Saltwater is also tested annually for all parameters listed under ASTM 2002a.

6.9 Detection limit is not an applicable aspect of the quality control requirements for this method and procedure.

7.0 Calculations and Reporting

7.1 Data Analysis

Survival and development data from each treatment will be subjected to calculation of LC-50 and EC-50 values using linear interpolation or trimmed Spearman-Karber methods. Statistical evaluations will be made with CETIS® software.

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7.2 The final report will provide the following: cover page with title, name and address of the laboratory, unique identifier of the test report, date of issue, name and address of the client; introduction, materials, methods, sample receipt information, results of test, discussion of deviations, references, summary of reference toxicant tests. Copies of all bench sheets, raw data and statistical output will be provided in an appendix included with the report. Both the report and the data appendix will be paginated.

8.0 Corrective Actions

8.1 If survival in the laboratory control fails to meet minimum test acceptability criteria, the test may be considered "provisionally valid" as long as the other controls and all test treatments meet the minimum acceptability criteria.

8.2 In the event that an element of the assay falls outside acceptable limits, the deviation must be documented and corrective actions initiated by the analyst. The Laboratory Manager should be notified. The Laboratory Manager, using sound scientific practice, will determine if the study requires repeating or if the data is allowed to be accepted.

9.0 Health and Safety

9.1 As with all samples, gloves should be worn when handling sediment samples and chemicals. It is advisable to wear a lab coat to protect clothing.

9.2 At the end of an assay excess sample material and materials used in the assay will be disposed of appropriately. Material will be placed in an appropriate container for disposal at an approved disposal facility. If the material is classified as non-hazardous, the material may be disposed of in an appropriate waste container.

9.3 Assays and sample disposal will be conducted using procedures to minimize potential pollution of surface and ground waters, soils and sediments.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedures outlined in this SOP.

10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOP's, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification will be based upon a review of the person's demonstrated abilities.

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TITLE: Acute Toxicity of Proposed Dredged Sediment Suspended Particulate Phase Solution to the Inland Silverside Minnow*, Menidia beryllina*, based on Region II Guidance

11.0 Summary of Test Conditions
(Unless otherwise specified by client)

1.	Test Mode:	Static non renewal
2.	Salinity:	30±2 ppt
3.	Temperature:	20±2°C
4.	Photoperiod:	16 hr light/ 8 hr dark
5.	Light Source:	Ambient laboratory illumination
6.	Test Chamber:	250 mL (minimum), glass beakers - 600 mL preferred
7.	Solution Volume:	200 mL (minimum), 400 mL preferred
8.	Organisms/Chamber:	20
9.	Replicates/Treatment:	5
10.	Treatments:	Laboratory control (0%), SPP elutriate solution (100%) and diluted elutriate solutions (concentrations 50%,10% and 1%)
11.	Age of Organisms:	9 to 14 days, 24-hour range in age
12.	Food Source:	<24 hour old *Artemia* nauplii
13.	Feeding Regime:	Once after 48 hours of testing
14.	pH:	7.8±0.5 (target)
15.	Aeration:	None unless dissolved oxygen is below 40% saturation
16.	Dilution Water:	Clean natural seawater, alternatives could be Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water.
17.	Test Duration:	96 Hours
18.	Endpoint Measured:	Mortality (LC50) - Record survivorship per replicate at 0, 24, 48, 72 & 96 hours
19.	Acceptability:	≥90% survival in the laboratory water control
20.	Analysis:	Computation of 96 Hour LC-50 and NOEC
21.	Water Quality:	In all treatments: daily measurement of DO, pH, salinity and temperature in all replicates; ammonia in the controls and undiluted elutriate at the start and end of assay.

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TITLE: Acute Toxicity of Proposed Dredged Sediment Suspended Particulate Phase Solution to the Mysid Shrimp, *Americamysis bahia*, based on Region II Guidelines

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
0	Creation, based on QA-1452	J. T. Provencher	01/2018

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TITLE: Acute Toxicity of Proposed Dredged Sediment Suspended Particulate Phase Solution to the Mysid Shrimp, *Americamysis bahia*, based on Region II Guidelines

1.0 Purpose and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedure for determining the 96 hour median lethal concentration (LC-50) of a dredged sediment elutriate solution to the mysid, *Americamysis bahia*. This protocol is designed for use with samples from US EPA Region II and the New York District ACOE. Testing is conducted as a static exposure and is referred to as a suspended particulate phase (abbreviated as SPP) evaluation.

Mysids, 1-5 days old within 24-hour age range, are exposed to a series of elutriate concentrations including a laboratory control and an untreated disposal site reference water control for a period of 96 hours. Survival and toxic symptoms are recorded at 1-2 hours, and at 24±2 hour intervals throughout the assay. Water quality data are collected daily on the static test treatments.

2.0 Definitions

Assay Review Checklist (ARC): A single document designed to ensure that the data package is complete on a daily basis and all data sheets are reviewed entirely.

Elutriate: Liquid solution recovered from a mix of sediment and water after a defined settling period and subsequent filtration or centrifugation to remove suspended material.

LC-50: Theoretical concentration of a toxicant or sample that results in 50% mortality of the test population.

NOEC: No Observed Effect Concentration; highest concentration which is not statistically different from the control.

Sediment: Particulate material that usually lies below water.

Suspended Particulate Phase (SPP): The liquid and material (sediment) remaining in suspension after a 1-hr settling period, unfiltered.

3.0 Applicable Documents and References

ASTM. 2017. Annual Book of ASTM Standards. Volume 11.06. *Standard Guide for Conducting Acute Toxicity on Test Materials with Fishes, Macroinvertebrates, and Amphibians* E729-96 (2014). West Conshohocken, PA.

US EPA. 2002. *Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms*. Fifth Edition. EPA-821-R-02-012.

US EPA, US ACE. 1991. *Evaluation of Dredged Material Proposed for Ocean Disposal - Testing Manual.* EPA-503/8-91/001, February 1991.

US EPA, US ACE. 1998. *Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual*. EPA-823-B-98-004, February 1998.

US EPA Region 2, US Army Corps of Engineers, New York District. 2016. *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal*.

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ASTM. 2002a. Standard Guide for Conducting Acute Toxicity Test with Fishes, Macroinvertebrates, and Amphibians In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05, E729-96. Philadelphia, PA., pp 135-156.

4.0 Materials and Apparatus

Americamysis bahia, 1-5 days old, 24-hour range in age
Artemia nauplii, <24 hour old
600 mL glass beakers
Graduated cylinders
Incubator: 20 ± 2°C, 16:8 photoperiod
Large bore pipette
Light table
Marine salts
Plastic trays for beakers
Shallow dish for organism addition process
Water quality meters

5.0 Methods and Procedures

5.1 Test Material

5.1.1 Test substances will be clearly identified as to source, collection date, period of collection and description of collection methods. The laboratory will identify sample storage procedures and maintain receipt records. Records for sample storage, usage and disposal will be generated when necessary.

5.1.2 Test material is stored at 4±2°C in the original shipping container. Samples may be held for up to 8 weeks and water may be held for up to 2 weeks from date/time of collection. As required, elutriate solutions will be prepared by EnviroSystems, Incorporated (ESI) from dredged sediments. If water is being used to create elutriates for chemical analysis, then water must be used within 7 days.

5.1.3 Any overlying water in the sediment sample container will be assumed to be pore water that has come out of the sediment due to settling since collection. Prior to sub-sampling the water will be mixed back into the sample.

5.2 Test Species

Organisms are obtained from a commercial source which meets ESI quality assurance standards. Confirmation of species is provided by the supplier. Each lot of organisms obtained from a commercial supplier must be taxonomically verified. A citation of the taxonomic key used and the distinguishing characteristics must be documented during verification.

5.2.1 Healthy juveniles all of the same source and age, 1-5 days post-hatch, will be used

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in the tests. Mysids received from a commercial supplier are assigned a unique lot ID and maintained until the start of the assay. Organism history data must accompany each batch of organisms. Organism history data must accompany each batch of organisms. Organisms are held under static-renewal conditions at similar water quality, photoperiod and temperature as will be used during testing.

5.2.2 The recommended acclimation time for organisms is 48 hours and no less than 24 hours.

5.2.2.1 Overlying water will be clean sea water (SPP control/dilution water) and should be renewed every 24 - 48 hours during the acclimation period.

5.2.2.2 Water quality measurements (temperature, pH, salinity, and dissolved oxygen) must be measured daily during acclimation both pre and post renewal.

5.2.2.3 Water temperature and salinity changes during holding should be <3°C and <3 ppt over any 24 hour period.

5.2.2.4 The organisms will be fed <24 hour old *Artemia* nauplii daily (approximately 100 *Artemia* nauplii per mysid) until the start of the test.

5.2.2.5 If mortality exceeds 10% over the entire acclimation period and holding period, or 5% over the final 24 hours of acclimation, then it is recommended that the affected lot of organisms be discarded and the acclimation be started with a new batch of organisms.

5.2.3 Pretest observation data concerning the source, handling procedures, disease treatment (if any), health, feeding, mean dry weight and mortality of test animals is recorded and reported as necessary.

5.3 Exposure Conditions and Study Conduct

Exposure conditions and monitoring requirements detailed in the following paragraphs are the minimum requirements specified by ESI for this assay.

5.3.1 *A. bahia* are exposed to elutriate solutions and control treatments for 96 hours under static non-renewal conditions. Dilution water is natural seawater, but may be modified GP2, Forty Fathoms® or equivalent or artificial seawater prepared with Milli-Q® water or equivalent DI water. Treatments include a laboratory control (0%), undiluted SPP elutriate solution (100%), and elutriate solution diluted to 50%, 10% and 1% concentrations.

5.3.2 The laboratory control water is natural seawater obtained from the Hampton-Seabrook estuary. Water is pumped through a ≤50 µm filter to remove debris and provide constant aeration. Salinity of the water should be 30±2‰. If the seawater salinity is below 28 ppt, the sample is adjusted with EDTA free commercially available marine salts.

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5.3.3 Water temperature is 20±2°C and salinity should be 30±2 ppt. Samples with salinity <28 ppt will be adjusted with EDTA free commercially available marine salts and noted on the bench record. Samples with salinity >32 ppt is typically run as it; however, a manager must be consulted.

5.3.4 Dissolved oxygen concentration should be maintained at ≥40% saturation. If dissolved oxygen (DO) is low, close the elutriate cubitainer allowing for some overlying air in the headspace and vigorously shake the cubitainer for approximately 1 minute. Check the DO reading again and if the level remains low, then aerate the samples prior to mixing and the assay must be aerated.

5.3.5 Incubator temperatures are recorded daily in all incubators used for *A. bahia* testing. Photoperiod in incubators is 16 hours light:8 hours dark.

5.3.6 Each treatment group will consist of 5 replicates with 20 organisms per replicate. Before adding solutions to test vessels or mixing dilutions, the salinity and DO of elutriates and natural seawater are checked. Dilution treatments are measured using graduated cylinders and mixed thoroughly. Each replicate contains 400 mL of solution in 600 mL glass beakers. Data sheets are clearly labeled and arranged in a logical manner.

5.3.7 Water qualities (salinity, pH, dissolved oxygen and temperature) are measured in each test replicate daily, and are monitored at 24±2 hour intervals throughout the assay. Ammonia is measured in the undiluted elutriate and control treatments at the start and end of the assay.

5.3.8 *A. bahia* are added to the test vessels with the aid of a light table. Mysids are added gently, using an inverted glass pasteur pipette to prevent trauma to the organisms. Organisms are randomly assigned to test vessels. Addition to test chambers is ordered so that all replicates are loaded in A, B, C, D and E order. Within replicate groups the order is alternated between low to high and high to low concentrations. This is referred to internally as “alternating allocation”.

5.3.9 Mysids will be fed ≤24 hour old *Artemia* nauplii twice daily during the assay, at the beginning and end of the workday. Feeding will be at a rate of approximately 100 nauplii per mysid. Excess nauplii should be removed daily, during survival and water quality analysis.

5.3.10 Mortality and any observed toxic symptoms, are recorded 1-2 hours after organisms are added. Survival is then recorded every 24±2 hours throughout the assay. The assay is terminated after 96 hours exposure.

5.3.10.1 In addition to counting the number of surviving organisms, any observed sub-lethal effects, such as behavioral or physical abnormalities should be recorded.

5.4 Calibration and standardization are not applicable components of this method and procedure.

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6.0 Quality Control Requirements

6.1 For the assay to be considered valid, conditions should meet those specified in Section 11.0 "Summary of Test Conditions".

6.2 Acceptance criteria for survival in the laboratory control is ≥90%. If control survival is <90%, the client should be contacted immediately as the affected test(s) may have to be repeated. The client may choose to contact the New York District. Any delays in contacting the client may lead to problems meeting holding times if a retest is required.

6.3 A reference toxicant evaluation must be successfully conducted each month. The assay can run concurrent with a client assay. The reference toxicant used is ammonia (NH3). For more information see SOP QA-1114 “Conduct of Reference Toxicant Assays”.

6.4 It is crucial to refer to each project’s scope of work before the beginning of testing to ensure that all individual requirements are satisfied.

6.5 Data sheets should be reviewed on a daily basis by personnel performing the assay to ensure that all applicable data are available and accurate. The Assay Review Checklist (ARC) data sheet is a single document designed to ensure that the data package is complete on a daily basis and all data sheets are reviewed entirely.

6.6 During testing if the salinity exceeds 32ppt due to evaporation, DI water is added to the solution to bring the salinity closer to the initial value. This needs to be noted on the bench record.

6.7 Occasionally, organisms occur naturally in client water samples. These organisms should be removed, as they may be predatory or pathogenic and could affect test organism survival.

6.8 The feeding regimen must be strictly followed. *A. bahia* will consume one another if not regularly fed.

6.9 The salinity and pH of natural seawater obtained from the Hampton-Seabrook Estuary and used for laboratory salt water are monitored regularly. On a quarterly basis, water quality analyses are performed to monitor the following parameters: total mercury, total metals, pesticides, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners.

6.9.1 Saltwater is also tested annually for all parameters listed under ASTM 2002a.

6.10 Detection limit is not an applicable aspect of the quality control requirements for this method and procedure.

7.0 Calculations and Reporting

7.1 Survival and development data from each treatment will be subjected to calculation of LC-50

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and EC-50 values using linear interpolation or trimmed Spearman-Karber methods. Statistical evaluations will be made with CETIS® software.

7.2 The final report will provide the following: cover page with title, name and address of the laboratory, unique identifier of the test report, date of issue, name and address of the client; introduction, materials, methods, sample receipt information, results of test, discussion of deviations, references, summary of reference toxicant tests. Copies of all bench sheets, raw data and statistical output will be provided in an appendix included with the report. Both the report and the data appendix will be paginated.

8.0 Corrective Actions

8.1 If survival in the laboratory control fails to meet minimum test acceptability criteria, the test may be considered “provisionally valid” as long as the other controls and all test treatments meet the minimum acceptability criteria.

8.2 In the event that an element of the assay falls outside acceptable limits, the deviation must be documented and corrective actions initiated by the analyst. The laboratory manager should be notified. The laboratory manager, using sound scientific practice, will determine if the study requires repeating or if the data is allowed to be accepted.

9.0 Health and Safety

9.1 As with all samples, gloves should be worn when handling sediment samples and chemicals. It is advisable to wear a lab coat to protect clothing.

9.2 At the end of an assay excess sample material and materials used in the assay will be disposed of appropriately. Material will be placed in an appropriate container for disposal at an approved disposal facility. If the material is classified as non-hazardous, the material may be disposed of in an appropriate waste container.

9.3 Assays and sample disposal will be conducted using procedures to minimize potential pollution of surface and ground waters, soils and sediments.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedures outlined in this SOP.

10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification may include reading this and associated SOP’s, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification will be based upon a review of the person’s demonstrated abilities.

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TITLE: Acute Toxicity of Proposed Dredged Sediment Suspended Particulate Phase Solution to the Mysid Shrimp, *Americamysis bahia*, based on Region II Guidelines

11.0 Summary of Test Conditions
(Unless otherwise specified by client)

1.	Test Mode:	Static non-renewal
2.	Salinity:	30±2 ppt
3.	Temperature:	20±2°C
4.	Photoperiod:	16 hr light/ 8 hr dark
5.	Light Source:	Ambient laboratory illumination
6.	Test Chamber:	600 mL beaker (minimum)
7.	Solution Volume:	400 mL
8.	Organisms/Chamber:	20
9.	pH:	7.8±0.5 (target)
10.	Replicates/Treatment:	5
11.	Organsims/Concentration:	100
12.	Treatments:	Laboratory control (0%), undiluted elutriate solution (100%) and diluted elutriate solution (concentrations 50%,10% and 1%)
13.	Age of Organisms:	1-5 days; 24-hour range in age
14.	Food Source:	<24 hour old *Artemia* nauplii
15.	Feeding Regime:	Twice daily feeding of newly hatched *Artemia*
16.	Aeration:	None, unless dissolved oxygen is below 40% saturation
17.	Dilution Water:	Clean natural seawater, alternatives could be Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water.
18.	Test Duration:	96 Hours
19.	Endpoint:	Mortality (LC50) - Record survivorship per replicate at 0, 24, 48, 72 & 96 hours
20.	Acceptability:	≥90% survival in the laboratory water control
21.	Water Quality:	In all treatments: daily measurement of DO, pH, salinity; ammonia in the controls and undiluted elutriate at the start and end of assay.

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TITLE: Acute Toxic Effects of Waters on the Development of Marine Bivalve Larvae, based on Region II Guidelines

Approved By:

QA Manager:_____ Date:_____

Manager:_____ Date:_____

Revision History:

Revision Number	Changes	Revised By	Date
1	SOP Preparation	J. T. Provencher	01/2018

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TITLE: Acute Toxic Effects of Waters on the Development of Marine Bivalve Larvae, based on Region II Guidelines

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) describes a method for assessing the impact of waters on the development of embryos based on ASTM method E 724-98 for marine bivalves. *Mytilus edulis, Crassostrea virginica*, *Mercenaria mercenaria*, *Mulinia lateralis* and *M. Galloprovincialis* larvae may all be used, as well as potential other species depending on program requirements and availability. Waters include surface water, pore water, and aqueous solutions. This protocol is designed for use with samples from US EPA Region II and the New York District ACOE.

An acute toxicity test is conducted with bivalve embryos to obtain information concerning the acute effects of a short-term exposure of organisms to a test material using specific experimental design. Fertilized bivalve embryos are exposed to a series of concentrations plus a laboratory control for a period of 48 to 72 hours. Surrogate controls are prepared and run concurrent to the treatments. The test is terminated when $\geq$90% of the surrogate control embryos have reached the straight hinge or Prodissoconch I developmental stage.

2.0 Definitions

LC-50: Lethal Concentration; the theoretical concentration of a toxicant or sample that results in 50% mortality of the test population.

EC-50: Effective Concentration; the theoretical concentration of a toxicant or sample that results in a 50% reduction in a non-lethal endpoint, such as normal development, for the test population.

Embryo: Denotes the stages of development between fertilization of the egg and the straight hinge or Prodissoconch I larvae.

Straight Hinge or Prodissoconch I Larvae: Stage of development for the bivalve species generally occurring 48 hours after fertilization (see Figure 1).

3.0 Applicable Documents and References

ASTM. 2017. Annual Book of ASTM Standards. Volume 11.06. *Standard Guide for Conducting Static Acute Toxicity Tests Starting with Bivalve Embryos for Four Species of Saltwater Bivalve Molluscs*. Method E 724-98 (2012). West Conshohocken, PA.

US EPA Region 2, US Army Corps of Engineers, New York District. 2016. *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal*.

ASTM. 2002a. Standard Guide for Conducting Acute Toxicity Test with Fishes, Macroinvertebrates, and Amphibians In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05, E729-96. Philadelphia, PA., pp 135-156.

4.0 Materials and Apparatus

0.5 M Potassium iodide solution
20 mL Scintillation vials, with caps

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250 mL Beakers (minimum)
Adult Bivalves, healthy and sexually mature
Calibrated pipettes, macro (5 mL) and micro (100 µL) scale pipettes
Compound microscope, capable of up tp 100x magnification
Depression slide and Sedgwick-Rafter count cell
Dissecting microscope, 10 to 40X magnification
Disposable petri dishes, 47 mm diameter
Formaldehyde (10% buffered formalin phosphate)
Graduated cylinders, glass
Hemacytometer
Incubator, capable of 16 ± 2°C
Large glass bowls or baking dishes
Perforated plunger, constructed of acrylic plastic or fiberglass
Pipette tips
Plastic cups for gamete collection
Plastic transfer pipettes
Tally counter, two fields
Water quality meters
Water bath
Water heater
Centrifuge (max 1925 rpms)

5.0 Methods and Procedures

5.1 Test Material

5.1.1 Test substances will be clearly identified as to source, collection date, period of collection and description of collection methods. The laboratory will identify sample storage procedures and maintain receipt records. Records for sample storage, usage and disposal will be generated when necessary.

5.1.2 Test material is stored at 4±2°C in the original shipping container. Samples may be held for up to 8 weeks and water may be held for up to 2 weeks from date/time of collection. As required, elutriate solutions will be prepared by EnviroSystems, Incorporated (ESI) from dredged sediments. If water is being used to create elutriates for chemical analysis, then water must be used within 7 days.

5.1.3 Any overlying water in the sediment sample container will be assumed to be pore water that has come out of the sediment due to settling since collection. Prior to sub-sampling the water will be mixed back into the sample.

5.2 Test Species

Organisms are obtained from a commercial source which meets ESI quality assurance standards. Confirmation of species is provided by the supplier. Each lot of organisms obtained from a commercial supplier must be taxonomically verified. A citation of the taxonomic key used and the distinguishing characteristics must be documented during verification.

5.2.1 Bivalve embryos from in-house spawning of mature and healthy adult male and female organisms obtained from commercial suppliers are used in the test. All organisms must be collected from the same location. Adult bivalves are maintained in aquaria made

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from non-contaminating materials. Each aquaria has aeration added and is maintained in a flow-through water renewal system. All bivalve lots received from commercial suppliers are assigned a unique lot ID and maintained until the start of the assay.

5.2.2 Upon arrival, adult bivalves are inspected for signs of damage and cleaned to remove foreign material. Damaged bivalves are discarded. Reproductive condition of the bivalves should be determined if not specified by the provider. In this case, a number of organisms are sacrificed and the condition of the gonads are determined. Tissue overlying the digestive gland that is creamy white, pink, or orange indicates ripe gonads. Gametes from a ripe male are minute and active in salt water within 30 minutes, and gametes from a ripe female are large and change from teardrop shaped to spherical in a gelatinous matrix in salt water within 30 minutes.

5.2.3 Bivalves are typically shipped in moist seagrass or newspaper and can be induced to spawn without a formal acclimation period.

5.2.3.1 If any acclimation period occurs, holding would take place in clean natural sea water (SPP control/dilution water) using flow
-through conditions.

5.2.4 Pretest observation data concerning the source, handling procedures, disease treatment (if any), health, feeding and mortality of test animals will be documented and reported as necessary.

5.2.5 The following table provides a summary of recommended temperatures (°C) for testing selected bivalves.

Species	Testing Temperature
M. edulis	16 ± 2°C
M. galloprovincialis	16 ± 2°C
M. mercenaria	25 ± 2°C
C. virginica	25 ± 2°C
M. lateralis	25 ± 2°C

5.3 Collection of Embryos

Several options are available for collection of gametes from the bivalves. Unless otherwise requested, ESI will utilize temperature change to stimulate spawning. Other options include injection of potassium chloride and addition of heat killed sperm to the culture water. Sperm suspension should be placed adjacent to the incurrent siphon. Use of heat killed sperm works best with temperature manipulation, and should be applied 1-2 hours following the temperature change.

In order to minimize potential effects associated with genetic variation in the adult population, tests should be conducted using gametes from three (3) male/female pairs of adults. Embryos from each adult pair should be mixed equally for the final pool of embryos used for testing.

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5.3.1 Induction of Spawning using Temperature Change

Select an appropriate number of adult organisms from the available pool, the larger the number the greater the chance of successful gamete production/spawning. Place the adults, either singularly or in small groups in glass bowls or baking dishes at the conditioning temperature. Salinity of the water in the containers should be 30‰. Once organisms are pumping, bring the temperature up to the spawning temperature (5 - 10°C above the conditioning temperature) over a 5 minute period. This is best accomplished placing the spawning chamber in a water bath with a higher temperature. Once the desired temperature is achieved, remove the spawning chamber and maintain it at the desired level. If spawning does not start within 60 minutes, drain the warm water from the container, replace it with water from the conditioning tanks, and repeat the process. If desired, 1 mL of 0.5 M potassium iodide solution can be injected into the posterior adductor muscle of the organism(s) immediately before being placed into a spawning chamber.

Prior to spawning, pumping rates will increase. Just prior to spawning, *Crassostrea* will clap their shells together quickly one or two times, while clams will extend their siphons. Spawning will be evidenced by the presence of a whitish stream of gametes into the spawning chamber. Once spawning has started, remove other individuals to a different chamber to prevent fertilization or mixing of gametes.

5.3.2 Once spawning has started, eggs and sperm should be collected and fertilization success checked. After collection of the gametes, one drop each of eggs and sperm are mixed together to determine if fertilization will occur. The fertilized eggs are checked approximately five minutes later under a compound microscope to determine if the fertilization event was successful. Successful fertilization will be observed as a perfect transparent ring around the egg. Greater than or equal to 90% of the mated subsample (egg+sperm) must be fertilized to be used in testing. If <90% of the eggs are fertilized, then a new male/female pair may be injected and a mated subsample observed.

5.3.3 Viable sperm are diluted in natural seawater water to obtain an approximate concentration of $1X10^5$ to $1X10^7$ sperm/mL (see Table 1: Sperm Dilutions). Viable eggs are diluted in natural seawater so that the egg concentration is approximately 40-50 eggs/mL (see Table 2: Egg Dilutions). At the end of this document is an example worksheet detailing embryo preparation prior to addition to test solutions.

5.3.4 Sperm are added to eggs, and fertilization success is checked after 15 minutes exposure. Successful fertilization will be observed as a transparent ring around the egg. Fertilization should be accomplished at or just below the spawning temperature. Once fertilization is confirmed, embryos should be held at 20±1°C and must be added to test solutions within 4 hours of fertilization. After fertilization is confirmed, the embryo concentration (embryos/mL) is determined using a Sedgwick-Rafter counting cell and compound microscope.

The Sedgwick-Rafter counting cell provides a known volume (1 mL) and is used to determine egg densities. Place the counting cell on the microscope stage. Thoroughly mix the embryo suspension with a perforated plunger and remove a 1 mL aliquot with

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a macro pipette. Place the tip of the pipette in the counting cell and dispense the solution into the cell.

Using the stage micrometer, move the cell back and forth and side to side counting all eggs until the entire area of the cell is covered. This process is to be conducted on three subsamples and the mean, in embryos/mL, is calculated.

5.4 Exposure Conditions

Exposure conditions and monitoring requirements detailed in the following paragraphs are the minimum requirements specified by ESI for this assay.

5.4.1 Embryos are exposed to elutriate solutions and control treatments for 96 hours under static non-renewal conditions.

5.4.1.1 Treatments include a laboratory control (0%), SPP elutriate solution (100%), and elutriate solution diluted to 50%, 10% and 1% concentrations.

5.4.2 The laboratory control water is natural seawater obtained from the Hampton-Seabrook estuary. Water may be passed through a ≤50 µm filter to remove debris and provide continuous aeration. Salinity of the water should be 30±2 ppt. If the seawater salinity is below 28 ppt, then the sample is adjusted with EDTA free commercially available marine salts.

5.4.2.1 Alternate dilution water may be; modified GP2, Forty Fathoms® or equivalent, artificial seawater prepared with Milli-Q® water or equivalent DI water.

5.4.3 Water samples are monitored for salinity, dissolved oxygen, specific conductance and pH before starting the assay. If dissolved oxygen levels are below 60% saturation, samples may be aerated prior to the addition of embryos. If aeration is necessary, all replicates must be aerated. Samples must not be aerated after the addition of the embryos.

5.5 Study Conduct

Each treatment group will consist of 5 replicates. Test vessels are 250 mL glass beakers containing 200 mL of solution. Test beakers and data sheets are clearly labeled with client name and ESI number, and arranged in a logical manner.

5.5.1 Sample treatments are measured using graduated cylinders and 200 mL of solution is added to each vessel. Salinity, pH, dissolved oxygen and temperature are measured in one replicate of each test treatment before embryos are added, and at the end of the assay, once embryo samples have been collected. Ammonia may be measured in the sample and control treatments at the start and end of the assay if specified in the project scope of work.

5.5.2 Plunge the embryo suspension periodically to keep the embryos from settling. One hour after adding the sperm suspension to the egg suspension, determine the concentration of embryos in the embryo suspension by mixing the solution with a perforated plunger, withdrawing a 1 mL sample, placing it in a Sedgewick-Rafter cell, and counting the number of embryos that have developed to a 2 cell stage or beyond.

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The concentration of embryos in the test solutions should be between 20 and 30 embryos/mL. Although concentrations up to 100 embryos/mL do not impair normal development of Pacific oyster embryos, the other three species noted develop abnormally at concentrations above 30 embryos/mL.

5.5.3 A minimum of three additional laboratory diluent controls are prepared for the assay. These controls, called surrogates, are used to determine the initial concentration of embryos in test solution, and also to determine when the embryos have reached the straight hinge prodissoconch stage, the stage at which the assay is terminated.

The transfer of embryo suspension to these vessels should be ordered so that one surrogate receives the embryo suspension at the start, one in the middle and one at the end of the series. Test replicates are not randomized during testing; rather, embryo suspension is added at test initiation by replicate across test solutions in an alternating fashion (alternating allocation).

5.5.4 Within 4 hours of fertilization, sufficient embryo suspension is added to the test treatments to yield approximately 4000 to 6000 embryos per 200 mL sample (20 to 30 embryos/mL). The maximum loading rate shall not exceed 30 embryos per mL.

Prior to transferring the embryos from the embryo suspension to the test chambers, homogenize the distribution of embryos in suspension by mixing the solution with a perforated plunger. The plunger may be constructed by drilling holes in a disc of acrylic plastic or fiberglass of suitable diameter and attaching it to a PVC or acrylic rod of suitable length. The diameter of the disc should be just small enough that the plunger moves freely up and down in the test vessel. After mixing, an aliquot will be removed with a calibrated pipette. The volume of embryo stock solution added to the test chamber should not exceed 2 mL, or 1% of the treatment volume, and is determined by the embryo density.

5.5.5 The density of the embryos in the test solution at the start of the assay will be confirmed by measurement of embryo density in a minimum of three of the surrogate test chambers. Bivalve larvae initial densities must be recorded at the start of the test.

Test chambers will be placed in an incubator set at the test temperatures specified in Section 5.2.5. Mean temperatures for the assay will be ±3°C of the target test temperature.

5.5.6 The exposure period will be between 48 and 72 hours. The development of the embryos in the surrogate test vessels will be observed at 48 hours. Once 90% of the surrogate embryos have reached the straight hinge prodissoconch I (D-shape, see Figure 1 for illustrations of normal and abnormal development), the test may be terminated. The total incubation time will be recorded.

Final observations must be made of at least 100 organisms per replicate (if available) after 48 or 72 hours. If mortality in an exposure chamber prevents 100 embryos from being evaluated, observations should be made on a volume of SPP that would be expected to yield 100 embryos had mortality not occurred (based on controls or the pretest chambers stocking information). The test is terminated when 90% of the larvae in the control treatment reach the prodissoconch I (D shape) stage of development.

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Determination and confirmation of survival counts must be made prior to preservation of organisms upon test termination.

5.5.7 After the required incubation period, each test chamber is homogenized using a perforated plunger and, immediately after plunging, 5 mL of embryo/test solution is removed from each chamber using a macro pipette and transferred to a 20 mL scintillation vial. Using a pipette, 200 µL of formaldehyde solution is added to each vial before capping.

5.5.8 Within 48 hours of preservation, the entire population of straight hinge prodissoconch I larvae in each 5 mL replicate are counted. The number of normally and abnormally developed embryos will be recorded separately. Several techniques can be used for counting. One option for accurate enumeration is as follows:

5.5.8.1 A disposable petri dish cover (47 mm) is divided into 6 sections by drawing 5 parallel lines on the outside with a fine-tipped permanent marker. Place the cover on the stage of a dissecting microscope at 10x to 20x magnification.

5.5.8.2 Transfer the entire contents of the scintillation vial to the petri dish. Rinse the inside of the vial twice with 1 to 2 mL portions of DI water. The petri dish has sufficient volume to hold the 5 mL plus the washings.

5.5.8.3 Let the embryos settle for 5 to 10 seconds and then count embryos in each row by moving the dish across the field under the microscope. Actual counting time for each vial will vary with the number embryos and clarity of the sample.

5.5.8.4 As there are many subtle differences in development of embryos, it is suggested that one analyst make all of the final counts and observations. If more than one analyst must participate, normal and abnormal developmental morphologies must be agreed upon in advance.

5.5.9 Final observations for bivalve larvae should include survival, normal “D”-shaped shell, misshapen shell, empty shell (no meat), and trocophore stage. Misshapen shells and trocophores are included as alive for the survival endpoint, however, only larvae with normal “D” shell shape.

5.6 Calibration and standardization are not applicable components of this method and procedure.

6.0 Quality Control Requirements

6.1 For the assay to be considered successful, conditions must meet those specified in Section 11.0: "Summary of Test Conditions".

6.2 Test acceptability criteria requires that water quality parameters fall within the ranges specified. Mean survival and normal development rate in the laboratory control treatment must be ≥70%. If control mean survival and normal development is <70%, the client should be contacted immediately as the affected test(s) may have to be repeated. The client may choose to contact the New York District. Any delays in contacting the client may lead to problems meeting holding times if a retest is required.

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6.3 A reference toxicant test shall be conducted each month when the assay is performed on a regular basis. The assay can run concurrent with a client assay. If the assay is not conducted during a particular month, no reference toxicant assay needs be conducted. If the assay is run sporadically, then the reference toxicant is run concurrently. The reference toxicant used is ammonia (NH3). For more information see SOP QA-1114 “Conduct of Reference Toxicant Assays”.

6.4 The salinity and pH of natural seawater obtained from the Hampton-Seabrook Estuary and used for laboratory salt water are monitored regularly. On a quarterly basis, water quality analyses are performed to monitor the following parameters: total mercury, total metals, pesticides, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners.

6.4.1 Saltwater is also tested annually for all parameters listed under ASTM 2002a.

6.5 It is crucial to refer to each project’s scope of work before the beginning of testing to ensure that all individual requirements are satisfied.

6.6 It is imperative that all materials (i.e. data sheets) are reviewed on a daily basis by personnel performing the assay to ensure that all applicable data are available and accurate. See Figure 2 for an example of the embryo worksheet.

6.7 When adding solutions to test vessels during study conduct, ensure volumes in each replicate are equal across test concentrations. Inconsistencies in solution volume may attribute to variability in final embryo counts.

6.8 Interferences

The most likely source of interference for this assay is the presence of predatory organisms in the sample or dilution water. Water samples should be visually inspected prior to use and, if necessary, filtered through a fine mesh screen to remove any organisms. The size of the mesh used as a filter shall be recorded on the Embryo Bench Sheet or elsewhere.

6.9 Detection limit is not an applicable component of the quality control requirements for this method and procedure.

7.0 Calculations and Reporting

7.1 Calculations

Percent survival and percent normal development are calculated as follows:

Average Surrogate Count = Start Exposure Count

[Survival Count] / [Start Exposure Count] = % Survival

[Normally Developed Count] / [Start Exposure Count] = % Normal Development

Note that if the “Normally Developed Count” is greater than the “Start Exposure Count”, then the “Normally Developed Count” is substituted as the “Start Exposure Count” for the applicable replicate.

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7.2 Data Analysis

Survival and development data from each treatment will be subjected to calculation of LC-50 and EC-50 values using linear interpolation or trimmed Spearman-Karber methods. Statistical evaluations will be made with CETIS® software.

7.2.1 Prior to statistical analysis, the survival and development data will be reviewed to determine the presence of outliers. If outliers are found, an explanation must be sought. If a reasonable source for the deviation is found, the value may be excluded from further analysis. If no explanation is found, the analysis should be performed both with and without the questionable data point and both sets of results reported. All data sets are evaluated to determine sample variance homogeneity and normality. Those data sets meeting the criteria for normality and homogeneity are evaluated with parametric statistical models, while those that do not meet both criteria are evaluated using non-parametric models. See SOP QA-1320 “Statistical Analysis of Acute and Chronic Exposure Bioassay Data”.

7.2.2 Endpoints to be evaluated are survival and normal development.

7.2.3 Statistical comparisons for each sample site will be made against the laboratory control treatment plus each project reference site, or as specified. Project reference sites may also be compared to one another.

7.2.4 The primary statistical methods for computation of the LC-50 and EC-50 values with 95% confidence limits are the Spearman-Karber and Probit methods. The LC-50 value with 95% confidence limits are graphically displayed. If survival/development in the highest test concentration is >50%, the LC/EC-50 value will be determined by direct observation of the raw data and presented as >100%.

7.3 Reporting

Reports generated from test data will include a body of text describing the methods and materials used in the assay, discussion of results and general findings, and variation from the proposed work scope. Survival and water quality data, results of statistical analysis, and results of chemical analysis will be presented and will be paginated in tabular form. Copies of statistical data and of all bench sheets will be included in an appendix. See SOP QA-1119 “Data Review and Reporting” for specific details.

8.0 Corrective Actions

8.1 If any parameter fails to meet the criteria specified in the "Summary of Test Conditions", the Laboratory Manager must be notified immediately.

8.2 If the results of the reference toxicant assay are outside two standard deviations of the historic laboratory mean, then the Laboratory Manager must be notified immediately.

8.3 In the event that an element of the assay falls outside acceptable limits, or there is a change in the protocol, a Corrective Action Report (CAR) must be initiated and completed. See SOP QA-

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1134 “Corrective Action Report and Client Comment/Complaint/Compliment Tracking” for CAR guidance.

8.4 If a spawning event does not result in successful fertilization, then new organisms must be spawned until fertilization is achieved.

9.0 Health and Safety

9.1 Formalin, which contains formeldahyde, is a known teratogen. It can be irritating to the skin, eyes, digestive tract, or respiratory tract. Ingestion or inhalation can also be fatal. Be extremely careful and refer to the MSDS when working with this chemical. Gloves and safety glasses should be worn when handling sediment samples and chemicals. It is advisable to wear a lab coat to protect clothing.

9.2 At the end of the process, excess sample material and material used in the collection process will be disposed of appropriately. Material may be returned to the client, air dried and placed in an appropriate container for disposal at an approved disposal facility or, in the case where the material is non-hazardous, the material may be disposed in an appropriate waste container.

9.3 Sample disposal will be conducted using procedures to minimize potential pollution of surface and ground waters, soils and sediments.

10.0 Responsibilities

10.1 It is the Laboratory Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedures outlined in this SOP.

10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification will include reading this and associated SOP’s, review of the primary literature, and participation in similar procedures under the direct supervision of a trained staff member. Certification will be based upon a review of the analysts’ demonstrated abilities.

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11.0 Summary of Test Conditions
(Unless otherwise specified by client)

1. Test Mode:	Static, non-renewal
2. Salinity:	30±3 ppt
3. Temperature:	16±2°C
4. Photoperiod:	16 hour light, 8 hour dark
5. pH:	7.8±0.5 (target)
6. Light Source:	Ambient laboratory illumination
7. Test Chamber:	250 mL glass beakers (minimum)
8. Solution Volume:	200 mL/replicate (minimum)
9. Embryo Concentration:	20 - 30 embryos/mL
10. Replicates/Treatment:	5
11. Organisms/Chamber	$N = S(V_s/V_t)$, where: N = embryo density in test chamber S = mean embryo density in stock suspension V_s = volume of stock added to test chamber V_t = total volume of test solution Embryos per test chamber divided by embryos per mL equals mL of stock per chamber
12. Treatments:	Laboratory control, undiluted elutriate solution and diluted elutriate solution (concentrations 50%,10% and 1%)
13. Age of Embryos:	Use embryos within 4 hours of fertilization
14. Feeding Regime:	None during test as uneaten food might decrease DO and biological activity of some test materials, and the embryo/larvae can survive without feeding for 72+ hours.
15. Test Solution Aeration:	None, since bubbles can collect within larval mantle cavity. Aerate only if DO falls below 60% saturation.
16. Dilution Water:	Clean natural seawater, alternatives include Modified GP2, Forty Fathoms® or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water.
17. Test Duration:	48 - 72 hours development into straight hinge prodissoconch larvae I (D-shape stage).
18. DO:	DO concentrations in each test chamber must be between 60-100% saturation at all times
19. Test Acceptability Criterion:	The number of embryos that result in live larvae with completely developed shells at the end of the control (i.e. 0%) test must be at least 60% of the initial number with at least 90% of all introduced embryos alive at the end of test.
20. Endpoint:	Mortality (LC50) - mortality / abnormality (EC50)
21. SPP Holding Time:	Use within 24 hours of preparation

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TITLE: Acute Toxic Effects of Waters on the Development of Marine Bivalve Larvae, based on Region II Guidelines

Table 1. Sperm Dilutions

Note that the sperm must be used within one hour of collection. Add 30 ± 3 ppt seawater to the cup of collected sperm to "mildly" dilute. The cup of mildly diluted sperm is considered vessel "A" and will have a final concentration count of approximately 5 X 10^7 sperm/mL. The goal is to obtain a final concentration of approximately 1 X 10^7 sperm/mL in vial "D" (110-130 hemacytometer count).

Step 1: Add 30 ± 3 ppt seawater at 16 ± 2°C to vials as follows:

Vial "B" - 5 mL
Vial "C" - 5 mL
Vial "D" - 4 mL

Step 2: Dilute the sperm concentration further:

Remove 5 mL from "A" and add to "B". Cap and mix gently.
Remove 5 mL from "B"; add to "C". Cap and mix gently.
Remove 5 mL from "C"; discard.

Step 3: Add 5 mL of acetic acid to vial "C". Cap and mix gently.

Step 4: Add 1 mL from vial "C" to vial "D". Cap and mix gently.

Add a subsample of vial "D" to hemacytometer and let settle for 15 minutes. Take counts of 400 central squares on both sides of the hemacytometer. Average the two counts.

Step 5: Calculate:

Sperm in Vessel "A" = 40 x Sperm in Vial "D"
Sperm in Vial "B" = 20 x Sperm in Vial "D"
Sperm in Vial "C" = 5 x Sperm in Vial "D"

The sperm in vial "D" = Average hemacytometer count x 10^4

Step 6: Add 0.15 x the volume of egg suspension of the appropriate diluted sperm to the egg suspension.

Table 2. Egg Dilutions

Eggs should be frequently dispersed (using a perforated plunger), thus preventing settling and premature death.

Step 1: Add 30 ± 3 ppt seawater at 16 ± 2°C to the 8 oz. cup containing collected eggs.

Step 2: Remove 1 mL of diluted eggs. Add to graduated cylinder and bring up to 100 mL with 30 ppt sea water. Invert to mix.

Step 3: With a macro pipette, add 1 mL of sample on a Sedgwick-Rafter counting cell.

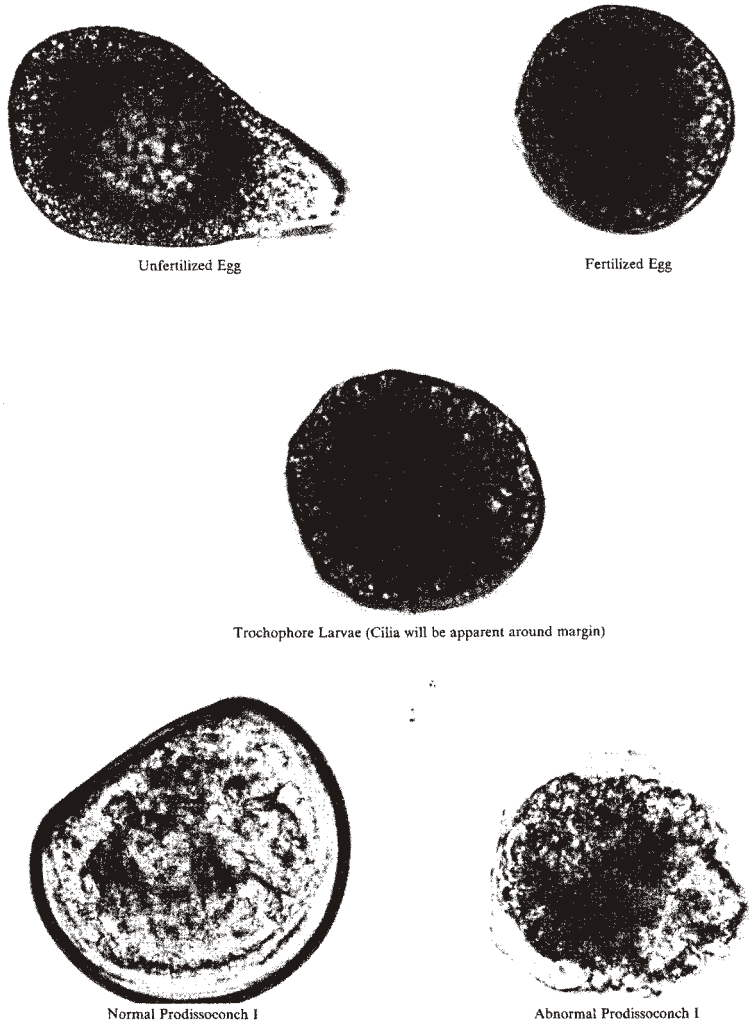
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Step 4: Count the eggs on the slide using 40x magnification.

Step 5: The egg concentration should be about 40 to 50 eggs/mL.

Step 6: Continue diluting and counting until the appropriate egg concentration has been achieved, *i.e.* 40 to 50 eggs /mL in the 100 mL container.

Figure 1. Drawings Exemplifying Key Developmental Stages of Normal and Abnormal Bivalve Embryonic Development During the First 48 hours of Development



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TITLE: Acute Toxic Effects of Waters on the Development of Marine Bivalve Larvae, based on Region II Guidelines

Figure 2. Embryo Worksheet

EMBRYO WORKSHEET

DATE: ESI STUDY:

CLIENT: PROJECT:

Eggs Collected @: Pre-assay fertilization check: ANALYST:

Sperm Collected @: *A mated subsample of egg+sperm must achieve ≥90% fertilization in order to be used in testing.*

Egg Stock Suspension Count:

Take 1mL of egg stock suspension and using a glass graduated cylinder dilute with seawater to a final volume of 100 mL. Count a subsample of the diluted suspension to get an estimate of the egg concentration. The diluted suspension should have 40 - 50 eggs/mL, (which would represent an egg concentration of 4,000 - 5,000 in the stock suspension.)

Egg Count (per mL) of diluted suspension:

Sperm Stock Suspension Count:

Once added to the egg stock, the final sperm concentration should be $1x10^5$ - $1x10^7$ in solution D.

1. Hemocytometer Count (D):
2. Hemocytometer Count (D):

Average Count (D): X10^4 = spm solution D =

Sperm Concentrations:

Solution D X 40 = Solution A =

Solution D X 20 = Solution B =

Solution D X 5 = Solution C =

Sperm Count (per mL):

mL of Eggs to Add:

mL of Sperm to Add: Gametes mixed @:

Gametes must be mixed within 1 hour of collection.

Calculated Embryo Stock Concentration (per mL):

The test concentration should be 20 - 30 embryos per mL.

Calculated Embryo Stock (mL) needed per chamber:

Add calculated amount of embryo stock to a surrogate chamber, gently mix, then count a 5mL aliquot.

Embryo Concentration Check: *If the check concentration is acceptable, then proceed with embryo addition to the test.*

Volume Embryo Stock (mL) added to test solutions: Embryos Added to Test Solutions @:

INITIAL COUNTS: Embryos/ 5 mL

SURROGATE A
SURROGATE B
SURROGATE C
Mean:

Organism Lot ID:

Mean per mL:

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Appendix E Checklists for Biological and Chemical Data Reporting Requirements

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APPENDIX G: Checklist –Biological Data

FORMAT AND CONTENT FOR BIOLOGICAL DATA PACKAGES
NYD AND EPA REGION 2 DREDGED MATERIAL PROGRAM

1.0. Summary Tabular Data and Project Narrative

TEST PERFORMANCE SUMMARY
A summary table listing the percent survival in all control, reference, and test samples.

STATISTICAL ANALYSIS SUMMARY
A summary table containing the LC/EC_{50} values for the suspended particulate phase (SPP) tests and t-tests from the solid phase tests

DEVIATIONS/QUALIFIER PAGE
A narrative which summarizes all of the deviations from the Green Book and Regional Guidance Manual protocols. Deviations of sample handling, test conditions, ammonia purging procedures, control performance, reference toxicant test performance, organism handling/acclimation, and water quality parameters should be provided in this section.

SAMPLING SUMMARY
A summary table which documents collection dates and holding times for the test, control, and reference sediment samples. Holding times for site water, SPP, and lab saltwater for all tests should be included in this table.

REPORT NARRATIVE
The data narrative should describe the major biological project activities and results. Computerized tables of results, water quality, and other pertinent information should be placed in this portion of the biological data package.

2.0 General Data Reporting Requirements

RAW BIOLOGICAL AND WATER QUALITY DATA FROM TESTS	
	Survival and bivalve development data
	Water quality parameters
	Feeding schedule and amount (if applicable)
	Organism observations
	Summary of test conditions
	Project notebook/log entries and deviations from protocol
TEST ORGANISM HOLDING, HANDLING AND ACCLIMATION	
	Organism shipping data sheet provided by supplier
	Copy of overnight shipping airbill (if applicable)
	Internal receiving and distribution data
	Holding/acclimation records (incl. water quality, renewals, and feeding)
	Mortality during holding and acclimation
	Taxonomic identification for each species
	Feeding records
REFERENCE TOXICANT DATA	
	Raw bench sheets for reference toxicant tests
	Reference toxicant stock & test solution preparation sheet
	LC/EC_{50} statistical calculations
	Updated reference toxicant control charts w/ acceptability limits
STATISTICAL DATA FROM DREDGED MATERIAL TESTS	
	Provide all computer generated LC_{50}, EC_{50} and/or t-test spreadsheets or graphical interpolations for the SPP, solid phase tests, and t-tests comparing test and reference tissue chemistry.
INVALID TEST DATA	
	If a test was repeated for any reason, then the data from the original test must be included in the final report. If a serious deviation occurs which has the potential to affect test acceptability, then NYD and EPA Region 2 must be contacted immediately to determine if a retest is needed (to avoid missing established holding times)

3.0 Test Specific Information (additional to items specified in 2.0)

	AMPHIPOD SOLID PHASE TEST
	Pretest overlying water renewal log and total porewater ammonia data
	Total/un-ionized porewater ammonia measured in surrogate jars during testing
	MYSID SOLID PHASE TEST
	Pretest overlying water renewal log and total porewater ammonia data
	Total/un-ionized overlying un-ionized ammonia measured during testing
	SUSPENDED PARTICULATE PHASE TESTS (SPP)
	SPP preparation log (all volumes, mixing times, centrifuge info. etc.)
	Raw data for bivalve gamete collection and preparation
	BIOACCUMULATION TESTING
	Pretest ammonia purging data (Static Renewal)
	Flow calibration log – Initial and final adjusted flows (Flow-throughs)
	Dates and times of water removals and renewals (Static Renewal)
	Description of Siphon and Renewal Techniques (Static Renewal)
	Pre- and post-test depuration logs – time started/ended and flow rates
	Receiving logs for all natural saltwater (if collected)
	Preparation logs for all artificial saltwater
	If control survival <90%, provide DETAILED narrative for the 5 factors
	Raw statistical data comparing test and reference tissue chemistry
	SAMPLING / SAMPLE HANDLING
	Chain of custody forms for all test, control, and reference samples
	Field data sheets and/or sampling Logs (incl. photos if available)
	Log of test sediment composite preparation
	Sieving – size of mesh used , other sample preparation procedures/notations
	Holding times for all samples (test, reference, control, elutriate, lab saltwater)

_____ _____

Signature of Laboratory Representative Date

The above signature indicates that the contracted laboratory has reviewed the data package deliverables and the data/QC summary tables and acknowledges that all of the information included in this checklist has been provided in this submittal.

APPENDIX H: Checklist – Chemical Data

FORMAT AND CONTENT FOR CHEMICAL DATA PACKAGES
NYD AND EPA REGION 2 DREDGED MATERIAL PROGRAM

MATRIX (circle one): Sediment Tissue Water/Elutriate

(To be completed for each matrix)

Custody Information - Laboratories should ensure that sample custody has been maintained.

_____Custody seals or tape
_____Sample receipt form (including date and time rec'd at laboratory/cooler temperature)
_____Chain of custody record
_____Laboratory sample login report

Project Analytical Data Narrative - Discuss compliance and noncompliance with all analytical and QC requirements listed in the RTM. Provide a narrative for each of the following headings.

_____Project name
_____Parameter analyzed
_____Name of laboratory performing the analysis
_____Sample custody and processing
_____QA/QC data quality indicators/objectives
_____Analytical method summary (including any deviations, modifications or observations)
_____Holding times are specified
_____Detection limits
_____Method blanks
_____Replicate precision
_____Laboratory control sample
_____Matrix spike (MS)
_____Standard reference material (SRM)
_____Data validation (statement and signature indicating that the data was reviewed/validated)
_____Problems/corrective actions (if any)

Analytical Data Reporting

_____Names of laboratories performing the analyses
_____Name of laboratory contact
_____Table of contents
_____Raw and reduced data
_____Sampling logs/field data sheets copies of log books etc.
_____Sample tracking and traceability (from sample collection to disposal and/or archive)
_____Initial and continuing calibration checks
_____Data/QC summary tables for both wet and dry weight

A) Table Headings

_____Laboratory name (performing the analyses)
_____Project name
_____Project number
_____Analyses and medium (e.g., metals in site water and elutriate)
_____For tissue data, specify type (*M. nasuta or N. virens*)
_____Sample Identification Number (traceable to the COC documentation. Samples must be traceable from laboratory to laboratory using the same sample ID number)
_____Date sample was prepared/extracted and analyzed
_____Summary of sample weights

B) Reports results using the appropriate units.

____Metals in tissue (ug/g) wet weight and dry weight
____Metals in water (ug/L)
____Metals in sediment (ug/g) dry weight
____Organics in tissue (ng/g) wet weight and dry weight
____Organics in water (ug/L)
____Organics in sediment (ng/g) dry weight
____Dioxins/furans in tissue (pg/g) wet weight and dry weight
____Dioxins/furans in sediment (ng/g) dry weight

C) Quality Assurance and Quality Control

1) Matrix Spike Triplicate Samples Analyzed

____Sample weight/volume
____Unspiked sample results
____Spiked sample results
____Concentration of spike level used and amount of spike added
____% recovery for each analyte

2) Standard Reference Material Analyzed (if applicable)

____Sample weight/volume
____SRM contains relevant of the analytes to be tested
____Manufacturer and catalog # of SRM
____Copy of certificate of analysis (certified or consensus value)
____Results of the SRM samples
____% recovery calculated for each analyte

3) Internal/Surrogate Standard Analyzed
____Results reported for each sample analyzed
____% recovery for each internal/surrogate standard

_____ _____
Signature of Laboratory Representative Date

The above signature indicates that the contracted laboratory has reviewed the data package deliverables and the data/QC summary tables and acknowledges that all of the information included in this checklist has been provided in this submittal.

Appendix F USACE and EPA Sampling and Testing Requirements

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US ARMY CORPS
OF ENGINEERS
NEW YORK DISTRICT

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2

DATE: 20 November 2017

SUBJECT: SAMPLING AND TESTING REQUIREMENTS FOR

APPLICATION No. NAN-2016-00908-EHA

APPLICANT: Transcontinental Gas Pipe Line (TRANSCO)

WATERWAY: Lower New York Bay

The New York District Corps of Engineers is submitting the attached draft sampling scheme and testing requirements for EPA Region 2's review and concurrence.

Preparer: Kelly Vega

Approving Supervisor: Chris Sandy

DATE: 11/28/17

The US Environmental Protection Agency has reviewed the subject sampling scheme and testing requirements.

☒ EPA concurs with sampling scheme and testing requirements as submitted.*
* See attached Memo

☐ EPA has modified the sampling scheme as indicated.

☐ EPA has modified the testing requirements as indicated.

Reviewer: Matthew M. Dias

Approving Supervisor: Alan Steinberg

MEMORANDUM

TO:	File: Transcontinental Gas Pipe Line (TRANSCO)
FROM:	Kathryn Drisco
DATE:	11/28/17
SUBJECT:	Sampling Scheme

EPA has determined that prisms A-1 and A-2 do not meet all the requirements for exclusionary criteria stated in 40 CFR 227.13(b)(1). To fully satisfy the requirements of 40 CFR 227.13(b)(1), the proposed dredged material must be found in areas of high current or wave energy such as streams with large bed loads or coastal areas with shifting bars and channels. The areas where prisms A-1 and A-2 are located do not meet this condition.

40 CFR 227.13(b) states:

"(b) Dredged material which meets the criteria set forth in the following paragraphs (b)(1), (2), or (3) of this section is environmentally acceptable for ocean dumping without further testing under this section:

(1) Dredged material is composed predominantly of sand, gravel, rock, or any other naturally occurring bottom material with particle sizes larger than silt, and the material is found in areas of high current or wave energy such as streams with large bed loads or coastal areas with shifting bars and channels; or

(2) Dredged material is for beach nourishment or restoration and is composed predominantly of sand, gravel or shell with particle sizes compatible with material on the receiving beaches; or

(3) When:

(i) The material proposed for dumping is substantially the same as the substrate at the proposed disposal site; and

(ii) The site from which the material proposed for dumping is to be taken is far removed from known existing and historical sources of pollution so as to provide reasonable assurance that such material has not been contaminated by such pollution."

In order for EPA to determine if the dredge material in prism A-3 meets the exclusionary criteria, TRANSCO will need to provide acceptable grain size data, from the locations specified in the sampling plan, and any relevant information, data, modelling, etc. that demonstrates that the bottom conditions are met.

SAMPLING SCHEME and LIST of REQUIRED TESTING as OUTLINED in the 2016 ACENYD/EPA REGION II REGIONAL IMPLEMENTATION MANUAL on Dredged Material Proposed for Ocean Disposal and the 1991 GREEN BOOK

Applicant Transcontinental Gas Pipe Line Company, LLC (TRANSCO)

Applicant No. NAN-2016-00908-EHA

Address 2800 Post Oak Boulevard, P.O. Box 1396, Houston, TX 77251-1396

Waterway Lower New York Bay

Proposed Volume:

Area A-1:	35,950	cubic yards	(based on 5/4/2016 – 9/14/2016	bathymetric survey)
Area A-2:	24,450	cubic yards	(based on 5/4/2016 – 9/14/2016	bathymetric survey)
Area A-3:	32,450	cubic yards	(based on 5/4/2016 – 9/14/2016	bathymetric survey)
Area C:	64,050	cubic yards	(based on 5/4/2016 – 9/14/2016	bathymetric survey)
Area D:	33,750	cubic yards	(based on 5/4/2016 – 9/14/2016	bathymetric survey)
Area E1:	63450	cubic yards	(based on 5/4/2016 – 9/14/2016	bathymetric survey)
Area E2:	63450	cubic yards	(based on 5/4/2016 – 9/14/2016	bathymetric survey)
Area F:	67750	cubic yards	(based on 5/4/2016 – 9/14/2016	bathymetric survey)
Area G:	14050	cubic yards	(based on 5/4/2016 – 9/14/2016	bathymetric survey)

*The depths of each Area (aka. Prism) are set based on anticipated burial depth and backfill requirements. The areas are not defined to specific depths below Mean Lower Low Water (MLLW) because of (a) changes in the sediment surface, (b) changes in geometry required to bury the pipe and account for side sloping, and (c) differing burial depth requirements for certain use areas (e.g. anchorage areas and navigation channels.

NOTE:. Since the sediment surface is variable, Transco plans to bury the pipeline to a certain depth which will change over the route and this is referred to as the "design depth". The applicant is required to collect cores from each sampling location to design depth plus 1 ft overdepth.

Applicant will be required to submit profiles of each sample location on engineering size bathymetric survey's indicating the design depth of each location prior to sampling.

A preapplication meeting X was held on 7/25/2017 at NY District Offices in New York

____ was not held at the NY District Offices in New York.

The applicant has indicated that testing will be performed by the following laboratories:

Biological: N/A Analytical: N/A Dioxin: N/A

The sampling scheme and required testing described on pages 2-3 were approved for the proposed project area based upon the information contained in the attached map.

Sample dredging sites and reaches are indicated on attached map.

The proposed dredging area is subdivided into NINE areas for sampling and testing purposes:

Area A1: 4 Locations (9, 10, 11, 12)
Area A2:: 3 Locations (24, 25, 26)
Area A3: 3 Locations (29, 30, 31)
Area C : 5 Locations (1, 2, 3, 4, 5)
Area D: 3 Locations (6, 7, 8)
Area E1: 3 Locations (13, 14, 15)
Area E2: 3 Locations (16, 17, 18)
*Area F: 5 Locations (19, 20, 21, 22, 23)
Area G : 2 Locations (27, 28)

The applicant is required to collect cores from each sampling location to design depth plus 1 ft.

***Area F consists of full lengths of cores from Prism F on engineering size survey map, dated 3 March, 2017 and from 4 feet below top of sediment to full depth in sample locations #21 and #22. The sediment in the top 4 feet of Prism B-4 will be tested for Upland Disposal (refer to hand drawn diagram with approximation of profiles for both areas). A material separation plan for Prism B-4 must be submitted by the applicant and approved by USACE and EPA, Region 2 prior to dredging.**

- The District reserves the right to require additional sampling and testing at any time.

COMMENTS:

1. If there is any evidence of stratification in core samples, contact NYD prior to compositing.

2. Additional cores may have to be taken from each station in order to ensure adequate volumes of sediment to meet testing requirements. If so, the same number of cores must be taken and composited from each location.

3. Core locations may have to be moved if shoaling patterns have changed. If so, contact NYD during sampling to confirm new locations.

4. Bioassay/bioaccumulation testing may not be necessary in Area A3, if the applicant provides acceptable grain size data from the locations specified in the sampling plan, and any relevant information, data modeling, etc. that demonstrates that the bottom conditions meet the requirements stated in 40 CFR 227.13(b)(1). It is strongly advised that the applicant perform grain size analyses on each core and submit all the information required in 40 CFR 227.13(b)(1) as soon as possible to NYD. Based on the submitted information, a determination will be made by NYD and EPA Region 2 regarding the need for further testing per the requirements in 40 CFR 227.13(b)(1).

5. Bulk sediment chemistry will not be required.

* If you have any questions regarding sampling, test protocols, test species, QA/QC, etc. please contact the Dredged Material Management Section of the USACE/NYD at (917) 790-8429 or (917) 790-8539.
** Sampling and Testing Scheme is only for S&T request submitted by letters from Transco dated through 30 October 2017 to USACE-NYD.

PREPARED BY: KELLY VEGA DATE: 04 DECEMBER 2017 PHONE: (917) 790-8429

SEE PAGES 2-3 FOR SAMPLING AND TESTING REQUIREMENTS

APPLICANT: #NAN-2016-00908-EHA – TRANSCO

Any Box Checked Off Indicates an Analysis or Assay that Is Required for a Given Project.
Archiving requires saving a sample for possible analysis at a later time pending further instruction.
X=Per Homogenized Sediment Core + Reference (Composited Grabs) + Control
C=Per Bioassay Sediment Composite
A=Archive
W=Site Water and Elutriate
T=Per Tissue Replicate (Ref, Test, Pre-test, A=Archive Ctl and any remaining tissue from Ref, Test, Pre-test)

1. SEDIMENT PHYSICAL ANALYSIS (If stratification is observed, each stratum within a core must be analyzed separately)
   a. X **Grain Size Analysis (% sand, % silt, & % clay)**
   b. X **% Moisture**
   c. C **Specific Gravity**
   d. C **Bulk Density**
   e. C **Plastic and Liquid limits (Atterberg limits)**

2. SEDIMENT CHEMICAL ANALYSIS
   REQUIRED
   a. X **% Total Organic Carbon**
   CASE BY CASE BASIS
   a. ___ **Metals (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)**
   b. ___ **PAHs (LMWs: acenapthene, acenaphthylene, anthracene, fluorene, naphthalene, phenanthrene)**
   c. ___ **PAHs (HMWs: benzo(a)anthracene, benzo(a)pyrene, benzo(g,h,i)perylene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthrene, Indeno(1,2,3-c,d)pyrene, pyrene)**
   d. ___ **Semi-volatiles (1,4 dichlorobenzene)**
   e. ___ **Pesticides (aldrin, alpha chlordane, trans nonachlor, dieldrin, p,p' and o,p' DDT/DDD/DDE, endosulfans(I,II, and sulfate), heptaclor, heptachlor epoxide)**
   f. ___ **PCBs (#8,18,28,44,49,52,66,87,101,105,118,128,138,153,170,180,183,184,187,195,206,209)**
   g. ___ **PCB coplanar (#77,126,156,169)**
   h. ___ **Dioxins/Furans (2,3,7,8 - substituted isomers, n=17)**
   i. ___ **Other:**
   j. ___ **LIS pesticides**
   k. ___ **LIS PAHS**

3. CHEMICAL ANALYSIS OF SITE WATER AND ELUTRIATE
   REQUIRED
   a. W **Metals (Ag, Cd, Cr, Cu, Hg, Ni, Pb, Zn)**
   b. W **PCBs (#8,18,28,44,49,52,66,87,101,105,118,128,138,153,170,180,183,184,187,195,206,209)**
   c. W **Pesticides (aldrin, alpha chlordane, trans nonachlor, dieldrin, p,p' and o,p' DDT/DDD/DDE, endosulfans(I,II, and sulfate), heptaclor, heptachlor epoxide)**
   d. ___ **Other:**
   CASE BY CASE BASIS
   a. ___ **PAHs (_ all 16, _ LMW, _ HMW, _ as specified)**
   b. ___ **2,3,7,8-TCDD**
   c. ___ **2,3,7,8-TCDF**
   d. ___ **PCB coplanar (#77,126,156,169)**
   e. ___ **LIS pesticides**

d. ___ **Other:**

4. BIOASSAYS (species listed in guidance manual)
   a. C **Water Column Acute Tox.** (bivalve larvae, M. bahia, Menidia sp.)
   b. C **10-Day Benthic Acute Tox.** (A. abdita, R. abronius, E. estuarius, or L. plumulosus)
   c. C **10-Day Benthic Acute Tox.** (M. bahia)
   d. C **28-Day Bioaccumulation** (N. virens, and Macoma secta or M. nasuta) - INCLUDING DIOXIN,
   e. __ **28-Day Bioaccumulation** (N. virens and Macoma secta or M. nasuta)): DIOXIN ONLY

5. 28-DAY WHOLE-SEDIMENT BIOACCUMULATION TISSUE ANALYSIS

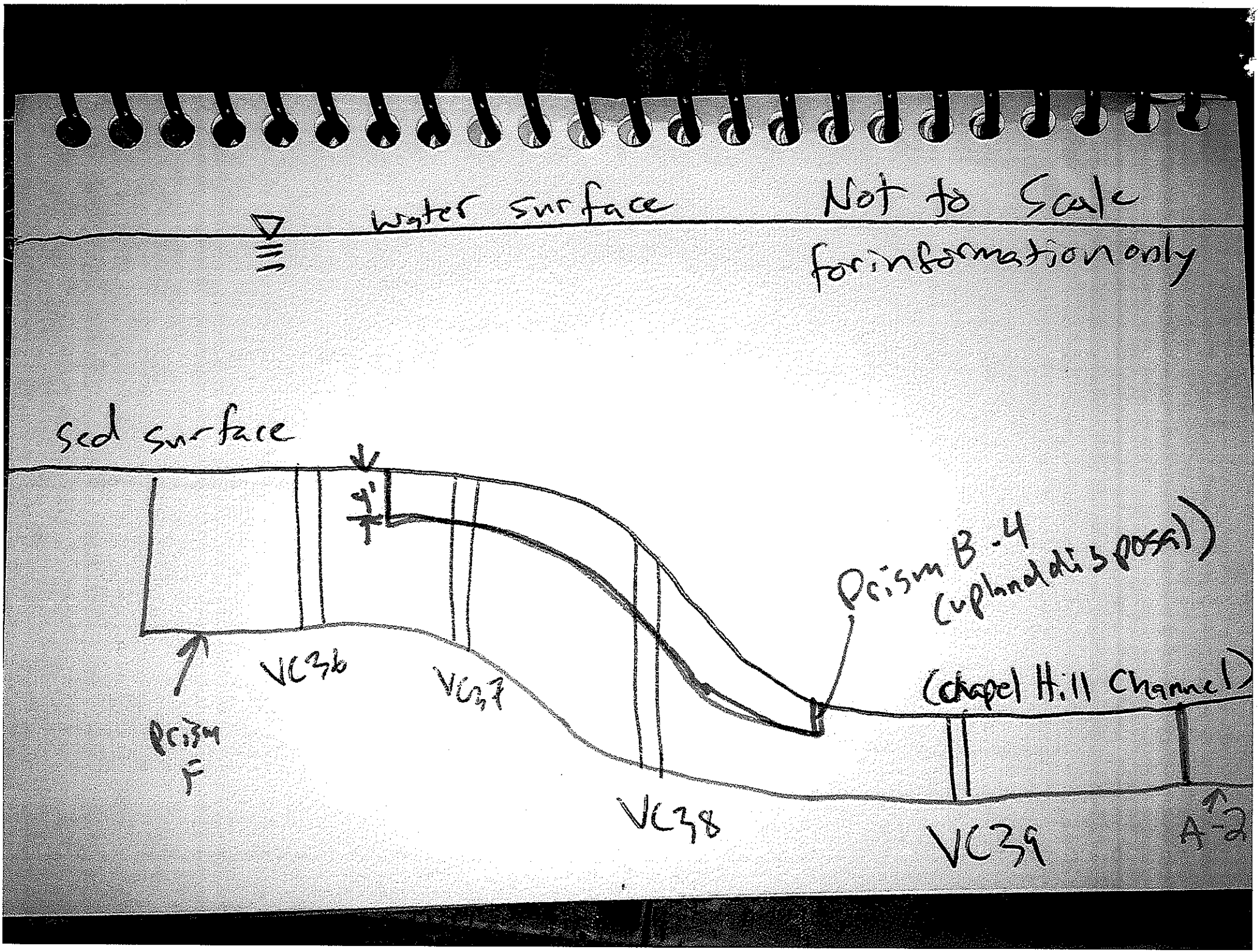
REQUIRED

   a. T A **Metals** (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)
   b. T A **Pesticides** (aldrin, alpha chlordane, trans nonachlor, dieldrin,p,p' and o,p' DDT/DDD/DDE, endosulfans(I,II, and sulfate), heptachlor, heptachlor epoxide)
   c. T A **PCBs** (#8,18,28,44,49,52,66,87,101,105,118,128,138,153,170,180,183,184,187,195,206,209
   d. T A **Semi-volatiles** (1,4 dichlorobenzene)
   e. T A **PAHs (LMWs:** acenapthene, acenaphthylene, anthracene, fluorene, naphthalene, phenanthrene)
   f. T A **PAHs (HMWs:** benzo(a)anthracene, benzo(a)pyrene,benzo(g,h,i)perylene,benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthrene, indeno(1,2,3-c,d)pyrene, pyrene)

CASE BY CASE BASIS

   a. T A **Dioxins/Furans** (2,3,7,8 - substituted isomers, n=17)
   b. ___ **LIS pesticides**
   c. ___ **LIS PAHs**
   d. ___ **PCB coplanar** (#77,126,156,169)
   e. ___ **Other:**

REFER TO COMMENT SECTION ON PAGE 2 FOR ADDITIONAL INFORMATION


water surface
Not to Scale
for information only
sed surface
9'
Pcism B-4
(uplandalis (pos?))
VC36
VC37
(Chapel Hill Channel)
Pcism
F
VC38
VC39
A-2

US ARMY CORPS
OF ENGINEERS
NEW YORK DISTRICT

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2

DATE: 8 January 2018

SUBJECT: SAMPLING AND TESTING REQUIREMENTS FOR

APPLICATION No. NAN-2016-00908-EHA

APPLICANT: Transcontinental Gas Pipe Line (TRANSCO)

WATERWAY: Lower New York Bay – Alternate (Deeper) Channel Crossing Scenario

The New York District Corps of Engineers is submitting the attached draft sampling scheme and testing requirements for EPA Region 2's review and concurrence.

Preparer: Kelly Vega

Approving Supervisor: Chris Jacobs

DATE: 1/11/18

The US Environmental Protection Agency has reviewed the subject sampling scheme and testing requirements.

- [x] EPA concurs with sampling scheme and testing requirements as submitted.
- [ ] EPA has modified the sampling scheme as indicated.
- [ ] EPA has modified the testing requirements as indicated.

Reviewer: Mark Dix

Approving Supervisor: Mark Rice

SAMPLING SCHEME and LIST of REQUIRED TESTING as OUTLINED in the 2016 ACENYD/EPA REGION II REGIONAL IMPLEMENTATION MANUAL on Dredged Material Proposed for Ocean Disposal and the 1991 GREEN BOOK

Applicant Transcontinental Gas Pipe Line Company, LLC (TRANSCO)

Applicant No. NAN-2016-00908-EHA

Address 2800 Post Oak Boulevard, P.O. Box 1396, Houston, TX 77251-1396

Waterway Lower New York Bay

Proposed Volume for **Alternate (Deeper) Channel Crossing**:

Area A-2:	82,350 cubic yards	(based on	5/4/2016 – 9/14/2016 bathymetric survey)
Area E1:	161,000 cubic yards	(based on	5/4/2016 – 9/14/2016 bathymetric survey)
Area E2:	161,000 cubic yards	(based on	5/4/2016 – 9/14/2016 bathymetric survey)
Area F:	142,550 cubic yards	(based on	5/4/2016 – 9/14/2016 bathymetric survey)

*The depths of each Area (aka. Prism) are set based on anticipated burial depth and backfill requirements. The areas are not defined to specific depths below Mean Lower Low Water (MLLW) because of (a) changes in the sediment surface, (b) changes in geometry required to bury the pipe and account for side sloping, and (c) differing burial depth requirements for certain use areas (e.g. anchorage areas and navigation channels.

NOTE:. Since the sediment surface is variable, Transco plans to bury the pipeline to a certain depth which will change over the route and this is referred to as the "design depth". The applicant is required to collect cores from each sampling location to design depth plus 1 ft overdepth.

Applicant will be required to submit profiles of each sample location on engineering size bathymetric survey's indicating the design depth of each location prior to sampling.

A preapplication meeting X was held on 7/25/2017 at NY District Offices in New York

____ was not held at the NY District Offices in New York.

The applicant has indicated that testing will be performed by the following laboratories:

Biological: N/A Analytical: N/A Dioxin: N/A

The sampling scheme and required testing described on pages 2-3 were approved for the proposed project area based upon the information contained in the attached map.

Sample dredging sites and reaches are indicated on attached map.

The proposed dredging area is subdivided into FOUR areas for sampling and testing purposes:

Area A2:: 3 Locations (24, 25, 33)
Area E1: 3 Locations (13, 14, 15)
Area E2: 3 Locations (16, 17, 32)
*Area F: 5 Locations (19, 20, 21, 22, 23)

Sample locations, 13 – 17 and 19 – 25 are the same locations as those listed in the sampling scheme dated 4 December 2017. The only difference is they will be sampled to the deeper depths for the alternate (deeper) channel crossing scenario.

The applicant is required to collect cores from each sampling location to design depth plus 1 ft.

***Area F consists of full lengths of cores from Prism F on engineering size survey map, dated 3 March, 2017 and from 4 feet below top of sediment to full depth in sample locations #21 and #22. The sediment in the top 4 feet of Prism B-4 will be tested for Upland Disposal. A material separation plan for Prism B-4 must be submitted by the applicant and approved by USACE and EPA, Region 2 prior to dredging.**

- The District reserves the right to require additional sampling and testing at any time.

COMMENTS:

1. If there is any evidence of stratification in core samples, contact NYD prior to compositing.

2. Additional cores may have to be taken from each station in order to ensure adequate volumes of sediment to meet

testing requirements. If so, the same number of cores **must** be taken and composited from each location.

3. Core locations may have to be moved if shoaling patterns have changed. If so, contact NYD during sampling to confirm new locations.

4. Bulk sediment chemistry will not be required.

5. This sampling and testing scheme is based on Transco's letter, dated December 20, 2017 and HARS Placement Information Sheet for Alternate (Deeper) Channel Crossing Scenario, dated December 18, 2017.

* If you have any questions regarding sampling, test protocols, test species, QA/QC, etc. please contact the Dredged Material Management Section of the USACE/NYD at (917) 790-8429 or (917) 790-8539.

PREPARED BY: KELLY VEGA DATE: 08 JANUARY 2018 PHONE: (917) 790-8429

SEE PAGES 2-3 FOR SAMPLING AND TESTING REQUIREMENTS

APPLICANT: #NAN-2016-00908-EHA – TRANSCO

Any Box Checked Off Indicates an Analysis or Assay that is Required for a Given Project.
Archiving requires saving a sample for possible analysis at a later time pending further instruction.

X=Per Homogenized Sediment Core + Reference (Composited Grabs) + Control
C=Per Bioassay Sediment Composite
A=Archive
W=Site Water and Elutriate
T=Per Tissue Replicate (Ref, Test, Pre-test, A=Archive Ctl and any remaining tissue from Ref, Test, Pre-test)

1. SEDIMENT PHYSICAL ANALYSIS (If stratification is observed, each stratum within a core must be analyzed separately)
   a. X **Grain Size Analysis** (% sand, % silt, & % clay)
   b. X **% Moisture**
   c. C **Specific Gravity**
   d. C **Bulk Density**
   e. C **Plastic and Liquid limits (Atterberg limits)**

2. SEDIMENT CHEMICAL ANALYSIS

   REQUIRED

   a. X **% Total Organic Carbon**

   CASE BY CASE BASIS

   a. ___ **Metals** (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)
   b. ___ **PAHs (LMWs:** acenapthene, acenaphthylene, anthracene, fluorene, naphthalene, phenanthrene)
   c. ___ **PAHs (HMWs:** benzo(a)anthracene, benzo(a)pyrene, benzo(g,h,i)perylene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthrene, indeno(1,2,3-c,d)pyrene, pyrene)
   d. ___ **Semi-volatiles** (1,4 dichlorobenzene)
   e. ___ **Pesticides** (aldrin, alpha chlordane, trans nonachlor, dieldrin, p,p' and o,p' DDT/DDD/DDE, endosulfans(I,II, and sulfate), heptaclor, heptachlor epoxide)
   f. ___ **PCBs** (#8,18,28,44,49,52,66,87,101,105,118,128,138,153,170,180,183,184,187,195,206,209)
   g. ___ **PCB coplanar** (#77,126,156,169)
   h. ___ **Dioxins/Furans** (2,3,7,8 - substituted isomers, n=17)
   i. ___ **Other:**
   j. ___ **LIS pesticides**
   k. ___ **LIS PAHS**

3. CHEMICAL ANALYSIS OF SITE WATER AND ELUTRIATE

   REQUIRED

   a. W **Metals** (Ag, Cd, Cr, Cu, Hg, Ni, Pb, Zn)
   b. W **PCBs** (#8,18,28,44,49,52,66,87,101,105,118,128,138,153,170,180,183,184,187,195,206,209)
   c. W **Pesticides** (aldrin, alpha chlordane, trans nonachlor, dieldrin, p,p' and o,p' DDT/DDD/DDE, endosulfans(I,II, and sulfate), heptaclor, heptachlor epoxide)
   d. ___ **Other:**

   CASE BY CASE BASIS

   a. ___ **PAHs** (_ all 16, _ LMW, _ HMW, _ as specified)
   b. ___ **2,3,7,8-TCDD**
   c. ___ **2,3,7,8-TCDF**
   d. ___ **PCB coplanar** (#77,126,156,169)
   e. ___ **LIS pesticides**
   d. ___ **Other:**

4. BIOASSAYS (species listed in guidance manual)
   a. C **Water Column Acute Tox.** (bivalve larvae, M. bahia, Menidia sp.)
   b. C **10-Day Benthic Acute Tox.** (A. abdita, R. abronius, E. estuarius, or L. plumulosus)
   c. C **10-Day Benthic Acute Tox.** (M. bahia)
   d. C **28-Day Bioaccumulation** (N. virens, and Macoma secta or M. nasuta) - INCLUDING DIOXIN,

e. __ **28-Day Bioaccumulation** (N. virens and Macoma secta or M. nasuta)): DIOXIN ONLY

5. 28-DAY WHOLE-SEDIMENT BIOACCUMULATION TISSUE ANALYSIS

REQUIRED

a. T A **Metals** (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)

b. T A **Pesticides** (aldrin, alpha chlordane, trans nonachlor, dieldrin,p,p' and o,p' DDT/DDD/DDE, endosulfans(I,II, and sulfate), heptachlor, heptachlor epoxide)

c. T A **PCBs** (#8,18,28,44,49,52,66,87,101,105,118,128,138,153,170,180,183,184,187,195,206,209

d. T A **Semi-volatiles** (1,4 dichlorobenzene)

e. T A **PAHs (LMWs:** acenapthene, acenaphthylene, anthracene, fluorene, naphthalene, phenanthrene)

f. T A **PAHs (HMWs:** benzo(a)anthracene, benzo(a)pyrene,benzo(g,h,i)perylene,benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthrene, indeno(1,2,3-c,d)pyrene, pyrene)

CASE BY CASE BASIS

a. T A **Dioxins/Furans** (2,3,7,8 - substituted isomers, n=17)

b. ___ **LIS pesticides**

c. ___ **LIS PAHs**

d. ___ **PCB coplanar** (#77,126,156,169)

e. ___ **Other:**

REFER TO COMMENT SECTION ON PAGE 2 FOR ADDITIONAL INFORMATION

C Complete Sediment Chemistry Data Tables

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Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID: / Sample Name: / Date: VC 15 / VC15-D15-18E / 2/8/2018		VC 15 / VC15-D18-21E / 2/8/2018		VC 15 / VC15-D21-24E / 2/8/2018		VC 15 / VC15-D24-25.1E / 2/8/2018		VC 16 / VC16-D15-18E / 2/10/2018		VC 16 / VC16-D18-21E / 2/10/2018		VC 16 / DUP6-D18-21E / 2/10/2018		VC 16 / VC16-D21-24E / 2/10/2018	
Polycyclic Aromatic Hydrocarbons by Method SW-846 8270D (µg/kg)																		
2-Chloronaphthalene	N/A	N/A	0.607	U	0.562	U	0.574	U	**0.955**	J	0.594	U	0.557	U	0.550	U	0.535	U
2-Methylnaphthalene	70	N/A	0.961	U	**1.28**	J	**0.969**	J	**1.45**	J	**44.0**	J	**5.80**	J	**4.88**	J	0.848	U
Acenaphthene	16	N/A	0.868	U	**0.866**	J	0.822	U	**1.18**	J	**13.8**	J	**1.99**	J	**2.21**	J	0.766	U
Acenaphthylene	44	N/A	0.519	U	**0.639**	J	0.491	U	**0.662**	J	**34.9**		**3.73**	J	**3.64**	J	0.458	U
Anthracene	85	N/A	**2.05**	J	**1.60**	J	**1.65**	J	**1.90**	J	**66.5**		**9.22**	J	**9.26**	J	**1.22**	J
Benzo(A)Anthracene	261	N/A	2.07	U	1.91	U	1.96	U	2.11	U	**192**		**21.1**	J	**25.5**	J	1.82	U
Benzo(A)Pyrene	430	N/A	0.907	U	0.839	U	0.858	U	0.924	U	**233**		**24.6**		**27.2**		0.800	U
Benzo(B)Fluoranthene	1800*	N/A	**2.45**	J	**1.76**	J	**2.35**	J	**2.01**	J	**187**		**22.5**	J	**25.9**	J	**1.40**	J
Benzo(G,H,I)Perylene	170	N/A	**1.54**	J	**1.14**	J	**1.51**	J	**1.45**	J	**149**		**17.1**	J	**18.1**	J	**1.22**	J
Benzo(K)Fluoranthene	240	N/A	0.798	U	0.739	U	0.756	U	**0.908**	J	**137**		**14.9**	J	**17.1**	J	0.704	U
Chrysene	384	N/A	**2.15**	J	**1.82**	J	**2.10**	J	**2.00**	J	**195**		**22.2**	J	**27.1**	J	**1.36**	J
Dibenz(A,H)Anthracene	63	N/A	0.798	U	0.739	U	0.756	U	0.814	U	**37.0**		**3.91**	J	**4.83**	J	0.704	U
Fluoranthene	600	N/A	**4.39**	J	**3.41**	J	**4.11**	J	**3.99**	J	**287**		**38.6**	J	**37.8**	J	**2.38**	J
Fluorene	19	N/A	**3.12**	J	**2.84**	J	**3.09**	J	**3.73**	J	**31.1**		**6.31**	J	**6.33**	J	**2.20**	J
Indeno(1,2,3-C,D)Pyrene	200	N/A	2.21	U	2.04	U	2.09	U	2.25	U	**134**		**16.0**	J	**16.9**	J	1.95	U
Naphthalene	160	N/A	1.30	U	2.13	U	1.91	U	2.43	U	**69.4**	J	**9.94**	J	**7.05**	J	0.896	U
Phenanthrene	240	N/A	**4.30**	J	**3.67**	J	**4.08**	J	**4.93**	J	**130**	J	**19.4**	J	**19.2**	J	**2.62**	J
Pyrene	665	N/A	**3.21**	J	**1.85**	J	**2.04**	J	**1.96**	J	**275**		**33.6**	J	**40.5**	J	**1.45**	J
Total PAHs	4000	4000	**34.2**	J	**29.8**	J	**32.1**	J	**35.7**	J	**2216**	J	**271**	J	**294**	J	**23.3**	J
Sum of Low-Molecular Weight PAHs[3]	N/A	N/A	**12.2**	J	**11.7**	J	**12.0**	J	**14.8**	J	**346**	J	**50.6**	J	**47.7**	J	**8.16**	J
Sum of High-Molecular Weight PAHs[3]	N/A	N/A	**20.5**	J	**16.2**	J	**18.5**	J	**18.4**	J	**1826**		**215**	J	**241**	J	**13.8**	J
Dioxins and Furans by EPA Method 1613B (pg/g)																		
1,2,3,4,6,7,8-Heptachlorodibenzofuran	N/A	N/A	--		--		--		--		**44.6**		1.66	U	1.67	U	1.66	U
1,2,3,4,6,7,8-Heptachlorodibenzo-P-Dioxin	N/A	N/A	--		--		--		--		**132**		**17.8**		**26.6**		1.66	U
1,2,3,4,7,8,9-Heptachlorodibenzofuran	N/A	N/A	--		--		--		--		1.66	U	1.66	U	1.67	U	1.66	U
1,2,3,4,7,8-Hexachlorodibenzofuran	N/A	N/A	--		--		--		--		**6.46**		1.66	U	1.67	U	1.66	U
1,2,3,4,7,8-Hexachlorodibenzo-P-Dioxin	N/A	N/A	--		--		--		--		1.66	U	1.66	U	1.67	U	1.66	U
1,2,3,6,7,8-Hexachlorodibenzofuran	N/A	N/A	--		--		--		--		1.66	U	1.66	U	1.67	U	1.66	U
1,2,3,6,7,8-Hexachlorodibenzo-P-Dioxin	N/A	N/A	--		--		--		--		**20.9**		1.66	U	1.67	U	1.66	U
1,2,3,7,8,9-Hexachlorodibenzofuran	N/A	N/A	--		--		--		--		1.66	U	1.66	U	1.67	U	1.66	U
1,2,3,7,8,9-Hexachlorodibenzo-P-Dioxin	N/A	N/A	--		--		--		--		**11.9**		1.66	U	1.67	U	1.66	U
1,2,3,7,8-Pentachlorodibenzofuran	N/A	N/A	--		--		--		--		1.66	U	1.66	U	1.67	U	1.66	U
1,2,3,7,8-Pentachlorodibenzo-P-Dioxin	N/A	N/A	--		--		--		--		1.66	U	1.66	U	1.67	U	1.66	U
2,3,4,6,7,8-Hexachlorodibenzofuran	N/A	N/A	--		--		--		--		1.66	U	1.66	U	1.67	U	1.66	U
2,3,4,7,8-Pentachlorodibenzofuran	N/A	N/A	--		--		--		--		1.66	U	1.66	U	1.67	U	1.66	U
2,3,7,8-Tetrachlorodibenzofuran	N/A	N/A	--		--		--		--		**6.79**		0.334	U	0.334	U	0.334	U
2,3,7,8-Tetrachlorodibenzo-P-Dioxin	N/A	0.5	--		--		--		--		**5.58**		0.334	U	0.334	U	0.334	U
Octachlorodibenzofuran	N/A	N/A	--		--		--		--		**75.8**		3.34	U	3.34	U	3.34	U
Octachlorodibenzo-P-Dioxin	N/A	N/A	--		--		--		--		**1670**		**378**		**581**		**113**	J
Total Toxicity Equivalency Factor	3.65	4.5	--		--		--		--		**16.1**		**3.86**		**4.17**		**3.44**	J

Key at end of table.

Source: Ecology and Environment, Inc. 2018

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID:	VC 15		VC 15		VC 15		VC 15		VC 16		VC 16		VC 16		VC 16	
			Sample Name:	VC15-D15-18E		VC15-D18-21E		VC15-D21-24E		VC15-D24-25.1E		VC16-D15-18E		VC16-D18-21E		DUP6-D18-21E		VC16-D21-24E	
			Date:	2/8/2018		2/8/2018		2/8/2018		2/8/2018		2/10/2018		2/10/2018		2/10/2018		2/10/2018	
Organochlorine Pesticides by Method SW-846 8081B (µg/kg)																			
Aldrin	2	N/A		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Alpha BHC	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Alpha Endosulfan	N/A	0.1		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Beta BHC	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Beta Endosulfan	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Chlordane	7	63		18.9	U	18.0	U	19.2	U	19.9	U	19.1	U	18.1	U	17.2	U	17.8	U
Cis-Chlordane	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	**0.424**		0.360	U	0.343	U	0.355	U
Delta BHC	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Dieldrin	2	6		0.376	U	0.359	U	0.383	U	0.397	U	**1.03**	J	0.360	U	0.343	U	0.355	U
Endosulfan Sulfate	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Endrin	3	6		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Endrin Aldehyde	N/A	N/A		1.13	U	1.08	U	1.15	U	1.19	U	**2.54**	J	1.08	U	1.03	U	1.06	UJ
Endrin Ketone	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Gamma BHC	N/A	1		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Heptachlor	0.3*	71		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Heptachlor Epoxide	5	15		0.753	U	0.718	U	0.766	U	0.794	U	0.760	U	0.720	U	0.686	U	0.710	U
Methoxychlor	N/A	59		3.76	U	3.59	U	3.83	U	3.97	U	3.80	U	3.60	U	3.43	U	3.55	UJ
Mirex	7	120		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
O,P'-DDD	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	**7.98**	J	0.360	U	0.343	U	0.355	U
O,P'-DDE	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	**4.99**	J	0.360	U	0.343	U	0.355	U
O,P'-DDT	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
P,P'-DDD	2	N/A		0.376	U	0.359	U	0.383	U	0.397	U	**2.07**		0.360	U	**0.499**		0.355	U
P,P'-DDE	2.2	N/A		0.376	U	0.359	U	0.383	U	0.397	U	**6.47**		0.360	U	0.343	U	0.355	U
P,P'-DDT	1	N/A		0.376	U	0.359	U	0.383	U	0.397	U	**0.742**	J	0.360	U	0.343	U	0.355	U
Toxaphene	N/A	54		18.9	U	18.0	U	19.2	U	19.9	U	19.1	U	18.1	U	17.2	U	17.8	U
Trans-Chlordane	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Trans-Nonachlor	N/A	N/A		0.376	U	0.359	U	0.383	U	0.397	U	0.380	U	0.360	U	0.343	U	0.355	U
Sum of DDT + DDE + DDD	1.6	44		2.26	U	2.15	U	2.30	U	2.38	U	**22.6**	J	2.16	U	2.21	U	2.13	U
PCB Aroclors by Method SW-846 8082A (µg/kg)																			
Aroclor 1016	7	N/A		15.1	U	14.4	U	15.3	U	15.9	U	15.2	UJ	14.4	UJ	13.7	UJ	14.2	U
Aroclor 1221	N/A	N/A		15.1	U	14.4	U	15.3	U	15.9	U	15.2	U	14.4	U	13.7	U	14.2	U
Aroclor 1232	N/A	N/A		15.1	U	14.4	U	15.3	U	15.9	U	15.2	U	14.4	U	13.7	U	14.2	U
Aroclor 1242	N/A	N/A		15.1	U	14.4	U	15.3	U	15.9	U	15.2	U	14.4	U	13.7	U	14.2	U
Aroclor 1248	30	N/A		15.1	U	14.4	U	15.3	U	15.9	U	15.2	U	14.4	U	13.7	U	14.2	U
Aroclor 1254	60	N/A		15.1	U	14.4	U	15.3	U	15.9	U	15.2	U	14.4	U	13.7	U	14.2	U
Aroclor 1260	5	N/A		15.1	U	14.4	U	15.3	U	15.9	U	15.2	UJ	14.4	UJ	13.7	UJ	14.2	U
Total PCB Aroclors	23	100		106	U	101	U	107	U	111	U	106	UJ	101	UJ	95.9	UJ	99.4	U

Key at end of table.

Source: Ecology and Environment, Inc. 2018

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID:	VC 15		VC 15		VC 15		VC 15		VC 16		VC 16		VC 16		VC 16	
			Sample Name:	VC15-D15-18E		VC15-D18-21E		VC15-D21-24E		VC15-D24-25.1E		VC16-D15-18E		VC16-D18-21E		DUP6-D18-21E		VC16-D21-24E	
			Date:	2/8/2018		2/8/2018		2/8/2018		2/8/2018		2/10/2018		2/10/2018		2/10/2018		2/10/2018	
Metals by Method SW-846 6020A (mg/kg)																			
Aluminum	18000*	N/A		**13800**		**15100**		**16300**		**13800**		**15600**		**15600**		**15800**		**14600**	J
Antimony	9.3*	N/A		0.352	U	0.343	U	0.263	U	0.257	U	**1.46**	J	**0.461**	J	**0.392**	J	**0.442**	J
Arsenic	8.2	8.2		**10.3**		**12.6**		**12.8**		**11.0**		**23.0**		**12.6**		**13.0**		**11.4**	
Barium	48*	N/A		**27.3**		**28.8**		**32.0**		**27.8**		**64.8**		**32.4**	J	**36.1**	J	**28.6**	J
Beryllium	N/A	N/A		**0.853**		**0.952**		**1.02**		**1.03**		**0.921**		**0.939**		**0.951**		**0.931**	
Cadmium	1.2	1.2		**0.103**	J	**0.121**	J	**0.127**	J	**0.099**	J	**1.67**	J	**0.311**	J	**0.280**	J	**0.106**	J
Calcium	N/A	N/A		**5140**	J	**5860**		**6630**		**6980**		**8400**		**8730**		**9240**		**7770**	
Chromium, Total	81	81		**32.1**		**35.7**		**39.3**		**33.8**		**111**		**45.1**		**47.3**		**34.2**	
Cobalt	10*	N/A		**10.5**		**11.3**		**11.6**		**9.71**		**10.8**		**10.2**		**10.4**		**10.2**	
Copper	34	34		**13.0**	J	**13.9**	J	**14.3**	J	**12.3**	J	**115**	J	**24.9**	J	**26.8**	J	**13.4**	J
Iron	N/A	N/A		**33200**		**37400**		**39800**		**33400**		**37000**		**34700**		**35000**		**34800**	
Lead	47	47		**12.6**		**13.9**		**14.4**		**12.2**		**117**		**24.5**		**26.3**		**12.5**	
Magnesium	N/A	N/A		**8770**		**9180**		**10100**		**8600**		**9000**		**9010**		**9190**		**8600**	
Manganese	260*	N/A		**401**		**561**		**485**		**387**		**495**		**415**		**444**		**378**	J
Molybdenum	N/A	N/A		**1.78**		**1.25**	J	**1.47**		**1.16**	J	**1.67**		**1.53**		**1.61**		**1.36**	J
Nickel	21	21		**27.0**	J	**28.9**	J	**30.4**	J	**26.3**	J	**35.2**		**28.3**		**28.2**		**27.0**	
Potassium	N/A	N/A		**3570**		**3940**		**4220**		**3740**		**3570**	J	**4030**	J	**4120**	J	**3620**	J
Selenium	1*	N/A		**2.82**	J	**2.99**	J	**2.92**	J	**2.40**	J	**3.99**		**3.10**	J	**3.57**	J	**3.06**	J
Silver	1	1		0.096	U	0.085	U	0.090	U	0.093	U	**3.69**		**0.517**	J	**0.572**	J	**0.121**	J
Sodium	N/A	N/A		**9270**	J	**8210**	J	**8730**	J	**8430**	J	**9350**	J	**6560**	J	**6850**	J	**5350**	J
Strontium	N/A	N/A		**35.2**		**38.1**		**42.8**		**42.0**		**59.1**		**50.6**		**58.9**		**44.8**	J
Thallium	N/A	N/A		**0.134**	J	**0.138**	J	**0.154**	J	**0.128**	J	**0.230**	J	**0.147**	J	**0.143**	J	**0.688**	
Tin	N/A	N/A		0.736	U	0.814	U	0.807	U	0.627	U	**14.9**		**2.82**		**2.58**	J	**3.76**	
Titanium	N/A	N/A		**254**		**246**		**295**		**261**		**396**		**330**		**344**		**295**	J
Vanadium	57*	N/A		**39.8**		**41.2**		**44.1**		**37.6**		**52.7**		**41.8**		**44.0**		**38.8**	
Zinc	150	150		**74.8**	J	**82.2**	J	**85.3**	J	**71.4**	J	**230**	J	**91.4**	J	**93.6**	J	**72.4**	J
Mercury by Method SW-846 7474 (mg/kg)																			
Mercury	0.15	0.15		**0.021**	J	**0.0192**	J	0.015	U	**0.016**	J	**1.93**		**0.219**		**0.275**		0.003	U
Total Organic Carbon by Method SW-846 9060A (mg/kg)																			
Total Organic Carbon	N/A	N/A		**17050**		**18450**		**18000**		**18400**		**23650**		**16500**		**16500**		**14900**	

Key at end of table.

Source: Ecology and Environment, Inc. 2018

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID:	VC 16		VC 17		VC 17		VC 17		VC 18		VC 18		VC 18		VC 18	
			Sample Name:	VC16-D24-27.9E		VC17-D15-18E		VC17-D18-21E		VC17-D21-24.1E		VC18-D15-18E		VC18-D18-21E		VC18-D21-24E		VC18-D24-27E	
			Date:	2/10/2018		2/10/2018		2/10/2018		2/10/2018		2/8/2018		2/8/2018		2/8/2018		2/8/2018	
Polycyclic Aromatic Hydrocarbons by Method SW-846 8270D (µg/kg)																			
2-Chloronaphthalene	N/A	N/A		0.528	U	0.544	U	0.573	U	0.538	U	0.594	U	0.534	U	**0.946**	J	0.516	U
2-Methylnaphthalene	70	N/A		0.837	U	0.861	U	0.908	U	0.852	U	**2.18**	J	0.846	U	**1.23**	J	**0.873**	J
Acenaphthene	16	N/A		0.756	U	0.778	U	0.820	U	0.770	U	0.849	U	0.765	U	**0.803**	J	0.738	U
Acenaphthylene	44	N/A		0.452	U	0.465	U	0.490	U	0.460	U	0.508	U	0.457	U	**0.627**	J	0.442	U
Anthracene	85	N/A		**1.18**	J	0.861	U	0.908	U	**1.03**	J	**1.30**	J	**1.10**	J	**1.45**	J	**1.11**	J
Benzo(A)Anthracene	261	N/A		1.80	U	1.85	U	1.95	U	1.83	U	2.02	U	1.82	U	1.90	U	1.76	U
Benzo(A)Pyrene	430	N/A		0.790	U	0.812	U	0.856	U	0.804	U	0.887	U	0.799	U	0.835	U	0.771	U
Benzo(B)Fluoranthene	1800*	N/A		**1.78**	J	**1.79**	J	**1.23**	J	**2.16**	J	**1.95**	J	**1.93**	J	**1.76**	J	**1.49**	J
Benzo(G,H,I)Perylene	170	N/A		**2.13**	J	**1.96**	J	**1.16**	J	**1.45**	J	**1.18**	J	**1.23**	J	**1.22**	J	**1.03**	J
Benzo(K)Fluoranthene	240	N/A		**0.887**	J	0.715	U	0.754	U	**0.867**	J	0.781	U	0.703	U	0.735	U	0.679	U
Chrysene	384	N/A		**1.50**	J	**1.05**	J	**0.851**	J	**1.84**	J	**1.78**	J	**1.62**	J	**1.77**	J	**1.36**	J
Dibenz(A,H)Anthracene	63	N/A		2.14	U	**2.54**	J	0.754	U	0.708	U	0.781	U	0.703	U	0.735	U	0.679	U
Fluoranthene	600	N/A		**2.84**	J	**2.16**	J	**1.92**	J	**2.94**	J	**3.30**	J	**3.10**	J	**3.46**	J	**2.80**	J
Fluorene	19	N/A		**2.25**	J	**1.65**	J	**1.48**	J	**2.71**	J	**2.30**	J	**1.96**	J	**2.73**	J	**2.09**	J
Indeno(1,2,3-C,D)Pyrene	200	N/A		**2.05**	J	1.98	U	2.09	U	1.96	U	2.16	U	1.94	U	2.03	U	1.88	U
Naphthalene	160	N/A		0.910	U	0.890	U	0.806	U	1.38	U	**16.0**	J	1.11	U	2.00	U	2.59	U
Phenanthrene	240	N/A		**2.84**	J	**2.34**	J	**2.35**	J	**3.31**	J	**3.10**	J	**3.03**	J	**3.95**	J	**2.89**	J
Pyrene	665	N/A		**1.67**	J	**1.36**	J	**1.06**	J	**1.62**	J	**1.63**	J	**1.72**	J	**1.82**	J	**1.42**	J
Total PAHs	4000	4000		**27.3**	J	**24.6**	J	**21.0**	J	**27.2**	J	**43.3**	J	**25.4**	J	**30.0**	J	**25.1**	J
Sum of Low-Molecular Weight PAHs[3]	N/A	N/A		**8.39**	J	**6.98**	J	**6.85**	J	**9.66**	J	**24.1**	J	**8.42**	J	**11.6**	J	**9.86**	J
Sum of High-Molecular Weight PAHs[3]	N/A	N/A		**17.6**	J	**16.2**	J	**12.6**	J	**16.2**	J	**16.5**	J	**15.6**	J	**16.3**	J	**13.9**	J
Dioxins and Furans by EPA Method 1613B (pg/g)																			
1,2,3,4,6,7,8-Heptachlorodibenzofuran	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
1,2,3,4,6,7,8-Heptachlorodibenzo-P-Dioxin	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
1,2,3,4,7,8,9-Heptachlorodibenzofuran	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
1,2,3,4,7,8-Hexachlorodibenzofuran	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
1,2,3,4,7,8-Hexachlorodibenzo-P-Dioxin	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
1,2,3,6,7,8-Hexachlorodibenzofuran	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
1,2,3,6,7,8-Hexachlorodibenzo-P-Dioxin	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
1,2,3,7,8,9-Hexachlorodibenzofuran	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
1,2,3,7,8,9-Hexachlorodibenzo-P-Dioxin	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
1,2,3,7,8-Pentachlorodibenzofuran	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
1,2,3,7,8-Pentachlorodibenzo-P-Dioxin	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
2,3,4,6,7,8-Hexachlorodibenzofuran	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
2,3,4,7,8-Pentachlorodibenzofuran	N/A	N/A		1.66	U	--		--		--		--		--		--		--	
2,3,7,8-Tetrachlorodibenzofuran	N/A	N/A		0.333	U	--		--		--		--		--		--		--	
2,3,7,8-Tetrachlorodibenzo-P-Dioxin	N/A	0.5		0.333	U	--		--		--		--		--		--		--	
Octachlorodibenzofuran	N/A	N/A		3.33	U	--		--		--		--		--		--		--	
Octachlorodibenzo-P-Dioxin	N/A	N/A		**145**		--		--		--		--		--		--		--	
Total Toxicity Equivalency Factor	3.65	4.5		**3.47**		--		--		--		--		--		--		--	

Key at end of table.

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID:	VC 16		VC 17		VC 17		VC 17		VC 18		VC 18		VC 18		VC 18	
			Sample Name:	VC16-D24-27.9E		VC17-D15-18E		VC17-D18-21E		VC17-D21-24.1E		VC18-D15-18E		VC18-D18-21E		VC18-D21-24E		VC18-D24-27E	
			Date:	2/10/2018		2/10/2018		2/10/2018		2/10/2018		2/8/2018		2/8/2018		2/8/2018		2/8/2018	
Organochlorine Pesticides by Method SW-846 8081B (µg/kg)																			
Aldrin	2	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Alpha BHC	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Alpha Endosulfan	N/A	0.1		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Beta BHC	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Beta Endosulfan	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Chlordane	7	63		16.8	U	17.8	U	18.0	U	17.8	U	18.9	U	18.2	U	17.5	U	16.2	U
Cis-Chlordane	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Delta BHC	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Dieldrin	2	6		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Endosulfan Sulfate	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Endrin	3	6		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Endrin Aldehyde	N/A	N/A		1.01	U	1.06	U	1.08	U	1.07	U	1.13	U	1.09	U	1.05	U	0.968	U
Endrin Ketone	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Gamma BHC	N/A	1		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Heptachlor	0.3*	71		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Heptachlor Epoxide	5	15		0.672	U	0.709	U	0.717	U	0.711	U	0.752	U	0.726	U	0.698	U	0.646	U
Methoxychlor	N/A	59		3.36	U	3.54	U	3.58	U	3.56	U	3.76	U	3.63	U	3.49	U	3.23	U
Mirex	7	120		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
O,P'-DDD	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
O,P'-DDE	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
O,P'-DDT	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
P,P'-DDD	2	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
P,P'-DDE	2.2	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
P,P'-DDT	1	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Toxaphene	N/A	54		16.8	U	17.8	U	18.0	U	17.8	U	18.9	U	18.2	U	17.5	U	16.2	U
Trans-Chlordane	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Trans-Nonachlor	N/A	N/A		0.336	U	0.354	U	0.358	U	0.356	U	0.376	U	0.363	U	0.349	U	0.323	U
Sum of DDT + DDE + DDD	1.6	44		2.02	U	2.12	U	2.15	U	2.14	U	2.26	U	2.18	U	2.09	U	1.94	U
PCB Aroclors by Method SW-846 8082A (µg/kg)																			
Aroclor 1016	7	N/A		13.4	UJ	14.2	UJ	14.3	UJ	14.2	UJ	15.0	U	14.5	U	14.0	U	12.9	U
Aroclor 1221	N/A	N/A		13.4	U	14.2	U	14.3	U	14.2	U	15.0	U	14.5	U	14.0	U	12.9	U
Aroclor 1232	N/A	N/A		13.4	U	14.2	U	14.3	U	14.2	U	15.0	U	14.5	U	14.0	U	12.9	U
Aroclor 1242	N/A	N/A		13.4	U	14.2	U	14.3	U	14.2	U	15.0	U	14.5	U	14.0	U	12.9	U
Aroclor 1248	30	N/A		13.4	U	14.2	U	14.3	U	14.2	U	15.0	U	14.5	U	14.0	U	12.9	U
Aroclor 1254	60	N/A		13.4	U	14.2	U	14.3	U	14.2	U	15.0	U	14.5	U	14.0	U	12.9	U
Aroclor 1260	5	N/A		13.4	UJ	14.2	UJ	14.3	UJ	14.2	UJ	15.0	U	14.5	U	14.0	U	12.9	U
Total PCB Aroclors	23	100		93.8	UJ	99.4	UJ	100	UJ	99.4	UJ	105	U	102	U	98.0	U	90.3	U

Key at end of table.

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Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID: / Sample Name: / Date:																	
			VC 16 VC16-D24-27.9E 2/10/2018		VC 17 VC17-D15-18E 2/10/2018		VC 17 VC17-D18-21E 2/10/2018		VC 17 VC17-D21-24.1E 2/10/2018		VC 18 VC18-D15-18E 2/8/2018		VC 18 VC18-D18-21E 2/8/2018		VC 18 VC18-D21-24E 2/8/2018		VC 18 VC18-D24-27E 2/8/2018	
Metals by Method SW-846 6020A (mg/kg)																				
Aluminum	18000*	N/A	**16600**		**16300**		**15500**		**16900**		**14300**		**13600**		**15500**		**14300**	
Antimony	9.3*	N/A	**0.272**	J	**0.281**	J	**0.267**	J	**0.281**	J	0.360	U	0.243	U	0.258	U	0.220	U
Arsenic	8.2	8.2	**11.8**		**11.8**		**11.5**		**12.4**		**13.2**		**10.1**		**12.2**		**12.6**	
Barium	48*	N/A	**31.1**	J	**30.0**	J	**29.4**	J	**31.2**	J	**27.9**		**26.2**		**30.9**		**28.3**	
Beryllium	N/A	N/A	**0.944**		**0.989**		**0.934**		**0.966**		**0.804**		**0.882**		**0.921**		**0.860**	
Cadmium	1.2	1.2	**0.101**	J	**0.115**	J	**0.106**	J	**0.111**	J	**0.127**	J	**0.114**	J	**0.113**	J	**0.128**	J
Calcium	N/A	N/A	**7150**		**8440**		**8210**		**8590**		**5040**	J	**6340**		**7150**		**7940**	
Chromium, Total	81	81	**38.1**		**38.5**		**37.8**		**40.4**		**32.9**		**31.9**		**36.5**		**33.2**	
Cobalt	10*	N/A	**10.8**		**11.1**		**10.5**		**11.5**		**10.7**		**9.63**		**10.7**		**9.47**	
Copper	34	34	**14.4**	J	**16.7**	J	**14.6**	J	**15.8**	J	**12.9**	J	**11.9**	J	**13.2**	J	**12.2**	J
Iron	N/A	N/A	**35600**		**35900**		**34600**		**37700**		**34200**		**32700**		**37400**		**33100**	
Lead	47	47	**13.0**		**13.4**		**12.8**		**13.6**		**12.8**		**12.2**		**13.8**		**12.5**	
Magnesium	N/A	N/A	**9430**		**9410**		**8960**		**9470**		**8900**		**8550**		**9500**		**8460**	
Manganese	260*	N/A	**421**		**397**		**402**		**415**		**528**		**376**		**443**		**382**	
Molybdenum	N/A	N/A	**1.43**		**1.60**		**1.40**	J	**1.87**		**1.20**	J	**1.12**	J	**1.43**		**1.42**	
Nickel	21	21	**27.8**		**29.0**		**27.6**		**29.5**		**27.3**	J	**25.3**	J	**27.8**	J	**25.8**	J
Potassium	N/A	N/A	**4200**	J	**4200**	J	**4110**	J	**4340**	J	**3630**		**3800**		**4070**		**3810**	
Selenium	1*	N/A	**3.05**	J	**3.53**	J	**3.78**		**3.58**		**2.64**	J	**2.39**	J	**2.81**	J	**2.56**	J
Silver	1	1	**0.110**	J	**0.098**	J	**0.099**	J	**0.096**	J	0.095	U	0.088	U	0.087	U	0.079	U
Sodium	N/A	N/A	**5340**	J	**5660**	J	**4470**	J	**3640**	J	**10300**	J	**8900**	J	**9190**	J	**7530**	J
Strontium	N/A	N/A	**42.9**	J	**47.8**		**49.3**		**50.1**		**35.4**		**37.7**		**44.4**		**46.9**	
Thallium	N/A	N/A	**0.150**	J	**0.406**		**0.231**	J	**0.200**	J	**0.126**	J	**0.124**	J	**0.144**	J	**0.132**	J
Tin	N/A	N/A	**1.06**	J	**2.50**	J	**1.50**	J	**1.43**	J	0.660	U	0.631	U	0.754	U	0.637	U
Titanium	N/A	N/A	**342**		**328**		**327**		**354**		**254**		**224**		**281**		**266**	
Vanadium	57*	N/A	**44.0**		**45.0**		**44.2**		**48.3**		**36.9**		**35.3**		**40.4**		**38.2**	
Zinc	150	150	**76.7**	J	**77.4**	J	**74.4**	J	**79.5**	J	**76.5**	J	**73.6**	J	**79.0**	J	**71.6**	J
Mercury by Method SW-846 7474 (mg/kg)																		
Mercury	0.15	0.15	**0.016**	J	**0.005**	J	**0.004**	J	**0.012**	J	**0.019**	J	**0.015**	J	**0.017**	J	**0.017**	J
Total Organic Carbon by Method SW-846 9060A (mg/kg)																		
Total Organic Carbon	N/A	N/A	**15150**		**15500**		**15900**		**16450**		**16750**		**17100**		**17600**		**17400**	

Key at end of table.

Source: Ecology and Environment, Inc. 2018

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID:	VC 18		VC 38		VC 38		VC 38		VC 38		VC 39		VC 39		VC 39	
			Sample Name:	VC18-D27-29.3E		VC38-D15-18E		VC38-D18-21E		VC38-D21-24E		VC38-D24-26.1E		VC39-D12-15E		VC39-D15-18E		VC39-D18-21.2E	
			Date:	2/8/2018		2/11/2018		2/11/2018		2/11/2018		2/11/2018		2/10/2018		2/10/2018		2/10/2018	
Polycyclic Aromatic Hydrocarbons by Method SW-846 8270D (µg/kg)																			
2-Chloronaphthalene	N/A	N/A		0.541	U	0.370	U	0.359	U	0.329	U	0.369	U	0.335	U	0.338	U	0.347	U
2-Methylnaphthalene	70	N/A		0.857	U	0.587	U	0.569	U	0.522	U	0.584	U	0.531	U	0.534	U	0.550	U
Acenaphthene	16	N/A		0.774	U	0.530	U	0.514	U	0.471	U	0.528	U	0.480	U	0.483	U	0.497	U
Acenaphthylene	44	N/A		0.463	U	0.317	U	0.307	U	0.282	U	0.316	U	0.287	U	0.289	U	0.297	U
Anthracene	85	N/A		**1.30**	J	0.587	U	0.569	U	0.522	U	0.584	U	0.531	U	0.534	U	0.550	U
Benzo(A)Anthracene	261	N/A		1.84	U	1.26	U	1.22	U	1.12	U	1.26	U	1.14	U	1.15	U	1.18	U
Benzo(A)Pyrene	430	N/A		0.809	U	0.554	U	0.537	U	0.492	U	0.551	U	0.501	U	0.504	U	0.519	U
Benzo(B)Fluoranthene	1800*	N/A		**1.72**	J	0.492	U	0.477	U	0.438	U	0.490	U	0.445	U	0.448	U	0.461	U
Benzo(G,H,I)Perylene	170	N/A		**1.24**	J	0.390	U	0.378	U	0.347	U	0.388	U	0.353	U	0.355	U	0.365	U
Benzo(K)Fluoranthene	240	N/A		**0.729**	J	0.487	U	0.472	U	0.433	U	0.485	U	0.441	U	0.444	U	0.457	U
Chrysene	384	N/A		**1.61**	J	0.416	U	0.403	U	0.370	U	0.414	U	0.376	U	0.379	U	0.390	U
Dibenz(A,H)Anthracene	63	N/A		0.712	U	0.487	U	0.472	U	0.433	U	0.485	U	0.441	U	0.444	U	0.457	U
Fluoranthene	600	N/A		**3.04**	J	0.866	U	0.840	U	0.770	U	0.862	U	0.784	U	0.789	U	0.812	U
Fluorene	19	N/A		**2.33**	J	0.316	U	0.306	U	0.281	U	0.315	U	0.286	U	0.288	U	0.296	U
Indeno(1,2,3-C,D)Pyrene	200	N/A		1.97	U	1.35	U	1.31	U	1.20	U	1.34	U	1.22	U	1.23	U	1.26	U
Naphthalene	160	N/A		1.07	U	0.468	U	0.715	U	0.504	U	0.692	U	0.471	U	0.418	U	0.397	U
Phenanthrene	240	N/A		**2.93**	J	0.558	U	0.541	U	0.496	U	0.556	U	0.505	U	0.509	U	0.523	U
Pyrene	665	N/A		**1.51**	J	0.483	U	0.468	U	0.429	U	0.481	U	0.437	U	0.440	U	0.452	U
Total PAHs	4000	4000		**25.4**	J	10.5	U	10.5	U	9.44	U	10.7	U	9.56	U	9.58	U	9.81	U
Sum of Low-Molecular Weight PAHs[3]	N/A	N/A		**8.87**	J	2.78	U	2.95	U	2.56	U	2.99	U	2.56	U	2.52	U	2.56	U
Sum of High-Molecular Weight PAHs[3]	N/A	N/A		**15.2**	J	6.79	U	6.58	U	6.03	U	6.76	U	6.14	U	6.18	U	6.35	U
Dioxins and Furans by EPA Method 1613B (pg/g)																			
1,2,3,4,6,7,8-Heptachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--		--		--	
1,2,3,4,6,7,8-Heptachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--		--		--	
1,2,3,4,7,8,9-Heptachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--		--		--	
1,2,3,4,7,8-Hexachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--		--		--	
1,2,3,4,7,8-Hexachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--		--		--	
1,2,3,6,7,8-Hexachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--		--		--	
1,2,3,6,7,8-Hexachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--		--		--	
1,2,3,7,8,9-Hexachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--		--		--	
1,2,3,7,8,9-Hexachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--		--		--	
1,2,3,7,8-Pentachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--		--		--	
1,2,3,7,8-Pentachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--		--		--	
2,3,4,6,7,8-Hexachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--		--		--	
2,3,4,7,8-Pentachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--		--		--	
2,3,7,8-Tetrachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--		--		--	
2,3,7,8-Tetrachlorodibenzo-P-Dioxin	N/A	0.5		--		--		--		--		--		--		--		--	
Octachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--		--		--	
Octachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--		--		--	
Total Toxicity Equivalency Factor	3.65	4.5		--		--		--		--		--		--		--		--	

Key at end of table.

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID:	VC 18		VC 38		VC 38		VC 38		VC 38		VC 39		VC 39		VC 39	
			Sample Name:	VC18-D27-29.3E		VC38-D15-18E		VC38-D18-21E		VC38-D21-24E		VC38-D24-26.1E		VC39-D12-15E		VC39-D15-18E		VC39-D18-21.2E	
			Date:	2/8/2018		2/11/2018		2/11/2018		2/11/2018		2/11/2018		2/10/2018		2/10/2018		2/10/2018	
Organochlorine Pesticides by Method SW-846 8081B (µg/kg)																			
Aldrin	2	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Alpha BHC	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Alpha Endosulfan	N/A	0.1		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Beta BHC	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Beta Endosulfan	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Chlordane	7	63		17.9	U	11.6	U	11.5	U	11.0	U	11.8	U	11.0	U	10.7	U	11.2	U
Cis-Chlordane	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Delta BHC	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Dieldrin	2	6		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Endosulfan Sulfate	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Endrin	3	6		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Endrin Aldehyde	N/A	N/A		1.07	U	0.694	U	0.685	U	0.657	U	0.706	U	0.656	U	0.640	U	0.667	U
Endrin Ketone	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Gamma BHC	N/A	1		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Heptachlor	0.3*	71		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Heptachlor Epoxide	5	15		0.714	U	0.463	U	0.457	U	0.438	U	0.471	U	0.437	U	0.427	U	0.445	U
Methoxychlor	N/A	59		3.57	U	2.31	U	2.28	U	2.19	U	2.35	U	2.19	U	2.13	U	2.22	U
Mirex	7	120		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
O,P'-DDD	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
O,P'-DDE	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
O,P'-DDT	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
P,P'-DDD	2	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
P,P'-DDE	2.2	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
P,P'-DDT	1	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Toxaphene	N/A	54		17.9	U	11.6	U	11.5	U	11.0	U	11.8	U	11.0	U	10.7	U	11.2	U
Trans-Chlordane	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Trans-Nonachlor	N/A	N/A		0.357	U	0.231	U	0.228	U	0.219	U	0.235	U	0.219	U	0.213	U	0.222	U
Sum of DDT + DDE + DDD	1.6	44		2.14	U	1.39	U	1.37	U	1.31	U	1.41	U	1.31	U	1.28	U	1.33	U
PCB Aroclors by Method SW-846 8082A (µg/kg)																			
Aroclor 1016	7	N/A		14.3	U	9.26	UJ	9.14	UJ	8.76	UJ	9.42	UJ	8.74	UJ	8.53	UJ	8.89	UJ
Aroclor 1221	N/A	N/A		14.3	U	9.26	U	9.14	U	8.76	U	9.42	U	8.74	U	8.53	U	8.89	U
Aroclor 1232	N/A	N/A		14.3	U	9.26	U	9.14	U	8.76	U	9.42	U	8.74	U	8.53	U	8.89	U
Aroclor 1242	N/A	N/A		14.3	U	9.26	U	9.14	U	8.76	U	9.42	U	8.74	U	8.53	U	8.89	U
Aroclor 1248	30	N/A		14.3	U	9.26	U	9.14	U	8.76	U	9.42	U	8.74	U	8.53	U	8.89	U
Aroclor 1254	60	N/A		14.3	U	9.26	U	9.14	U	8.76	U	9.42	U	8.74	U	8.53	U	8.89	U
Aroclor 1260	5	N/A		14.3	U	9.26	UJ	9.14	UJ	8.76	UJ	9.42	UJ	8.74	UJ	8.53	UJ	8.89	UJ
Total PCB Aroclors	23	100		100	U	64.8	UJ	64.0	UJ	61.3	UJ	65.9	UJ	61.2	UJ	59.7	UJ	62.2	UJ

Key at end of table.

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID:	VC 18		VC 38		VC 38		VC 38		VC 38		VC 39		VC 39		VC 39	
			Sample Name:	VC18-D27-29.3E		VC38-D15-18E		VC38-D18-21E		VC38-D21-24E		VC38-D24-26.1E		VC39-D12-15E		VC39-D15-18E		VC39-D18-21.2E	
			Date:	2/8/2018		2/11/2018		2/11/2018		2/11/2018		2/11/2018		2/10/2018		2/10/2018		2/10/2018	
Metals by Method SW-846 6020A (mg/kg)																			
Aluminum	18000*	N/A		**14600**		**1900**		2250		**3780**		**4170**		**4080**		**5820**		**6050**	
Antimony	9.3*	N/A		0.286	U	0.158	U	**0.425**	J	0.167	U	0.160	U	0.151	U	0.155	U	0.154	U
Arsenic	8.2	8.2		**12.4**		**2.59**		**8.35**		**5.67**		**3.93**		**6.47**		**6.20**		**2.03**	
Barium	48*	N/A		**28.6**		**3.64**	J	**23.9**		**42.7**		**45.0**		**5.39**	J	**5.94**	J	**8.22**	J
Beryllium	N/A	N/A		**0.878**		**0.200**	J	**0.180**	J	**0.185**	J	**0.146**	J	**0.309**	J	**0.375**		**0.304**	J
Cadmium	1.2	1.2		**0.106**	J	0.031	U	0.031	U	0.029	U	0.031	U	0.030	U	0.030	U	0.030	U
Calcium	N/A	N/A		**7400**		**212**	J	**156**	J	**154**	J	**175**	J	**249**	J	**257**	J	**209**	J
Chromium, Total	81	81		**33.3**		**10.2**		**10.7**		**16.3**		**5.26**	J	**16.0**		**12.6**		**10.2**	
Cobalt	10*	N/A		**9.90**		**2.94**		**2.91**		**6.71**		**2.62**		**5.94**		**5.66**		**4.02**	
Copper	34	34		**12.7**	J	**2.29**	J	**3.27**		**3.90**		**5.46**		**5.71**	J	**7.41**	J	**6.64**	J
Iron	N/A	N/A		**33700**		**9170**		**20300**		**15500**		**6690**		**18500**		**17600**		**8720**	
Lead	47	47		**12.5**		**1.32**		**2.64**		**2.69**		**1.63**		**4.49**		**4.72**		**6.60**	
Magnesium	N/A	N/A		**8700**		**721**	J	**882**	J	**1320**		**1120**		**1410**	J	**1620**	J	**1450**	J
Manganese	260*	N/A		**428**		**44.9**	J	**68.3**		**90.2**		**45.3**	J	**160**		**150**		**67.9**	
Molybdenum	N/A	N/A		**1.37**	J	**0.141**	J	**0.357**	J	0.171	U	0.128	U	**0.293**	J	**0.211**	J	**0.132**	J
Nickel	21	21		**26.2**	J	**7.54**		**5.82**	J	**22.0**		**6.21**	J	**9.37**	J	**11.4**	J	**9.84**	J
Potassium	N/A	N/A		**3820**		**276**		**986**		**619**		**383**		**1110**	J	**959**	J	**637**	J
Selenium	1*	N/A		**2.50**	J	0.884	U	0.876	U	0.820	U	0.896	U	0.844	U	0.867	U	**1.42**	J
Silver	1	1		0.088	U	0.057	U	**0.057**	J	0.053	U	0.058	U	0.055	U	0.056	U	0.056	U
Sodium	N/A	N/A		**8220**	J	**1220**	J	**1220**	J	**795**	J	**1460**	J	**981**	J	**996**	J	**1040**	J
Strontium	N/A	N/A		**43.2**		**2.74**	J	**5.45**	J	**6.03**	J	**4.96**	J	**5.83**	J	**5.82**	J	**6.73**	J
Thallium	N/A	N/A		**0.132**	J	0.060	U	0.060	U	0.056	U	0.061	U	0.058	U	0.059	U	0.059	U
Tin	N/A	N/A		0.936	U	**0.273**	J	**0.864**	J	0.430	U	0.567	U	**0.361**	J	**0.390**	J	**0.457**	J
Titanium	N/A	N/A		**276**		**95.1**		**69.9**		**102**		**117**		**87.2**		**115**		**104**	
Vanadium	57*	N/A		**37.1**		**10.2**		**22.8**		**15.4**		**6.98**		**22.2**		**20.5**		**13.3**	
Zinc	150	150		**75.3**	J	**12.8**	J	**12.6**	J	**20.6**	J	**15.7**	J	**20.7**	J	**27.6**	J	**23.8**	J
Mercury by Method SW-846 7474 (mg/kg)																			
Mercury	0.15	0.15		**0.018**	J	0.002	U	0.002	U	0.002	U	0.002	U	0.002	U	**0.003**	J	0.002	U
Total Organic Carbon by Method SW-846 9060A (mg/kg)																			
Total Organic Carbon	N/A	N/A		**16750**		500	U	500	U	500	U	500	U	**675.5**		**550.5**		500	U

Key at end of table.

Source: Ecology and Environment, Inc. 2018

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID: / Sample Name: / Date:	VC 40 / VC40-D15-18E / 2/10/2018		VC 40 / VC40-D18-21.7E / 2/10/2018		VC 41-ALT / VC41-ALT-D12-15E / 2/11/2018		VC 41-ALT / VC41-ALT-D15-18E / 2/11/2018		VC 41-ALT / DUP7-D15-18E / 2/11/2018		VC 41-ALT / VC41-ALT-D18-19.4E / 2/11/2018	
Polycyclic Aromatic Hydrocarbons by Method SW-846 8270D (µg/kg)															
2-Chloronaphthalene	N/A	N/A		0.335	U	0.338	U	0.373	U	0.395	U	0.389	U	0.388	U
2-Methylnaphthalene	70	N/A		0.531	U	0.536	U	0.590	U	0.626	U	0.616	U	0.614	U
Acenaphthene	16	N/A		0.480	U	0.484	U	0.533	U	0.565	U	0.556	U	0.554	U
Acenaphthylene	44	N/A		0.287	U	0.290	U	0.319	U	0.338	U	0.333	U	0.332	U
Anthracene	85	N/A		0.531	U	0.536	U	0.590	U	0.626	U	0.616	U	0.614	U
Benzo(A)Anthracene	261	N/A		1.14	U	1.15	U	1.27	U	1.35	U	1.32	U	1.32	U
Benzo(A)Pyrene	430	N/A		0.501	U	0.506	U	0.557	U	0.590	U	0.581	U	0.579	U
Benzo(B)Fluoranthene	1800*	N/A		0.446	U	0.449	U	0.495	U	0.525	U	0.516	U	0.515	U
Benzo(G,H,I)Perylene	170	N/A		**0.724**	J	**0.833**	J	0.392	U	0.416	U	0.409	U	0.408	U
Benzo(K)Fluoranthene	240	N/A		0.441	U	0.445	U	0.490	U	0.520	U	0.511	U	0.510	U
Chrysene	384	N/A		0.377	U	0.380	U	0.418	U	0.444	U	0.436	U	0.435	U
Dibenz(A,H)Anthracene	63	N/A		**1.24**	J	**1.61**	J	0.490	U	0.520	U	0.511	U	0.510	U
Fluoranthene	600	N/A		0.784	U	0.791	U	0.871	U	0.924	U	0.908	U	0.906	U
Fluorene	19	N/A		0.286	U	0.289	U	0.318	U	0.337	U	0.332	U	0.331	U
Indeno(1,2,3-C,D)Pyrene	200	N/A		1.22	U	1.23	U	1.36	U	1.44	U	1.41	U	1.41	U
Naphthalene	160	N/A		0.682	U	0.387	U	0.512	U	0.452	U	0.444	U	0.607	U
Phenanthrene	240	N/A		0.506	U	0.510	U	0.562	U	0.596	U	0.586	U	0.584	U
Pyrene	665	N/A		0.437	U	0.441	U	0.485	U	0.515	U	0.506	U	0.505	U
Total PAHs	4000	4000		**10.9**	J	**11.2**	J	10.6	U	11.2	U	11.0	U	11.1	U
Sum of Low-Molecular Weight PAHs[3]	N/A	N/A		2.77	U	2.50	U	2.83	U	2.91	U	2.87	U	3.02	U
Sum of High-Molecular Weight PAHs[3]	N/A	N/A		**7.31**	J	**7.84**	J	6.83	U	7.24	U	7.11	U	7.10	U
Dioxins and Furans by EPA Method 1613B (pg/g)															
1,2,3,4,6,7,8-Heptachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--	
1,2,3,4,6,7,8-Heptachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--	
1,2,3,4,7,8,9-Heptachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--	
1,2,3,4,7,8-Hexachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--	
1,2,3,4,7,8-Hexachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--	
1,2,3,6,7,8-Hexachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--	
1,2,3,6,7,8-Hexachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--	
1,2,3,7,8,9-Hexachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--	
1,2,3,7,8,9-Hexachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--	
1,2,3,7,8-Pentachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--	
1,2,3,7,8-Pentachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--	
2,3,4,6,7,8-Hexachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--	
2,3,4,7,8-Pentachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--	
2,3,7,8-Tetrachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--	
2,3,7,8-Tetrachlorodibenzo-P-Dioxin	N/A	0.5		--		--		--		--		--		--	
Octachlorodibenzofuran	N/A	N/A		--		--		--		--		--		--	
Octachlorodibenzo-P-Dioxin	N/A	N/A		--		--		--		--		--		--	
Total Toxicity Equivalency Factor	3.65	4.5		--		--		--		--		--		--	

Key at end of table.

Source: Ecology and Environment, Inc. 2018

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID: VC 40 Sample Name: VC40-D15-18E Date: 2/10/2018		VC 40 VC40-D18-21.7E 2/10/2018		VC 41-ALT VC41-ALT-D12-15E 2/11/2018		VC 41-ALT VC41-ALT-D15-18E 2/11/2018		VC 41-ALT DUP7-D15-18E 2/11/2018		VC 41-ALT VC41-ALT-D18-19.4E 2/11/2018	
Organochlorine Pesticides by Method SW-846 8081B (µg/kg)														
Aldrin	2	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Alpha BHC	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Alpha Endosulfan	N/A	0.1	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Beta BHC	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Beta Endosulfan	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Chlordane	7	63	11.0	U	10.6	U	12.4	U	12.4	U	12.7	U	12.7	U
Cis-Chlordane	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Delta BHC	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Dieldrin	2	6	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Endosulfan Sulfate	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Endrin	3	6	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Endrin Aldehyde	N/A	N/A	0.659	U	0.632	U	0.742	U	0.742	U	0.759	U	0.759	U
Endrin Ketone	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Gamma BHC	N/A	1	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Heptachlor	0.3*	71	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Heptachlor Epoxide	5	15	0.439	U	0.421	U	0.495	U	0.495	U	0.506	U	0.506	U
Methoxychlor	N/A	59	2.20	U	2.11	U	2.47	U	2.47	U	2.53	U	2.53	U
Mirex	7	120	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
O,P'-DDD	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
O,P'-DDE	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
O,P'-DDT	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
P,P'-DDD	2	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
P,P'-DDE	2.2	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
P,P'-DDT	1	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Toxaphene	N/A	54	11.0	U	10.6	U	12.4	U	12.4	U	12.7	U	12.7	U
Trans-Chlordane	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Trans-Nonachlor	N/A	N/A	0.220	U	0.211	U	0.247	U	0.247	U	0.253	U	0.253	U
Sum of DDT + DDE + DDD	1.6	44	1.32	U	1.27	U	1.48	U	1.48	U	1.52	U	1.52	U
PCB Aroclors by Method SW-846 8082A (µg/kg)														
Aroclor 1016	7	N/A	8.79	UJ	8.43	UJ	9.90	UJ	9.90	UJ	10.1	UJ	10.1	UJ
Aroclor 1221	N/A	N/A	8.79	U	8.43	U	9.90	U	9.90	U	10.1	U	10.1	U
Aroclor 1232	N/A	N/A	8.79	U	8.43	U	9.90	U	9.90	U	10.1	U	10.1	U
Aroclor 1242	N/A	N/A	8.79	U	8.43	U	9.90	U	9.90	U	10.1	U	10.1	U
Aroclor 1248	30	N/A	8.79	U	8.43	U	9.90	U	9.90	U	10.1	U	10.1	U
Aroclor 1254	60	N/A	8.79	U	8.43	U	9.90	U	9.90	U	10.1	U	10.1	U
Aroclor 1260	5	N/A	8.79	UJ	8.43	UJ	9.90	UJ	9.90	UJ	10.1	UJ	10.1	UJ
Total PCB Aroclors	23	100	61.5	UJ	59.0	UJ	69.3	UJ	69.3	UJ	70.7	UJ	70.7	UJ

Key at end of table.

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte	NJDEP Low Effects Range[1]	NYSDEC No Appreciable Contamination Threshold Values[2]	Location ID: VC 40 Sample Name: VC40-D15-18E Date: 2/10/2018		VC 40 VC40-D18-21.7E 2/10/2018		VC 41-ALT VC41-ALT-D12-15E 2/11/2018		VC 41-ALT VC41-ALT-D15-18E 2/11/2018		VC 41-ALT DUP7-D15-18E 2/11/2018		VC 41-ALT VC41-ALT-D18-19.4E 2/11/2018	
Metals by Method SW-846 6020A (mg/kg)														
Aluminum	18000*	N/A	**4300**		**3560**		**2380**		**2310**		**2880**		**2190**	
Antimony	9.3*	N/A	0.200	U	0.154	U	0.170	U	0.170	U	0.171	U	0.163	U
Arsenic	8.2	8.2	**3.56**		**2.60**		**1.99**		**1.93**		**2.27**		**1.90**	
Barium	48*	N/A	**3.86**	J	**3.45**	J	**6.13**		**6.75**		**8.09**		**6.03**	
Beryllium	N/A	N/A	**0.265**	J	**0.232**	J	**0.124**	J	**0.154**	J	**0.158**	J	**0.128**	J
Cadmium	1.2	1.2	**0.035**	J	**0.042**	J	0.033	U	0.033	U	0.033	U	0.032	U
Calcium	N/A	N/A	**170**	J	**241**	J	**1510**		**1420**		**1620**		**1600**	
Chromium, Total	81	81	**9.83**		**9.53**		**7.09**		**6.76**		**8.40**		**6.41**	
Cobalt	10*	N/A	**13.5**		**5.03**		**1.85**		**1.87**		**2.33**		**1.91**	
Copper	34	34	**7.35**	J	**8.44**	J	**2.45**	J	**2.02**	J	**2.48**	J	**1.88**	J
Iron	N/A	N/A	**9240**		**12500**		**5060**		**5240**		**6280**		**4860**	
Lead	47	47	**4.14**		**2.74**		**2.27**		**2.15**		**2.63**		**2.01**	
Magnesium	N/A	N/A	**1980**	J	**1680**	J	**1650**		**1680**		**2020**		**1520**	
Manganese	260*	N/A	**56.2**		**79.9**		**58.0**	J	**53.8**	J	**66.1**		**49.6**	J
Molybdenum	N/A	N/A	**0.689**	J	**0.227**	J	0.379	U	0.402	U	0.431	U	0.278	U
Nickel	21	21	**17.1**		**11.3**	J	**4.96**	J	**5.19**	J	**6.17**	J	**5.35**	J
Potassium	N/A	N/A	**409**	J	**418**	J	**850**		**835**		**958**		**711**	
Selenium	1*	N/A	**1.06**	J	0.862	U	0.953	U	0.949	U	0.958	U	0.911	U
Silver	1	1	0.056	UJ	0.056	U	0.062	U	0.061	U	0.062	U	0.059	U
Sodium	N/A	N/A	**937**	J	**855**	J	**2780**	J	**3200**	J	**3250**	J	**2550**	J
Strontium	N/A	N/A	**2.90**	J	**3.35**	J	**8.28**	J	**8.25**	J	**9.78**		**9.07**	
Thallium	N/A	N/A	**0.103**	J	0.059	U	0.065	U	0.065	U	0.065	U	0.062	U
Tin	N/A	N/A	**0.691**	J	**0.297**	J	0.330	U	0.250	U	0.269	U	0.306	U
Titanium	N/A	N/A	**90.2**		**112**		**124**		**104**		**120**		**113**	
Vanadium	57*	N/A	**12.3**		**11.8**		**8.08**		**7.75**		**9.03**		**7.09**	
Zinc	150	150	**21.4**	J	**17.5**	J	**12.6**	J	**12.2**	J	**15.6**	J	**11.8**	J
Mercury by Method SW-846 7474 (mg/kg)														
Mercury	0.15	0.15	0.002	U	0.002	U	0.002	U	0.002	U	0.002	U	0.002	U
Total Organic Carbon by Method SW-846 9060A (mg/kg)														
Total Organic Carbon	N/A	N/A	**638**		500	U	**908**		**1150**		**1055**		**853.5**	

Key at end of table.

Source: Ecology and Environment, Inc. 2018

Summary of Analytical Results for Vibracore Sediment Samples
Northeastern Supply Enhancement Project, New York and New Jersey

Analyte

Key:

Qualifiers

J = Estimated value

U = Not detected (method detection limit shown)

UJ = Estimated not detected value (method detection limit shown)

Other

µg/kg = micrograms per kilogram

mg/kg = milligrams per kilogram

pg/g = picograms per gram

-- = analyte not analyzed for

N/A = No standard or guidance value available.

Bold values denote a positive detection of the analyte.

Shaded values denote an exceedance of the NYSDEC screening crite

Underlined values denote an exceedance of the NJDEP screening crit

Underlined and shaded values denote an exceedance of both NYSDE

Notes:

'1. NJDEP Ecological Screening Criteria (2009). Screening criteria are for sediment, saline water criteria, effects range low. If an effect range low value is not available, the effects range medium value was used. The effects range medium values are denoted with *.

'2. The lowest value between the NYS TOGS 5.1.9 (2004) and NYS Screening and Assessment of Contaminated Sediment (2014) was used as the New York screening criteria.

'3. The sum of low molecular weight and high molecular weight PAHs was performed as recommended in the Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal (2016).

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D

Sediment Chemistry Laboratory Reports

If requested, electronic copies of the Sediment Chemistry Laboratory Reports will be provided.

E Data Usability Summary Reports

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Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: March 19, 2018	Completed by: Eridania Marte

The analytical data provided by the laboratory were reviewed for precision, accuracy, and completeness based on applicable sections of the following guidance:

- EPA Region 2 Data Validation Standard Operating Procedures.
  - *SOP NO. HW-33A, Rev. 0, Low/Medium Volatile Data Validation*
  - *SOP NO. HW-35A, Rev. 0, Semivolatile Data Validation*
  - *SOP NO. HW-36A, Rev. 0, Pesticide Data Validation*
  - *SOP NO. HW-37A, Rev. 0, Polychlorinated Biphenyl (PCB) Aroclor Data Validation*
  - *SOP NO. HW-3B, Rev. 0, ICP-MS Data Validation*
  - *SOP NO. HW-3C, Rev. 0, Mercury and Cyanide Data Validation*

Specific criteria for QC limits were obtained from the site specific QAPP: *Offshore Sampling and Analysis Plan / Quality Assurance Project Plan, Version 1.1, Northeast Supply Enhancement Project*. Compliance with the project QA program is indicated in the checklist and tables below. Any major or minor concerns affecting data usability are listed below. The checklist and tables also indicate whether data qualification is required and/or the type of qualifier assigned.

Reference:

Project ID	Lab Work Order	Laboratory
1000891.0020.05	L1804642	Alpha Analytical

Table 1: Sample List

SDG	Sample Name	Matrix	Lab ID	Sample Date	Sample Type	Original ID
L1804642	VC15-D15-18E	SE	L1804642-01	2/8/2018 12:34:00 PM	N	
L1804642	VC15-D18-21E	SE	L1804642-02	2/8/2018 12:34:00 PM	N	
L1804642	VC15-D21-24E	SE	L1804642-03	2/8/2018 12:34:00 PM	N	
L1804642	VC15-D24-25.1E	SE	L1804642-04	2/8/2018 12:34:00 PM	N	
L1804642	VC18-D15-18E	SE	L1804642-05	2/8/2018 3:00:00 PM	N	
L1804642	VC18-D18-21E	SE	L1804642-06	2/8/2018 3:00:00 PM	N	
L1804642	VC18-D21-24E	SE	L1804642-07	2/8/2018 3:00:00 PM	N	
L1804642	VC18-D24-27E	SE	L1804642-08	2/8/2018 3:00:00 PM	N	
L1804642	VC18-D27-29.3E	SE	L1804642-09	2/8/2018 3:00:00 PM	N	
L1804642	VC15-D15-18E MS	SQ	WG1089517-4	2/8/2018 12:34:00 PM	MS	
L1804642	RB-02082018	WH	L1804642-10	2/8/2018 4:30:00 PM	RB	
L1804642	RB-02082018 MS	WQ	WG1088773-3	2/8/2018 4:30:00 PM	MS	

Table 1A: Sample Test Summary

SDG	Matrix	Test Method	Prep Method	Number of Samples	Sample Type
L1804642	SE	6020A	3050B	9	N
L1804642	WH	6020A	SW3005A	1	RB
L1804642	SE	A2540G		9	N
L1804642	SE	E7474/SW846	METHOD	9	N
L1804642	WH	E7474/SW846	METHOD	1	RB
L1804642	SE	LLOYDKAHN		9	N

SDG	Matrix	Test Method	Prep Method	Number of Samples	Sample Type
L1804642	SE	SW8081B	SW3570	9	N
L1804642	WH	SW8081B	SW3510C	1	RB
L1804642	SE	SW8082A	SW3570	9	N
L1804642	WH	SW8082A	SW3510C	1	RB
L1804642	SE	SW8270DSIM	SW3570	9	N
L1804642	WH	SW8270DSIM	SW3510C	1	RB

Table 1B: Holding Time Exceedances

Method	Sample Name	Sample Date	Prep Date	Prep Hold Time	Analysis Date	Anal. Hold Time	Prep Exceedance	Analysis Exceedance
SW8081B	RB-02082018	2/8/2018	2/28/2018	19.72	3/1/2018	0.60	J Flag	
SW8082A	RB-02082018	2/8/2018	2/28/2018	19.72	2/28/2018	0.55	J Flag	

General Sample Information	
Do Samples and Analyses on COC check against Lab Sample Tracking Form?	Yes.
Did coolers arrive at lab between 2 and 6°C and in good condition as indicated on COC and Cooler Receipt Form?	Yes.
Frequency of Field QC Samples Correct? Field Duplicate - 1/20 samples MS/MSD – 1/20 samples Trip Blank - Every cooler with VOCs waters only Equipment Blank - 1/ set of samples per day?	Two field duplicates and two MS/MSDs were required to be collected over the course of the field sampling event; however, they were not included in this SDG. VOCs were not collected; trip blank not required. 1 equipment blank was collected.
Case narrative present and complete?	Yes.
Any holding time violations?	Yes. Due to surrogate failures by methods 8081B and 8082A, the equipment blank was re-extracted outside of the 7 day holding time. The surrogate recoveries were within control limits for the re-extraction; therefore, they were marked as reportable.

The following tables are presented at the end of this DUSR and provided summaries of results outside QC criteria:

- Method Blanks Results (Table 2)
- Surrogates Outside Limits (Table 3)
- MS/MSD/LR Outside Limits (Table 4)
- LCS/SRM Outside Limits (Table 5)
- Re-analysis Results (Table 6)
- Field Duplicate Results (Table 7)

Go to Tables List

Semi-volatile Organics by GC/MS SIM	
Description	**Notes and Qualifiers**
Any compounds present in method, trip, and rinsate blanks (see Table 2A)?	Yes.
For samples, if results are <5 times the blank or <10 times blank for common laboratory contaminants then "U" flag data.	Naphthalene was detected in method blank WG1088568-1. Eight sample results were less than 5X the blank detection. The results were U qualified as non-detect and the MDL was elevated to sample result. 2-Methylnaphthalene, acenaphthene, acenaphthylene, anthracene, benz(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, dibenz(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, and pyrene were detected in rinse blank sample RB-02082018. The analytes in all samples were J qualified as estimated.
Surrogate for method blanks and LCS within limits?	Yes.
Surrogate for samples and MS/MSD within limits? (See Table 3). Samples should re-analyzed if 1 base/neutral or 1 acid extractable surrogate has a recovery <10%. Matrix effects should be established.	Yes.
Laboratory QC frequency one blank and LCS with each batch and one set of MS/MSD per 20 samples?	Yes.
MS/MSD within QC criteria (see Table 4A and 4B)? If out and LCS is compliant, then J flag positive data in original sample due to matrix?	A client specific MS/MSD was not requested for this analysis.
LCS within QC criteria (see Table 5A and 5B)? If out, and the recovery high with no positive values, then no data qualification is required.	Yes.
Do internal standards areas and retention time meet criteria? If not was sample re-analyzed to establish matrix effects (see Table 6)?	Yes.
Is initial calibration for target compounds <15 %RSD or curve fit? Initial calibration verification <20%D?	Yes.
Is continuing calibration for target compounds < 20%D.	Yes.
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and dilutions is only one reportable result by flagged?	No.
Do field duplicate results show good precision for all compounds (see Table 7)?	Not applicable.

Pesticides by GC/ECD	
Description	**Notes and Qualifiers**
Are any compounds present in method and field blanks as noted on Table 2?	No.
For samples, if results are <5 times the blank then "U" flag data.	No qualification required.

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: March 19, 2018	Completed by: Eridania Marte

Pesticides by GC/ECD	
Description	**Notes and Qualifiers**
Are surrogates for method blanks and LCS within limits?	Yes.
Are surrogates for samples and MS/MSD within limits? (See Table 3). Matrix effects should be established.	No. Decachlorobiphenyl was recovered below the acceptance criteria in rinse sample RB-02082018. The sample was re-extracted outside of hold time with acceptable surrogate recoveries. The re-extracted results were marked as reportable and UJ qualified as estimated non-detect.
Is laboratory QC frequency one blank and LCS with each batch and one MS/MSD per 20 samples?	Yes.
Are MS/MSD within QC criteria (see Table 4A and 4B)? If out and LCS is compliant, then J flag positive data in original sample due to matrix.	A client specific MS/MSD was not requested for this analysis.
Is LCS within QC criteria (see Table 5A and 5B)? If out, and the recovery high with no positive values, then no data qualification is required.	Yes.
Is initial calibration for target compounds <20 %RSD or curve fit? Is initial calibration verification <25%D?	Yes.
Is continuing calibration for target compounds <20%D?	No. Alpha-BHC exhibited a %D outside of control limits in column 2 in CCV WG1089751-1. There were no associated sample results; therefore, no qualification of the data was made. Methyoxychlor exhibited a %D outside of control limits on column 1 in CCV WG1089372-1. There were no associated sample results; therefore, no qualification of the data was made.
Do internal standards areas meet criteria? If not was sample re-analyzed to establish matrix effects (see Table 6)?	The internal standards were not summarized in the report (the laboratory does not provide a summary form). There were no issues noted in the case narrative.
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and dilutions is only one reportable result by flagged?	Yes. Sample RB-02082018 was reanalyzed due to the surrogate failure noted above.
Spot check retention time windows and second column confirmations as complete.	Yes.
Spot check performance evaluation mixture? %Breakdown <20%	Yes.
Do field duplicate results show good precision for all compounds (see Table 7)?	Not applicable.

PCB Aroclors by GC/ECD	
Description	**Notes and Qualifiers**
Are any compounds present in method and field blanks as noted on Table 2?	No.
For samples, if results are <5 times the blank then "U" flag data.	No qualification required.

PCB Aroclors by GC/ECD	
Description	**Notes and Qualifiers**
Are surrogates for method blanks and LCS within limits?	The surrogate 2,4,5,6-tetrachloro-m-xylene (TMX) exhibited a %D outside of control limits on column 1 and 2 in the CCV WG1093673-1. The surrogates TMX and Decachlorobiphenyl (DCB) exhibited a %D outside of control limits on columns 1 and 2 in CCV WG1093673-2 The CCVs were associated with the re-extracted rinse blank sample. The sample results were already UJ qualified as estimated non-detect.
Are surrogates for samples and MS/MSD within limits? (See Table 3). Matrix effects should be established.	No. Decachlorobiphenyl was recovered below the acceptance criteria in rinse sample RB-02082018. The sample was re-extracted outside of hold time with acceptable surrogate recoveries. The re-extracted results were marked as reportable and UJ qualified as estimated non-detect.
Is laboratory QC frequency one blank and LCS with each batch and one MS/MSD per 20 samples?	Yes.
Are MS/MSD within QC criteria (see Table 4A and 4B)? If out and LCS is compliant, then J flag positive data in original sample due to matrix.	A client specific MS/MSD was not requested for this analysis.
Is LCS within QC criteria (see Table 5A and 5B)? If out, and the recovery high with no positive values, then no data qualification is required.	Yes.
Is initial calibration for target compounds <20 %RSD or curve fit? Is initial calibration verification <20%D?	No.
Is continuing calibration for target compounds <20%D?	All sample results were reported from column 1. The closing CCV WG1093673-2 exhibited an average of 20.4% D for Aroclor 1016 and an average of 22.9% D for Aroclor 1260 on column 1. The CCV was associated with the re-extracted rinse blank sample. The sample results were already UJ qualified as estimated non-detect.
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and dilutions is only one reportable result by flagged?	Yes. Sample RB-02082018 was reanalyzed due to the surrogate failure noted above.
Spot check retention time windows and second column confirmations as complete.	Yes.
Do field duplicate results show good precision for all compounds (see Table 7)?	Not applicable.

Metals & Mercury by ICP/MS and CVAA	
Description	**Notes and Qualifiers**
Are any compounds present in method and field blanks as noted on Table 2?	Yes.
For samples, if results are <5 times the blank then "U" flag data.	Tin was detected in method blank WG1088433-1. Six (out of nine) sample results were less than 5X the blank detection for tin. The results were U

Metals & Mercury by ICP/MS and CVAA	
Description	**Notes and Qualifiers**
	qualified as non-detect and the MDL was elevated to sample result. Titanium was detected in method blank WG1088773-1. The sample results associated with titanium were greater than 5X the blank detection or non-detect; therefore, no qualification was made. Mercury was detected in method blank WG1088775-1. The associated sample, RB-02082018, was less than 5X the blank detection. The result was U qualified as non-detect and the MDL was elevated to sample result. Mercury was detected in method blank WG1093026-1. The associated sample results were greater than 5X the blank detection or non-detect, no qualification was made. Aluminum, arsenic, barium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, nickel, potassium, sodium, strontium, titanium, and zinc were detected in rinse blank sample RB-02082018. The results in all associated samples were J qualified as estimated.
Is laboratory QC frequency one blank and LCS with each batch and one set of MS/MSD per 20 samples?	Yes.
Are MS/MSD within QC criteria (see Table 3)? If not, was a post digestion spike analyzed? QC limits are not applicable to sample results greater than 4 times spike amount.	No. Iron was recovered above the acceptance criteria in the MS for rinse sample RB-02082018. The sample result was J qualified as estimated.
Were elements recovered ≤30%? If so, "R" flag associated NDs.	No.
Is LCS within QC criteria (see Table 4)? If out, and the recovery is high with no positive values, then no data qualification is required.	Yes.
Is there one serial dilution per 20 samples? Flag all data reported with an "E" as "J".	Yes.
Spot check ICS recoveries 80-120%. Contact lab if unacceptable.	Yes.
Spot check ICV 90-110%. Contact lab if unacceptable.	Yes.
Spot check CCV 90-110%. Contact lab if unacceptable.	Yes.
Spot check ICB/CCB +/- RL. Contact lab if unacceptable.	Antimony and tin were detected in ICB R1047080-2, CCB R1047080-10, and CCB R1047080-12. The six positive detections for antimony and three positive detections for tin were U qualified as non-detect, and the MDL was elevated to the sample result. Antimony was detected in ICB R1048066-2, CCB R1048066-8 and CCB R1048066-10. The associated sample result was non-detect, therefore; no qualification was made. Strontium was detected

Metals & Mercury by ICP/MS and CVAA	
Description	**Notes and Qualifiers**
	in CCB R1048066-8; the associated result was greater than 5X the blank detection. No qualification was made. Mercury was detected in ICB R1051711-8 and CCB R1051711-10; the associated sample result was less than 5X blank detection. The result was U qualified as non-detect and the MDL was elevated to the sample result.
Are laboratory replicates within limits?	Yes.
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and dilutions is only one reportable result by flagged?	Yes. All samples were diluted for metals at a 10X dilution and mercury at a 5X dilution. There is no impact to data usability.
Do field duplicate results show good precision for all compounds (see Table 6)?	Not applicable.

Total Organic Carbon - Method Lloyd Khan	
Description	**Notes and Qualifiers**
Are any compounds present in method and field blanks as noted on Table 2?	No.
For samples, if results are <5 times the blank then "U" flag data.	No qualification required.
Is laboratory QC frequency one blank and LCS with each batch and one set of MS/MSD per 20 samples?	Yes.
Are MS/MSD within QC criteria (see Table 4)? QC limits are not applicable to sample results greater than 4 times spike amount.	Yes.
Is LCS and SRM within QC criteria (see Table 5)? If out, and the recovery high with no positive values, then no data qualification is required.	Yes.
Is initial calibration r ≥ 0.995 and slope ± 10% the historical curves? Is initial calibration verification 80-120% of True Value?	Yes.
Is continuing calibration for 80-120% of True Value?	Yes.
Spot check CCB < reporting limit. Contact lab if unacceptable.	The CCB results were acceptable.
Are laboratory duplicates within QC limits?	Yes.
Do field duplicate results show good precision for all compounds (see Table 7)?	Not applicable.

Summary of Findings

- 8270D: Naphthalene was detected in method blank WG1088568-1. Eight sample results were less than 5X the blank detection. The results were U qualified as non-detect and the MDL was elevated to sample result.

- 8270D: 2-Methylnaphthalene, acenaphthene, acenaphthylene, anthracene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, fluoranthene, fluorene, naphthalene, phenanthrene, and pyrene were detected in rinse blank sample RB-02082018. The analytes in all samples were J qualified as estimated.

- 8081B/8082A: Decachlorobiphenyl was recovered below the acceptance criteria for rinse sample RB-02082018. The criteria was achieved upon re-extraction outside of holding time. The re-extraction was reported and results were UJ qualified as estimated non-detect.

- 6020A: Tin was detected in method blank WG1088433-1. Six sample results were less than 5X the blank detection for tin. The results were U qualified as non-detect and the MDL was elevated to sample result.

- 6020A: Mercury was detected in method blank WG1088775-1. The associated sample RB-02082018 was less than 5X the blank detection. The result was U qualified as non-detect and the MDL was elevated to sample result.

- 6020A: Cadmium, calcium, copper, nickel, sodium, and zinc were detected in rinse blank sample RB-02082018. The analytes in all samples were J qualified as estimated.

- 6020A: Iron was recovered above the acceptance criteria in the MS for rinse sample RB-02082018. The sample result was J qualified as estimated.

- 6020A: Antimony and tin were present in ICB R1047080-2, the associated analytes were less than 5X blank detection; therefore, were U qualified as non-detect and the MDL was elevated to the sample result.

- 6020A: Mercury was present in ICB R1051711-8, the associated sample result was less than 5X blank detection; therefore, were U qualified as non-detect and the MDL was elevated to the sample result.

Table 2 – List of Positive Results for Blank Samples

Method	Sample Name	Matrix	Analyte	Result	Qualifier	Units	MDL	QL
6020A	WG1088433-1	LB	TIN, TOTAL	0.151	J	mg/kg	0.124	1.2
6020A	WG1088773-1	LB	TITANIUM, TOTAL	0.00012	J	mg/l	0.00007	0.0005
E7474/SW846	WG1088775-1	LB	MERCURY, TOTAL	0.00001	J	mg/l	0.00001	0.00005
E7474/SW846	WG1093026-1	LB	MERCURY, TOTAL	0.002	J	mg/kg	0.002	0.013
SW8270DSIM	WG1088568-1	LB	NAPHTHALENE	0.52	J	ug/kg	0.358	4
6020A	RB-02082018	RB	ALUMINUM, TOTAL	0.0846		mg/l	0.00327	0.01
6020A	RB-02082018	RB	ARSENIC, TOTAL	0.00042	J	mg/l	0.00016	0.0005
6020A	RB-02082018	RB	BARIUM, TOTAL	0.00514		mg/l	0.00017	0.0005
6020A	RB-02082018	RB	CADMIUM, TOTAL	0.00023		mg/l	0.00005	0.0002
6020A	RB-02082018	RB	CALCIUM, TOTAL	1.58		mg/l	0.0394	0.1
6020A	RB-02082018	RB	CHROMIUM, TOTAL	0.00492		mg/l	0.00017	0.0005
6020A	RB-02082018	RB	COBALT, TOTAL	0.00057		mg/l	0.00016	0.0005
6020A	RB-02082018	RB	COPPER, TOTAL	0.00589		mg/l	0.00038	0.001
6020A	RB-02082018	RB	IRON, TOTAL	3.25		mg/l	0.0191	0.05
6020A	RB-02082018	RB	LEAD, TOTAL	0.00142		mg/l	0.00034	0.001
6020A	RB-02082018	RB	MAGNESIUM, TOTAL	0.473		mg/l	0.0242	0.07
6020A	RB-02082018	RB	MANGANESE, TOTAL	0.07958		mg/l	0.00044	0.001
6020A	RB-02082018	RB	NICKEL, TOTAL	0.01105		mg/l	0.00055	0.002
6020A	RB-02082018	RB	POTASSIUM, TOTAL	0.194		mg/l	0.0309	0.1
6020A	RB-02082018	RB	SODIUM, TOTAL	6.45		mg/l	0.0293	0.1
6020A	RB-02082018	RB	STRONTIUM, TOTAL	0.008		mg/l	0.00003	0.0005
6020A	RB-02082018	RB	TITANIUM, TOTAL	0.00492		mg/l	0.00007	0.0005
6020A	RB-02082018	RB	ZINC, TOTAL	0.0506		mg/l	0.00341	0.01
E7474/SW846	RB-02082018	RB	MERCURY, TOTAL	0.00001	J	mg/l	0.00001	0.00005
SW8270DSIM	RB-02082018	RB	2-METHYLNAPHTHALENE	31.4		ng/l	1.07	10.1
SW8270DSIM	RB-02082018	RB	ACENAPHTHENE	33.6		ng/l	1.63	10.1

Method	Sample Name	Matrix	Analyte	Result	Qualifier	Units	MDL	QL
SW8270DSIM	RB-02082018	RB	ACENAPHTHYLENE	20.7		ng/l	1.79	10.1
SW8270DSIM	RB-02082018	RB	ANTHRACENE	113		ng/l	1.96	10.1
SW8270DSIM	RB-02082018	RB	BENZ(A)ANTHRACENE	130		ng/l	1.75	10.1
SW8270DSIM	RB-02082018	RB	BENZO(A)PYRENE	83.8		ng/l	0.848	10.1
SW8270DSIM	RB-02082018	RB	BENZO(B)FLUORANTHENE	87.9		ng/l	1.49	10.1
SW8270DSIM	RB-02082018	RB	BENZO(GHI)PERYLENE	52.7		ng/l	1.32	10.1
SW8270DSIM	RB-02082018	RB	BENZO(K)FLUORANTHENE	78.7		ng/l	1.18	10.1
SW8270DSIM	RB-02082018	RB	CHRYSENE	128		ng/l	0.945	10.1
SW8270DSIM	RB-02082018	RB	DIBENZ(A,H)ANTHRACENE	14.6		ng/l	0.692	10.1
SW8270DSIM	RB-02082018	RB	FLUORANTHENE	442		ng/l	1.5	10.1
SW8270DSIM	RB-02082018	RB	FLUORENE	100		ng/l	1.75	10.1
SW8270DSIM	RB-02082018	RB	INDENO(1,2,3-CD)PYRENE	59.9		ng/l	0.538	10.1
SW8270DSIM	RB-02082018	RB	NAPHTHALENE	59.5		ng/l	1.79	10.1
SW8270DSIM	RB-02082018	RB	PHENANTHRENE	659		ng/l	1.91	10.1
SW8270DSIM	RB-02082018	RB	PYRENE	280		ng/l	1.54	10.1
6020A	R1047080-2	ICB	ANTIMONY	0.913	J	ug/l		
6020A	R1047080-2	ICB	TIN	1.20	J	ug/l		
6020A	R1048066-2	ICB	ANTIMONY	0.909	J	ug/l		
6020A	R1048066-8	CCB	STRONIUM	0.0465	J	ug/l		
E7474/SW846	R1051711-8	ICB	MERCURY, TOTAL	0.00000825	J	mg/l		

Table 2A – List of Samples Qualified for Blank Contamination

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	Units	MDL	QL	Affected Sample	Sample Flag
6020A	WG1088433-1	SE	TIN, TOTAL	0.151	0.736	J	mg/kg	0.245	2.37	VC15-D15-18E	U Flag
6020A	WG1088433-1	SE	TIN, TOTAL	0.151	0.627	J	mg/kg	0.236	2.28	VC15-D24-25.1E	U Flag
6020A	WG1088433-1	SE	TIN, TOTAL	0.151	0.66	J	mg/kg	0.241	2.33	VC18-D15-18E	U Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	Units	MDL	QL	Affected Sample	Sample Flag
6020A	WG1088433-1	SE	TIN, TOTAL	0.151	0.631	J	mg/kg	0.223	2.16	VC18-D18-21E	U Flag
6020A	WG1088433-1	SE	TIN, TOTAL	0.151	0.754	J	mg/kg	0.221	2.14	VC18-D21-24E	U Flag
6020A	WG1088433-1	SE	TIN, TOTAL	0.151	0.637	J	mg/kg	0.201	1.95	VC18-D24-27E	U Flag
6020A	R1047080-2	ICB	ANTIMONY	0.913	0.446	J	ug/l	0.267	3.16	VC15-D15-18E	U Flag
6020A	R1047080-2	ICB	ANTIMONY	0.913	0.490	J	ug/l	0.236	2.8	VC15-D18-21E	U Flag
6020A	R1047080-2	ICB	ANTIMONY	0.913	0.344	J	ug/l	0.248	2.94	VC15-D21-24E	U Flag
6020A	R1047080-2	ICB	ANTIMONY	0.913	0.464	J	ug/l	0.262	3.11	VC18-D15-18E	U Flag
6020A	R1047080-2	ICB	ANTIMONY	0.913	0.362	J	ug/l	0.241	2.85	VC18-D21-24E	U Flag
6020A	R1047080-2	ICB	ANTIMONY	0.913	0.396	J	ug/l	0.244	2.88	VC18-D27-29.3E	U Flag
6020A	R1047080-2	ICB	TIN	1.20	1.164	J	ug/l	0.217	2.1	VC15-D18-21E	U Flag
6020A	R1047080-2	ICB	TIN	1.20	1.099	J	ug/l	0.228	2.2	VC15-D21-24E	U Flag
6020A	R1047080-2	ICB	TIN	1.20	1.298	J	ug/l	0.224	2.16	VC18-D27-29.3E	U Flag
E7474/SW846	R1051711-8	ICB	MERCURY, TOTAL	0.00825	0.0367	J	ug/l	0.003	0.021	VC15-D21-24E	U Flag
E7474/SW846	WG1088775-1	WH	MERCURY, TOTAL	0.00001	0.00001	J	mg/l	0.00001	0.00005	RB-02082018	U Flag
SW8270DSIM	WG1088568-1	SE	NAPHTHALENE	0.52	1.3	J	ug/kg	0.694	7.75	VC15-D15-18E	U Flag
SW8270DSIM	WG1088568-1	SE	NAPHTHALENE	0.52	2.13	J	ug/kg	0.642	7.17	VC15-D18-21E	U Flag
SW8270DSIM	WG1088568-1	SE	NAPHTHALENE	0.52	1.91	J	ug/kg	0.656	7.34	VC15-D21-24E	U Flag
SW8270DSIM	WG1088568-1	SE	NAPHTHALENE	0.52	2.43	J	ug/kg	0.707	7.9	VC15-D24-25.1E	U Flag
SW8270DSIM	WG1088568-1	SE	NAPHTHALENE	0.52	1.11	J	ug/kg	0.611	6.83	VC18-D18-21E	U Flag
SW8270DSIM	WG1088568-1	SE	NAPHTHALENE	0.52	2	J	ug/kg	0.639	7.14	VC18-D21-24E	U Flag
SW8270DSIM	WG1088568-1	SE	NAPHTHALENE	0.52	2.59	J	ug/kg	0.59	6.59	VC18-D24-27E	U Flag
SW8270DSIM	WG1088568-1	SE	NAPHTHALENE	0.52	1.07	J	ug/kg	0.619	6.91	VC18-D27-29.3E	U Flag
SW8270DSIM	RB-02082018	SE	2-METHYLNAPHTHALENE	31.4	1.28	J	ug/kg	0.889	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	2-METHYLNAPHTHALENE	31.4	0.969	J	ug/kg	0.91	7.34	VC15-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	2-METHYLNAPHTHALENE	31.4	1.45	J	ug/kg	0.979	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	2-METHYLNAPHTHALENE	31.4	2.18	J	ug/kg	0.94	7.58	VC18-D15-18E	J Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	Units	MDL	QL	Affected Sample	Sample Flag
SW8270DSIM	RB-02082018	SE	2-METHYLNAPHTHALENE	31.4	1.23	J	ug/kg	0.885	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	2-METHYLNAPHTHALENE	31.4	0.873	J	ug/kg	0.817	6.59	VC18-D24-27E	J Flag
SW8270DSIM	RB-02082018	SE	ACENAPHTHENE	33.6	0.866	J	ug/kg	0.803	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	ACENAPHTHENE	33.6	1.18	J	ug/kg	0.885	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	ACENAPHTHENE	33.6	0.803	J	ug/kg	0.799	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	ACENAPHTHYLENE	20.7	0.639	J	ug/kg	0.48	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	ACENAPHTHYLENE	20.7	0.662	J	ug/kg	0.529	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	ACENAPHTHYLENE	20.7	0.627	J	ug/kg	0.478	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	ANTHRACENE	113	2.05	J	ug/kg	0.961	7.75	VC15-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	ANTHRACENE	113	1.6	J	ug/kg	0.889	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	ANTHRACENE	113	1.65	J	ug/kg	0.91	7.34	VC15-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	ANTHRACENE	113	1.9	J	ug/kg	0.979	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	ANTHRACENE	113	1.3	J	ug/kg	0.94	7.58	VC18-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	ANTHRACENE	113	1.1	J	ug/kg	0.846	6.83	VC18-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	ANTHRACENE	113	1.45	J	ug/kg	0.885	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	ANTHRACENE	113	1.11	J	ug/kg	0.817	6.59	VC18-D24-27E	J Flag
SW8270DSIM	RB-02082018	SE	ANTHRACENE	113	1.3	J	ug/kg	0.857	6.91	VC18-D27-29.3E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(B)FLUORANTHENE	87.9	2.45	J	ug/kg	0.806	7.75	VC15-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(B)FLUORANTHENE	87.9	1.76	J	ug/kg	0.746	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(B)FLUORANTHENE	87.9	2.35	J	ug/kg	0.763	7.34	VC15-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(B)FLUORANTHENE	87.9	2.01	J	ug/kg	0.821	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(B)FLUORANTHENE	87.9	1.95	J	ug/kg	0.789	7.58	VC18-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(B)FLUORANTHENE	87.9	1.93	J	ug/kg	0.71	6.83	VC18-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(B)FLUORANTHENE	87.9	1.76	J	ug/kg	0.742	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(B)FLUORANTHENE	87.9	1.49	J	ug/kg	0.685	6.59	VC18-D24-27E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(B)FLUORANTHENE	87.9	1.72	J	ug/kg	0.719	6.91	VC18-D27-29.3E	J Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	Units	MDL	QL	Affected Sample	Sample Flag
SW8270DSIM	RB-02082018	SE	BENZO(GHI)PERYLENE	52.7	1.54	J	ug/kg	0.639	7.75	VC15-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(GHI)PERYLENE	52.7	1.14	J	ug/kg	0.591	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(GHI)PERYLENE	52.7	1.51	J	ug/kg	0.604	7.34	VC15-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(GHI)PERYLENE	52.7	1.45	J	ug/kg	0.651	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(GHI)PERYLENE	52.7	1.18	J	ug/kg	0.625	7.58	VC18-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(GHI)PERYLENE	52.7	1.23	J	ug/kg	0.562	6.83	VC18-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(GHI)PERYLENE	52.7	1.22	J	ug/kg	0.588	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(GHI)PERYLENE	52.7	1.03	J	ug/kg	0.543	6.59	VC18-D24-27E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(GHI)PERYLENE	52.7	1.24	J	ug/kg	0.57	6.91	VC18-D27-29.3E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(K)FLUORANTHENE	78.7	0.908	J	ug/kg	0.814	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	BENZO(K)FLUORANTHENE	78.7	0.729	J	ug/kg	0.712	6.91	VC18-D27-29.3E	J Flag
SW8270DSIM	RB-02082018	SE	CHRYSENE	128	2.15	J	ug/kg	0.681	7.75	VC15-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	CHRYSENE	128	1.82	J	ug/kg	0.63	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	CHRYSENE	128	2.1	J	ug/kg	0.645	7.34	VC15-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	CHRYSENE	128	2	J	ug/kg	0.694	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	CHRYSENE	128	1.78	J	ug/kg	0.667	7.58	VC18-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	CHRYSENE	128	1.62	J	ug/kg	0.6	6.83	VC18-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	CHRYSENE	128	1.77	J	ug/kg	0.627	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	CHRYSENE	128	1.36	J	ug/kg	0.579	6.59	VC18-D24-27E	J Flag
SW8270DSIM	RB-02082018	SE	CHRYSENE	128	1.61	J	ug/kg	0.608	6.91	VC18-D27-29.3E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORANTHENE	442	4.39	J	ug/kg	1.42	7.75	VC15-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORANTHENE	442	3.41	J	ug/kg	1.31	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORANTHENE	442	4.11	J	ug/kg	1.34	7.34	VC15-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORANTHENE	442	3.99	J	ug/kg	1.44	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORANTHENE	442	3.3	J	ug/kg	1.39	7.58	VC18-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORANTHENE	442	3.1	J	ug/kg	1.25	6.83	VC18-D18-21E	J Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	Units	MDL	QL	Affected Sample	Sample Flag
SW8270DSIM	RB-02082018	SE	FLUORANTHENE	442	3.46	J	ug/kg	1.31	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORANTHENE	442	2.8	J	ug/kg	1.21	6.59	VC18-D24-27E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORANTHENE	442	3.04	J	ug/kg	1.26	6.91	VC18-D27-29.3E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORENE	100	3.12	J	ug/kg	0.518	7.75	VC15-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORENE	100	2.84	J	ug/kg	0.479	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORENE	100	3.09	J	ug/kg	0.49	7.34	VC15-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORENE	100	3.73	J	ug/kg	0.528	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORENE	100	2.3	J	ug/kg	0.507	7.58	VC18-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORENE	100	1.96	J	ug/kg	0.456	6.83	VC18-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORENE	100	2.73	J	ug/kg	0.477	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORENE	100	2.09	J	ug/kg	0.44	6.59	VC18-D24-27E	J Flag
SW8270DSIM	RB-02082018	SE	FLUORENE	100	2.33	J	ug/kg	0.462	6.91	VC18-D27-29.3E	J Flag
SW8270DSIM	RB-02082018	SE	NAPHTHALENE	59.5	16		ug/kg	0.679	7.58	VC18-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	PHENANTHRENE	659	4.3	J	ug/kg	0.915	7.75	VC15-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	PHENANTHRENE	659	3.67	J	ug/kg	0.846	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	PHENANTHRENE	659	4.08	J	ug/kg	0.866	7.34	VC15-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	PHENANTHRENE	659	4.93	J	ug/kg	0.932	7.9	VC15-D24-25.1E	J Flag
SW8270DSIM	RB-02082018	SE	PHENANTHRENE	659	3.1	J	ug/kg	0.895	7.58	VC18-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	PHENANTHRENE	659	3.03	J	ug/kg	0.806	6.83	VC18-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	PHENANTHRENE	659	3.95	J	ug/kg	0.842	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	PHENANTHRENE	659	2.89	J	ug/kg	0.778	6.59	VC18-D24-27E	J Flag
SW8270DSIM	RB-02082018	SE	PHENANTHRENE	659	2.93	J	ug/kg	0.816	6.91	VC18-D27-29.3E	J Flag
SW8270DSIM	RB-02082018	SE	PYRENE	280	3.21	J	ug/kg	0.791	7.75	VC15-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	PYRENE	280	1.85	J	ug/kg	0.731	7.17	VC15-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	PYRENE	280	2.04	J	ug/kg	0.748	7.34	VC15-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	PYRENE	280	1.96	J	ug/kg	0.806	7.9	VC15-D24-25.1E	J Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	Units	MDL	QL	Affected Sample	Sample Flag
SW8270DSIM	RB-02082018	SE	PYRENE	280	1.63	J	ug/kg	0.774	7.58	VC18-D15-18E	J Flag
SW8270DSIM	RB-02082018	SE	PYRENE	280	1.72	J	ug/kg	0.696	6.83	VC18-D18-21E	J Flag
SW8270DSIM	RB-02082018	SE	PYRENE	280	1.82	J	ug/kg	0.728	7.14	VC18-D21-24E	J Flag
SW8270DSIM	RB-02082018	SE	PYRENE	280	1.42	J	ug/kg	0.672	6.59	VC18-D24-27E	J Flag
SW8270DSIM	RB-02082018	SE	PYRENE	280	1.51	J	ug/kg	0.705	6.91	VC18-D27-29.3E	J Flag
6020A	RB-02082018	SE	CADMIUM, TOTAL	0.23	0.131	J	ng/mL	0.052	0.395	VC15-D15-18E	J Flag
6020A	RB-02082018	SE	CADMIUM, TOTAL	0.23	0.173	J	ng/mL	0.046	0.35	VC15-D18-21E	J Flag
6020A	RB-02082018	SE	CADMIUM, TOTAL	0.23	0.173	J	ng/mL	0.048	0.367	VC15-D21-24E	J Flag
6020A	RB-02082018	SE	CADMIUM, TOTAL	0.23	0.13	J	ng/mL	0.05	0.38	VC15-D24-25.1E	J Flag
6020A	RB-02082018	SE	CADMIUM, TOTAL	0.23	0.163	J	ng/mL	0.051	0.388	VC18-D15-18E	J Flag
6020A	RB-02082018	SE	CADMIUM, TOTAL	0.23	0.158	J	ng/mL	0.047	0.359	VC18-D18-21E	J Flag
6020A	RB-02082018	SE	CADMIUM, TOTAL	0.23	0.158	J	ng/mL	0.047	0.356	VC18-D21-24E	J Flag
6020A	RB-02082018	SE	CADMIUM, TOTAL	0.23	0.197	J	ng/mL	0.043	0.325	VC18-D24-27E	J Flag
6020A	RB-02082018	SE	CADMIUM, TOTAL	0.23	0.148	J	ng/mL	0.048	0.361	VC18-D27-29.3E	J Flag
6020A	RB-02082018	SE	CALCIUM, TOTAL	1580	6488		ng/mL	118	971	VC18-D15-18E	J Flag
6020A	RB-02082018	SE	CALCIUM, TOTAL	1580	6514		ng/mL	120	986	VC15-D15-18E	J Flag
6020A	RB-02082018	SE	COPPER, TOTAL	5.89	16.44		ng/mL	0.383	3.95	VC15-D15-18E	J Flag
6020A	RB-02082018	SE	COPPER, TOTAL	5.89	19.84		ng/mL	0.339	3.5	VC15-D18-21E	J Flag
6020A	RB-02082018	SE	COPPER, TOTAL	5.89	19.54		ng/mL	0.356	3.67	VC15-D21-24E	J Flag
6020A	RB-02082018	SE	COPPER, TOTAL	5.89	16.14		ng/mL	0.369	3.8	VC15-D24-25.1E	J Flag
6020A	RB-02082018	SE	COPPER, TOTAL	5.89	16.64		ng/mL	0.377	3.88	VC18-D15-18E	J Flag
6020A	RB-02082018	SE	COPPER, TOTAL	5.89	16.61		ng/mL	0.348	3.59	VC18-D18-21E	J Flag
6020A	RB-02082018	SE	COPPER, TOTAL	5.89	18.49		ng/mL	0.346	3.56	VC18-D21-24E	J Flag
6020A	RB-02082018	SE	COPPER, TOTAL	5.89	18.82		ng/mL	0.315	3.25	VC18-D24-27E	J Flag
6020A	RB-02082018	SE	COPPER, TOTAL	5.89	17.55		ng/mL	0.35	3.61	VC18-D27-29.3E	J Flag
6020A	RB-02082018	SE	NICKEL, TOTAL	11.05	34.18		ng/mL	0.527	1.97	VC15-D15-18E	J Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qualifier	Units	MDL	QL	Affected Sample	Sample Flag
6020A	RB-02082018	SE	NICKEL, TOTAL	11.05	41.3		ng/mL	0.467	1.75	VC15-D18-21E	J Flag
6020A	RB-02082018	SE	NICKEL, TOTAL	11.05	41.4		ng/mL	0.49	1.83	VC15-D21-24E	J Flag
6020A	RB-02082018	SE	NICKEL, TOTAL	11.05	34.62		ng/mL	0.508	1.9	VC15-D24-25.1E	J Flag
6020A	RB-02082018	SE	NICKEL, TOTAL	11.05	35.17		ng/mL	0.519	1.94	VC18-D15-18E	J Flag
6020A	RB-02082018	SE	NICKEL, TOTAL	11.05	35.18		ng/mL	0.48	1.8	VC18-D18-21E	J Flag
6020A	RB-02082018	SE	NICKEL, TOTAL	11.05	38.9		ng/mL	0.476	1.78	VC18-D21-24E	J Flag
6020A	RB-02082018	SE	NICKEL, TOTAL	11.05	39.73		ng/mL	0.434	1.62	VC18-D24-27E	J Flag
6020A	RB-02082018	SE	NICKEL, TOTAL	11.05	36.37		ng/mL	0.482	1.8	VC18-D27-29.3E	J Flag
6020A	RB-02082018	SE	SODIUM, TOTAL	6450	11747		ng/mL	23.1	296	VC15-D15-18E	J Flag
6020A	RB-02082018	SE	SODIUM, TOTAL	6450	11734		ng/mL	20.5	262	VC15-D18-21E	J Flag
6020A	RB-02082018	SE	SODIUM, TOTAL	6450	11890		ng/mL	21.5	275	VC15-D21-24E	J Flag
6020A	RB-02082018	SE	SODIUM, TOTAL	6450	11089		ng/mL	22.3	285	VC15-D24-25.1E	J Flag
6020A	RB-02082018	SE	SODIUM, TOTAL	6450	13313		ng/mL	22.8	291	VC18-D15-18E	J Flag
6020A	RB-02082018	SE	SODIUM, TOTAL	6450	12388		ng/mL	21	269	VC18-D18-21E	J Flag
6020A	RB-02082018	SE	SODIUM, TOTAL	6450	12886		ng/mL	20.9	267	VC18-D21-24E	J Flag
6020A	RB-02082018	SE	SODIUM, TOTAL	6450	11592		ng/mL	19	244	VC18-D24-27E	J Flag
6020A	RB-02082018	SE	SODIUM, TOTAL	6450	11400		ng/mL	21.1	270	VC18-D27-29.3E	J Flag
6020A	RB-02082018	SE	ZINC, TOTAL	50.6	94.73		ng/mL	5.13	19.7	VC15-D15-18E	J Flag
6020A	RB-02082018	SE	ZINC, TOTAL	50.6	117.45		ng/mL	4.55	17.5	VC15-D18-21E	J Flag
6020A	RB-02082018	SE	ZINC, TOTAL	50.6	116.27		ng/mL	4.77	18.3	VC15-D21-24E	J Flag
6020A	RB-02082018	SE	ZINC, TOTAL	50.6	93.89		ng/mL	4.94	19	VC15-D24-25.1E	J Flag
6020A	RB-02082018	SE	ZINC, TOTAL	50.6	98.53		ng/mL	5.05	19.4	VC18-D15-18E	J Flag
6020A	RB-02082018	SE	ZINC, TOTAL	50.6	102.39		ng/mL	4.67	18	VC18-D18-21E	J Flag
6020A	RB-02082018	SE	ZINC, TOTAL	50.6	110.75		ng/mL	4.64	17.8	VC18-D21-24E	J Flag
6020A	RB-02082018	SE	ZINC, TOTAL	50.6	110.13		ng/mL	4.22	16.2	VC18-D24-27E	J Flag
6020A	RB-02082018	SE	ZINC, TOTAL	50.6	104.4		ng/mL	4.69	18	VC18-D27-29.3E	J Flag

Table 3 – List of Samples with Surrogates outside Control Limits

Method	Sample ID	Sample Type	Analyte	Rec %	Low Limit	High Limit	Dil Fac	Qualifier
SW8082A	RB-02082018	RB	DECACHLOROBIPHENYL	21	30	150	1	UJ Flag
SW8081B	RB-02082018	RB	DECACHLOROBIPHENYL	22	30	150	1	UJ Flag

Table 4 - List MS/MSD Recoveries outside Control Limits

Method	Parent Sample	Analyte	Dil Fac	Unit	MS	Low Limit	High Limit	Qualifier
6020A	RB-02082018	IRON, TOTAL	10	mg/l	133	75	125	J Flag

Table 4A – List RPDs outside Control Limits
None.

Table 4B – List of Laboratory Replicate outside Control Limits
None.

Table 5 - List LCS Recoveries outside Control Limits
None.

Table 5A – List RPDs outside Control Limits
None.

Table 5B - List SRM Recoveries outside Control Limits
None.

Table 6 –Samples that were Reanalyzed/Diluted

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
VC15-D15-18E	SE	6020A	N	N	1	10	INITIAL	Silver results non-detect and reported with elevated reporting limits.
VC15-D15-18E	SE	6020A	N	Y	25	10	INITIAL	
VC15-D15-18E	SE	E7474/SW846	N	Y	1	5	INITIAL	

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
VC15-D18-21E	SE	6020A	N	N	1	10	INITIAL	Silver results non-detect and reported with elevated reporting limits.
VC15-D18-21E	SE	6020A	N	Y	25	10	INITIAL	
VC15-D18-21E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC15-D21-24E	SE	6020A	N	N	1	10	INITIAL	Silver results non-detect and reported with elevated reporting limits.
VC15-D21-24E	SE	6020A	N	Y	25	10	INITIAL	
VC15-D21-24E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC15-D24-25.1E	SE	6020A	N	N	2	10	INITIAL	Silver and antimony results non-detect and reported with elevated reporting limits.
VC15-D24-25.1E	SE	6020A	N	Y	24	10	INITIAL	
VC15-D24-25.1E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC18-D15-18E	SE	6020A	N	N	1	10	INITIAL	Silver results non-detect and reported with elevated reporting limits.
VC18-D15-18E	SE	6020A	N	Y	25	10	INITIAL	
VC18-D15-18E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC18-D18-21E	SE	6020A	N	N	2	10	INITIAL	Silver and antimony results non-detect and reported with elevated reporting limits.
VC18-D18-21E	SE	6020A	N	Y	24	10	INITIAL	
VC18-D18-21E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC18-D21-24E	SE	6020A	N	N	1	10	INITIAL	Silver results non-detect and reported with elevated reporting limits.
VC18-D21-24E	SE	6020A	N	Y	25	10	INITIAL	
VC18-D21-24E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC18-D24-27E	SE	6020A	N	N	2	10	INITIAL	Silver and antimony results non-detect and reported with elevated reporting limits.

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
VC18-D24-27E	SE	6020A	N	Y	24	10	INITIAL	
VC18-D24-27E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC18-D27-29.3E	SE	6020A	N	N	1	10	INITIAL	Silver results non-detect and reported with elevated reporting limits.
VC18-D27-29.3E	SE	6020A	N	Y	25	10	INITIAL	
VC18-D27-29.3E	SE	E7474/SW846	N	Y	1	5	INITIAL	

Table 7 – Summary of Field Duplicate Results
N/A

Acronym List and Table Key:

BD	=	blank spike duplicate
BS	=	blank spike
COC	=	chain of custody
CVAA	=	cold vapor atomic absorption
DUSR	=	data usability summary report
FD	=	field duplicate sample
GC/ECD	=	gas chromatography / electron capture detection
GC/MS	=	gas chromatography / mass spectrometry
ICP/MS	=	Inductively coupled / mass spectrometry
LCS	=	laboratory control sample
LCSD	=	laboratory control sample duplicate
LR	=	laboratory replicate
MB	=	method blank
MS	=	matrix spike
MSD	=	matrix spike duplicate
N	=	normal field sample
NC	=	not calculated
ND	=	not detected

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: March 19, 2018	Completed by: Eridania Marte

Acronym List and Table Key:

NJDEP	=	New Jersey Department of Environmental Protection
NYSDEC	=	New York State Department of Environmental Conservation
PCB	=	polychlorinated biphenyl
PQL	=	practical quantitation limit
QA	=	quality assurance
QAPP	=	quality assurance project plan
QC	=	quality control
RB	=	rinsate blank sample
RPD	=	relative percent difference
SDG	=	sample delivery group
SRM	=	standard reference material
SVOC	=	semi-volatile organic compounds

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: April 18, 2018	Completed by: Eridania Marte

The analytical data provided by the laboratory were reviewed for precision, accuracy, and completeness based on applicable sections of the following:

- Standard Operating Procedure For The Analysis Of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans (PCDDs/PCDFs) by High-Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)

Specific criteria for QC limits were obtained from the site specific QAPP: *Offshore Sampling and Analysis Plan / Quality Assurance Project Plan, Version 1.1, Northeast Supply Enhancement Project*. Compliance with the project QA program is indicated in the checklist and tables below. Any major or minor concerns affecting data usability are listed below. The checklist and tables also indicate whether data qualification is required and/or the type of qualifier assigned.

Reference:

Project ID	Lab Work Order	Laboratory
1000891.0020.05	L1804795/WO11985	Alpha Analytical/Cape Fear Analytical

Table 1 - Sample Listing

SDG	Sample Name	Matrix	Lab ID	Sample Date	Sample Type	Original ID
L1804795	DUP6-D18-21E	SE	10235001	2/6/2018 7:35	N	
L1804795	RB-02102018	WH	10235002	2/13/2018 8:00	RB	
L1804795	VC16-D15-18E	SE	10235003	2/6/2018 7:35	N	
L1804795	VC16-D18-21E	SE	10235004	2/13/2018 8:00	N	
L1804795	VC16-D21-24E	SE	10235005	2/6/2018 7:35	N	
L1804795	VC16-D24-27.9E	SE	10235006	2/13/2018 8:00	N	

Table 1A: Sample Test Summary

SDG	Matrix	Test Method	Prep Method	Number of Samples	Sample Type
L1804795	SE	1613B	SW3540C	5	N
L1804795	WH	1613B	SW3520C	1	RB

General Sample Information	
Do Samples and Analyses on COC check against Lab Sample Tracking Form?	Yes.
Did coolers arrive at lab between 2 and 6°C and in good condition as indicated on COC and Cooler Receipt Form?	Yes
Frequency of Field QC Samples Correct? Field Duplicate - 1/20 samples MS/MSD – 1/20 samples Trip Blank - Every cooler with VOCs waters only Equipment Blank - 1/ set of samples per day?	Yes. 1 field duplicate sample was collected. 1 MS/MSD sample was collected. Trip blank is not applicable. 1 equipment blank was collected.
Case narrative present and complete?	Yes
Any holding time violations?	No

The following tables are presented at the end of this DUSR and provided summaries of results outside QC criteria:

- Method Blanks Results (Table 2)
- Surrogates Outside Limits (Table 3)
- MS/MSD Outside Limits (Table 4)
- LCS Outside Limits (Table 5)
- Re-analysis Results (Table 6)
- Field Duplicate Results (Table 7)

Go to Tables List

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: April 18, 2018	Completed by: Eridania Marte

Dioxins/Furans by HRGC/HRMS	
Description	**Notes and Qualifiers**
Any compounds present in method, trip, and rinsate blanks (see Table 2A)?	Yes.
For samples, if results are <5 times the blank or <10 times blank for common laboratory contaminants then "U" flag data.	1,2,3,4,6,7,8,9-OCDD was detected in the method blank for batch 36977, which was associated with the rinse blank sample RB-02102018. The sample results were ND; therefore, no qualification was made.
Surrogate for method blanks and LCS within limits?	Yes.
Surrogate for samples and MS/MSD within limits? (See Table 3). Matrix effects should be established.	Yes.
Laboratory QC frequency one blank and LCS with each batch and one set of MS/MSD per 20 samples?	Yes.
MS/MSD recoveries (see Lab SOP) and MS/MSD RPDs (<20%) within QC criteria (see Table 4A and 4B)? If out and LCS is compliant, then J flag positive data in original sample due to matrix?	No. 1,2,3,4,6,7,8,9-OCDD was recovered above the acceptance criteria in the MS and MSD for sample VC16-D21-24E. The parent sample result was J qualified as estimated.
LCS within QC criteria (see Table 5A and 5B)? If out, and the recovery high with no positive values, then no data qualification is required.	Yes.
Is initial calibration for target compounds <20 %RSD or curve fit and for labeled compounds < 35% RSD?	Yes.
Is continuing calibration for target compounds within QC limits (Table 8 of SOP)?	Yes.
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and dilutions is only one reportable result listed?	No.

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: April 18, 2018	Completed by: Eridania Marte

Dioxins/Furans by HRGC/HRMS	
Description	**Notes and Qualifiers**
Do field duplicate results show good precision for all compounds (see Table 7)?	The duplicate precision for Total Tetrachlorodibenzofuran and Total Tetrachlorodibenzo-p-dioxin were not calculated due to sample being detected in one sample and not the other. The sample results detected were less than 5X the PQL. No qualification of the data was made.
Was the mass spectrometer resolution $\geq$ 10,000 demonstrated at the beginning and end of each 12-hour analytical sequence?	Yes
Was the chromatographic peak separation on a DB-5 (or equivalent) column between the 2,3,7,8-TCDD peak and the 1,2,3,8-TCDD peak was resolved with a valley of $\leq$ 25%?	Yes
Was the CS3 standard analyzed at the beginning and end of each 12-hour analysis sequence and were the absolute retention times (RTs) of $^{13}C_{12}$ -1,2,3,4-TCDD within $\pm$ 15 seconds of the absolute RT obtained during initial calibration and exceeded 25.0 minutes on the DB-5 column and 15.0 minutes on the DB-225 column?	Yes
Were all CDDs/CDFs in the CS3 standard within their respective ion abundance ratio limits and the peaks representing both native and labeled analytes in the CS3 standard had Signal-to-Noise (S/N) ratios $\geq$ 10.0?	Yes
Second column confirmation was employed whenever 2,3,7,8-TCDF was detected above the PQL in any sample. Quantitation was performed on both columns. The result from the primary column was used for the sample result.	Yes

Summary of Findings
• 1,2,3,4,6,7,8,9-OCDD was recovered above the acceptance criteria in the MS and MSD for sample VC16-D21-24E. The original sample result was J qualified as estimated.

Table 2A – List of Positive Results for Blank Samples

Method	Sample ID	Sample Type	Analyte	Result	Qualifier	Units	MDL	PQL
1613B	MB for batch 36977	MB	1,2,3,4,6,7,8,9-OCDD	229		pg/L	33.4	100

Table 2B – List of Samples Qualified for Method Blank Contamination

None.

Table 2C – List of Samples Qualified for Field Blank Contamination

None.

Table 3 – List of Samples with Surrogates outside Control Limits

None.

Table 4A - List MS/MSD Recoveries outside Control Limits

Method	Sample ID	Sample Type	Analyte	Orig. Result	Spike Amount	MS	MSD	Dil Fac	Low Limit	High Limit	Sample Qualifier
1613B	VC16-D21-24E MS	MS	1,2,3,4,6,7,8,9-OCDD	113	200	281	324	1	70	130	J Flag

Table 4B – List RPDs outside Control Limits

None.

Table 5A - List LCS Recoveries outside Control Limits

None.

Table 5B – List RPDs outside Control Limits

None.

Table 6 –Samples that were Reanalyzed/Diluted

None.

Table 7 – Summary of Positive Field Duplicate Results

Method	Analyte	Unit	Matrix	PQL	VC16-D18-21E	DUP6-D18-21E	RPD	RPD Rating	Sample Qual
SW1613B	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin	pg/g	SE	10	378	581	42.3%	Good	None
SW1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	pg/g	SE	5	17.8	26.6	39.6%	Good	None
SW1613B	Total Heptachlorodibenzo-p-dioxin	pg/g	SE	5	58.8	91.1	43.1%	Good	None

Method	Analyte	Unit	Matrix	PQL	VC16-D18-21E	DUP6-D18-21E	RPD	RPD Rating	Sample Qual
SW1613B	Total Hexachlorodibenzo-p-dioxin	pg/g	SE	5	27.8	43.7	44.5%	Good	None
SW1613B	Total Tetrachlorodibenzofuran	pg/g	SE	1	ND	4.64	NC	--	None
SW1613B	Total Tetrachlorodibenzo-p-dioxin	pg/g	SE	1	ND	1.23	NC	--	None

Acronym List and Table Key:

COC = chain of custody
DUSR = data usability summary report
FD = field duplicate sample
HRGC = High Resolution Gas Chromatography
HRMS = High Resolution Mass Spectrometry
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
LR = laboratory replicate
MB = method blank
MS = matrix spike
MSD = matrix spike duplicate
N = normal field sample
NA = not applicable
NC = not calculated
ND = not detected
NJDEP = New Jersey Department of Environmental Protection
NYSDEC = New York State Department of Environmental Conservation
PCDDs = polychlorinated dibenzo-p-dioxins
PCDFs = polychlorinated dibenzofurans
PQL = practical quantitation limit
QA = quality assurance
QAPP = quality assurance project plan
QC = quality control
RB = rinsate blank sample
RPD = relative percent difference
SDG = sample delivery group

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: June 15, 2018	Completed by: Eridania Marte

The analytical data provided by the laboratory were reviewed for precision, accuracy, and completeness based on applicable sections of the following guidance:

- EPA Region 2 Data Validation Standard Operating Procedures.
  - *SOP NO. HW-35A, Rev. 0, Semivolatile Data Validation*
  - *SOP NO. HW-36A, Rev. 0, Pesticide Data Validation*
  - *SOP NO. HW-37A, Rev. 0, Polychlorinated Biphenyl (PCB) Aroclor Data Validation*
  - *SOP NO. HW-3B, Rev. 0, ICP-MS Data Validation*
  - *SOP NO. HW-3C, Rev. 0, Mercury and Cyanide Data Validation*

Specific criteria for QC limits were obtained from the site specific QAPP: *Offshore Sampling and Analysis Plan / Quality Assurance Project Plan, Version 1.1, Northeast Supply Enhancement Project*. Compliance with the project QA program is indicated in the checklist and tables below. Any major or minor concerns affecting data usability are listed below. The checklist and tables also indicate whether data qualification is required and/or the type of qualifier assigned.

Reference:

Project ID	Lab Work Order	Laboratory
1000891.0020.05	L1804795	Alpha Analytical

Table 1: Sample List

SDG	Sample Name	Matrix	Lab ID	Sample Date	Sample Type	Original ID
L1804795	DUP6-D18-21E	SE	L1804795-08	2/10/2018 10:15:00 AM	FD	
L1804795	DUP7-D15-18E	SE	L1804795-22	2/11/2018 10:16:00 AM	FD	
L1804795	VC16-D15-18E	SE	L1804795-04	2/10/2018 10:15:00 AM	N	
L1804795	VC16-D18-21E	SE	L1804795-05	2/10/2018 10:15:00 AM	N	
L1804795	VC16-D21-24E	SE	L1804795-06	2/10/2018 10:15:00 AM	N	
L1804795	VC16-D24-27.9E	SE	L1804795-07	2/10/2018 10:15:00 AM	N	
L1804795	VC17-D15-18E	SE	L1804795-01	2/10/2018 9:22:00 AM	N	
L1804795	VC17-D18-21E	SE	L1804795-02	2/10/2018 9:22:00 AM	N	
L1804795	VC17-D21-24.1E	SE	L1804795-03	2/10/2018 9:22:00 AM	N	
L1804795	VC38-D15-18E	SE	L1804795-15	2/11/2018 9:06:00 AM	N	
L1804795	VC38-D18-21E	SE	L1804795-16	2/11/2018 9:06:00 AM	N	
L1804795	VC38-D21-24E	SE	L1804795-17	2/11/2018 9:06:00 AM	N	
L1804795	VC38-D24-26.1E	SE	L1804795-18	2/11/2018 9:06:00 AM	N	
L1804795	VC39-D12-15E	SE	L1804795-09	2/10/2018 12:57:00 PM	N	
L1804795	VC39-D15-18E	SE	L1804795-10	2/10/2018 12:57:00 PM	N	
L1804795	VC39-D18-21.2E	SE	L1804795-11	2/10/2018 12:57:00 PM	N	
L1804795	VC40-D15-18E	SE	L1804795-13	2/10/2018 3:29:00 PM	N	
L1804795	VC40-D18-21.7E	SE	L1804795-14	2/10/2018 3:29:00 PM	N	
L1804795	VC41-ALT-D12-15E	SE	L1804795-19	2/11/2018 10:16:00 AM	N	VC41ALT-D12-15E

SDG	Sample Name	Matrix	Lab ID	Sample Date	Sample Type	Original ID
L1804795	VC41-ALT-D15-18E	SE	L1804795-20	2/11/2018 10:16:00 AM	N	VC41ALT-D15-18E
L1804795	VC41-ALT-D18-19.4E	SE	L1804795-21	2/11/2018 10:16:00 AM	N	VC41ALT-D18-19.4E
L1804795	VC16-D21-24E-MS	SQ	WG1088941-4	2/10/2018 10:15:00 AM	MS	
L1804795	VC16-D21-24E-MS	SQ	WG1089165-3	2/10/2018 10:15:00 AM	MS	
L1804795	VC16-D21-24E-MS	SQ	WG1089169-3	2/10/2018 10:15:00 AM	MS	
L1804795	VC16-D21-24E-MS	SQ	WG1089539-4	2/10/2018 10:15:00 AM	MS	
L1804795	VC16-D21-24E-MS	SQ	WG1089541-4	2/10/2018 10:15:00 AM	MS	
L1804795	VC16-D21-24E-MS	SQ	WG1090526-4	2/10/2018 10:15:00 AM	MS	
L1804795	VC16-D21-24E-MSD	SQ	WG1088941-5	2/10/2018 10:15:00 AM	MSD	
L1804795	VC16-D21-24E-MSD	SQ	WG1089165-4	2/10/2018 10:15:00 AM	MSD	
L1804795	VC16-D21-24E-MSD	SQ	WG1089169-4	2/10/2018 10:15:00 AM	MSD	
L1804795	VC16-D21-24E-MSD	SQ	WG1089539-5	2/10/2018 10:15:00 AM	MSD	
L1804795	VC16-D21-24E-MSD	SQ	WG1089541-5	2/10/2018 10:15:00 AM	MSD	
L1804795	VC16-D21-24E-MSD	SQ	WG1090526-5	2/10/2018 10:15:00 AM	MSD	
L1804795	VC40-D15-18E-MS	SQ	WG1088944-4	2/10/2018 3:29:00 PM	MS	
L1804795	VC40-D15-18E-MS	SQ	WG1088947-4	2/10/2018 3:29:00 PM	MS	
L1804795	VC40-D15-18E-MS	SQ	WG1088948-4	2/10/2018 3:29:00 PM	MS	
L1804795	VC40-D15-18E-MS	SQ	WG1089231-3	2/10/2018 3:29:00 PM	MS	
L1804795	VC40-D15-18E-MS	SQ	WG1089233-3	2/10/2018 3:29:00 PM	MS	
L1804795	VC40-D15-18E-MS	SQ	WG1090532-4	2/10/2018 3:29:00 PM	MS	
L1804795	VC40-D15-18E-MSD	SQ	WG1088944-5	2/10/2018 3:29:00 PM	MSD	
L1804795	VC40-D15-18E-MSD	SQ	WG1088947-5	2/10/2018 3:29:00 PM	MSD	
L1804795	VC40-D15-18E-MSD	SQ	WG1088948-5	2/10/2018 3:29:00 PM	MSD	
L1804795	VC40-D15-18E-MSD	SQ	WG1089231-4	2/10/2018 3:29:00 PM	MSD	
L1804795	VC40-D15-18E-MSD	SQ	WG1089233-4	2/10/2018 3:29:00 PM	MSD	
L1804795	VC40-D15-18E-MSD	SQ	WG1090532-5	2/10/2018 3:29:00 PM	MSD	
L1804795	RB-02102018	WH	L1804795-12	2/10/2018 3:21:00 PM	RB	
L1804795	RB-02112018	WH	L1804795-23	2/11/2018 11:05:00 AM	RB	
L1804795	RB-02102018-MS	WQ	WG1089511-3	2/10/2018 3:21:00 PM	MS	

Table 1A: Sample Test Summary

SDG	Matrix	Test Method	Prep Method	Number of Samples	Sample Type
L1804795	SE	6020A	3050B	19	N
L1804795	SE	6020A	3050B	2	FD
L1804795	WH	6020A	SW3020A	2	RB
L1804795	SE	A2540G		2	FD
L1804795	SE	A2540G		19	N

SDG	Matrix	Test Method	Prep Method	Number of Samples	Sample Type
L1804795	SE	E7474/SW846	METHOD	2	FD
L1804795	SE	E7474/SW846	METHOD	19	N
L1804795	WH	E7474/SW846	METHOD	2	RB
L1804795	SE	LLOYDKAHN		2	FD
L1804795	SE	LLOYDKAHN		19	N
L1804795	SE	SW8081B	SW3570	19	N
L1804795	SE	SW8081B	SW3570	2	FD
L1804795	WH	SW8081B	SW3510C	2	RB
L1804795	SE	SW8082A	SW3570	19	N
L1804795	SE	SW8082A	SW3570	2	FD
L1804795	WH	SW8082A	SW3510C	2	RB
L1804795	SE	SW8270DSIM	SW3570	2	FD
L1804795	SE	SW8270DSIM	SW3570	19	N
L1804795	WH	SW8270DSIM	SW3510C	2	RB

Table 1B: Holding Time Exceedances
None.

General Sample Information	
Do Samples and Analyses on COC check against Lab Sample Tracking Form?	A sample name was changed as noted in Table to be consistent with nomenclature from previous sampling event.
Did coolers arrive at lab between 2 and 6°C and in good condition as indicated on COC and Cooler Receipt Form?	Yes.
Frequency of Field QC Samples Correct? Field Duplicate - 1/20 samples MS/MSD – 1/20 samples Trip Blank - Every cooler with VOCs waters only Equipment Blank - 1/ set of samples per day?	Two field duplicates and two MS/MSDs were required to be collected over the course of the field sampling event. They were included in this SDG. 2 field duplicates 2 MS/MSDs VOCs not collected; trip blank not required. 2 equipment blanks (1 per day).
Case narrative present and complete?	Yes.
Any holding time violations?	No.

The following tables are presented at the end of this DUSR and provided summaries of results outside QC criteria:

- Method Blanks Results (Table 2)
- Surrogates Outside Limits (Table 3)
- MS/MSD/LR Outside Limits (Table 4)
- LCS/SRM Outside Limits (Table 5)
- Re-analysis Results (Table 6)
- Field Duplicate Results (Table 7)

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Semi-volatile Organics by GC/MS	
Description	**Notes and Qualifiers**
Any compounds present in method, trip, and rinsate blanks (see Table 2A)?	Yes.
For samples, if results are <5 times the blank or <10 times blank for common laboratory contaminants then "U" flag data.	Dibenz(a,h)anthracene was detected in method blank WG1088941-1. One sample result was less than 5X the blank detection. The result was U qualified as non-detect and the MDL was elevated to sample result. Naphthalene was detected in method blank WG1088941-1. Eight sample results were less than 5X the blank detection. The results were U qualified as non-detect and the MDL was elevated to the sample result. 2-Chloronaphthalene and 2-methylnaphthalene were detected in method blank WG1088944-1. The associated sample results were either ND or greater than 5X the blank detection. No qualification was made. Naphthalene was detected in method blank WG1088944-1. Six sample results were less than 5X the blank detection. The results were U qualified as non-detect and the MDL was elevated to the sample result. Dibenz(a,h)anthracene was detected in method blank WG1089187-1. Two sample results were less than 5X the blank detection. The results were U qualified as non-detect and the MDL was elevated to the sample result. Indeno(1,2,3-cd)pyrene was detected in method blank WG1089187-1. Two sample results were less than 5X the blank detection. The results were U qualified as non-detect and the MDL was elevated to the sample result. 2-Methylnaphthalene, acenaphthene, acenaphthylene, anthracene, benz(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, dibenz(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, and pyrene were detected in rinse blanks RB-02102018 and RB-02112018. The associated samples that were less than 5X the blank detection were J qualified as estimated.
Surrogate for method blanks and LCS within limits?	Yes.

Semi-volatile Organics by GC/MS	
Description	**Notes and Qualifiers**
Surrogate for samples and MS/MSD within limits? (See Table 3). Samples should re-analyzed if 1 base/neutral or 1 acid extractable surrogate has a recovery <10%. Matrix effects should be established.	Yes.
Laboratory QC frequency one blank and LCS with each batch and one set of MS/MSD per 20 samples?	Yes.
MS/MSD within QC criteria (see Table 4A and 4B)? If out and LCS is compliant, then J flag positive data in original sample due to matrix?	Yes.
LCS within QC criteria (see Table 5A and 5B)? If out, and the recovery high with no positive values, then no data qualification is required.	Yes.
Do internal standards areas and retention time meet criteria? If not was sample re-analyzed to establish matrix effects (see Table 6)?	Yes.
Is initial calibration for target compounds <15 %RSD or curve fit? Initial calibration verification <20%D?	Yes.
Is continuing calibration for target compounds < 20%D.	Yes.
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and dilutions is only one reportable result by flagged?	No.
Do field duplicate results show good precision for all compounds (see Table 7)?	Yes.

Pesticides by GC/ECD	
Description	**Notes and Qualifiers**
Are any compounds present in method and field blanks as noted on Table 2?	No.
For samples, if results are <5 times the blank then "U" flag data.	No qualification required.
Are surrogates for method blanks and LCS within limits?	Yes.
Are surrogates for samples and MS/MSD within limits? (See Table 3). Matrix effects should be established.	Yes.
Is laboratory QC frequency one blank and LCS with each batch and one MS/MSD per 20 samples?	Yes.
Are MS/MSD within QC criteria (see Table 4A and 4B)? If out and LCS is compliant, then J flag positive data in original sample due to matrix.	No. Endrin aldehyde was recovered below the acceptance limit in the MS for sample VC16-D21-24E. The RPD was also outside of the acceptance limit. The sample result was UJ qualified as estimated non-detect. Methoxychlor was recovered below the acceptance limit in the MS for sample VC16-D21-24E. The sample result was UJ qualified as estimated non-detect.

Pesticides by GC/ECD	
Description	**Notes and Qualifiers**
Is LCS within QC criteria (see Table 5A and 5B)? If out, and the recovery high with no positive values, then no data qualification is required.	Yes.
Is initial calibration for target compounds <20 %RSD or curve fit? Is initial calibration verification <25%D?	Yes.
Is continuing calibration for target compounds <20%D?	Yes. Alpha-BHC exhibited %D outside of the acceptance criteria in CCV WG1089751-1 on column 2. Alpha-BHC, gamma-BHC, delta-BHC, heptachlor, aldrin, and dieldrin exhibited a %D outside of acceptance criteria in CCV WG1090890-1 on column 2. Only heptachlor epoxide was reported from column 2 for the associated samples, RB-02102018 and RB-02112018; therefore, no qualification was made.
Do internal standards areas meet criteria? If not was sample re-analyzed to establish matrix effects (see Table 6)?	The internal standards were not summarized in the report (the laboratory does not provide a summary form). There were no issues noted in the case narrative.
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and dilutions is only one reportable result by flagged?	No.
Spot check retention time windows and second column confirmations as complete.	Yes. The concentrations of dieldrin, 4,4'-DDT, 2,4-DDE, and 2,4-DDD exceeded the RPD limit of 25% in sample VC16-D15-18E. The analytes were J qualified as estimated in the sample. The concentration of endrin aldehyde was not calculated in sample VC16-D15-18E due to not being detected in column 1 but detected in column 2. The sample result was J qualified as estimated.
Spot check performance evaluation mixture? %Breakdown <20%	Yes.
Do field duplicate results show good precision for all compounds (see Table 7)?	Yes.

PCB Aroclors by GC/ECD	
Description	**Notes and Qualifiers**
Are any compounds present in method and field blanks as noted on Table 2?	No.
For samples, if results are <5 times the blank then "U" flag data.	No qualification required.

PCB Aroclors by GC/ECD	
Description	**Notes and Qualifiers**
Are surrogates for method blanks and LCS within limits?	The surrogate 2,4,5,6-tetrachloro-m-xylene (TMX) exhibited a %D outside of control limits on column 1 and 2 in the CCV WG1093286-1. The surrogates TMX and Decachlorobiphenyl (DCB) exhibited a %D outside of control limits on columns 1 and 2 in CCV WG1093286-2. The surrogates TMX and DCB exhibited a %D outside of control limits on columns 1 and 2 in CCV WG1093286-3 and WG1093286-4. No qualification of the data was made.
Are surrogates for samples and MS/MSD within limits? (See Table 3). Matrix effects should be established.	Yes.
Is laboratory QC frequency one blank and LCS with each batch and one MS/MSD per 20 samples?	Yes.
Are MS/MSD within QC criteria (see Table 4A and 4B)? If out and LCS is compliant, then J flag positive data in original sample due to matrix.	Yes.
Is LCS within QC criteria (see Table 5A and 5B)? If out, and the recovery high with no positive values, then no data qualification is required.	Yes.
Is initial calibration for target compounds <20 %RSD or curve fit? Is initial calibration verification <20%D?	Yes.
Is continuing calibration for target compounds <20%D?	All sample results were reported from column 1. The opening CCV WG1093286-2 exhibited an average of 20.4% D for Aroclor 1016 and an average of 22.9% D for Aroclor 1260 on column 1. The closing CCV WG1093286-3 exhibited an average of 26.4% D for Aroclor 1016 and an average of 26.5% D for Aroclor 1260 on column 1. The associated samples, VC17-D15-18E, VC17-D18-21E, VC17-D21-24.1E, VC16-D15-18E, VC16-D18-21E, VC16-D24-27.9E, DUP6-D18-21E, VC39-D12-15E, VC39-D15-18E, and VC39-D18-21.2E, were UJ qualified as estimated non-detect. The opening CCV WG1093286-3 exhibited an average of 26.4% D for Aroclor 1016 and an average of 26.5% D for Aroclor 1260 on column 1. The closing CCV WG1093286-4 exhibited an average of 29.2% D for Aroclor 1016 and an average of 37.3% D for Aroclor 1260 on column 1. The associated samples, VC40-D15-18E, VC40-D18-21.7E, VC38-D15-18E, VC38-D18-21E, VC38-D21-24E, VC38-D24-26.1E, VC41ALT-D12-15E, VC41ALT-D15-18E, VC41ALT-D18-19.4E, and DUP7-D15-18E, were UJ qualified as estimated non-detect.

PCB Aroclors by GC/ECD	
Description	**Notes and Qualifiers**
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and dilutions is only one reportable result by flagged?	No.
Spot check retention time windows and second column confirmations as complete.	Yes.
Do field duplicate results show good precision for all compounds (see Table 7)?	Yes.

Metals & Mercury by ICP/MS and CVAA	
Description	**Notes and Qualifiers**
Are any compounds present in method and field blanks as noted on Table 2?	Yes.
For samples, if results are <5 times the blank then "U" flag data.	Antimony was detected in method blank WG1089165-1. Ten sample results were less than 5X the blank detection. The results were U qualified as non-detect due to ICB detections and the MDL was elevated to sample result. Tin was detected in method blank WG1089165-1. Fifteen sample results were less than 5X the blank detection. Thirteen sample results were U qualified as non-detect due to ICB detections and the MDL was elevated to sample result. Two sample results were U qualified as non-detect due to ICB detections and the PQL was elevated to the sample result. Tin was detected in method blank WG1089231-1. The associated samples were either ND or greater than 5X the blank detection. No qualification was made. Antimony was detected in method blank WG1089511-1. One sample results were less than 5X the blank detection. The results were U qualified as non-detect and the MDL was elevated to sample result. Tin was detected in method blank WG1089511-1. Two sample results were less than 5X the blank detection. The results were U qualified as non-detect and the MDL was elevated to sample result. Iron, manganese, and titanium were detected in method blank WG1089511-1. The associated samples were either ND or greater than 5X the blank detection. No qualification was made. Mercury was detected in method blank WG1088775-1. The associated samples were either ND or greater than 5X the blank detection. No qualification was made.

	Aluminum, antimony, arsenic, barium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, nickel, potassium, sodium, strontium, tin, titanium, and zinc were detected in rinse blank RB-02102018. The associated samples that were less than 5X the blank detection were J qualified as estimated. Aluminum, barium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, nickel, potassium, sodium, strontium, tin, titanium, and zinc were detected in rinse blank RB-02112018. The associated samples that were less than 5X the blank detection were J qualified as estimated.
Is laboratory QC frequency one blank and LCS with each batch and one set of MS/MSD per 20 samples?	Yes.
Are MS/MSD within QC criteria (see Table 3)? If not, was a post digestion spike analyzed? QC limits are not applicable to sample results greater than 4 times spike amount.	No. For sample VC16-D21-24E, aluminum, calcium, iron, magnesium, and manganese were recovered above control limits. The sample results were greater than 4X the spike amount; therefore, no qualification was required. Antimony, potassium, silver, sodium, and titanium were recovered outside the control limit. The associated sample results were J qualified as estimated. For sample VC40-D15-18E, iron and aluminum were recovered above control limits. The sample results were greater than 4X the spike amount; therefore, no qualification was required. Calcium and silver were recovered above the control limit. The samples result for calcium was J qualified as estimated and UJ qualified as estimated non-detect for silver. The post digestion associated with sample VC16-D21-24E was outside of control limits for aluminum, magnesium, manganese, and titanium. No qualification was made.
Were elements recovered ≤30%? If so, “R” flag associated NDs.	No.
Is LCS within QC criteria (see Table 4)? If out, and the recovery is high with no positive values, then no data qualification is required.	Yes.
Is there one serial dilution per 20 samples? Flag all data reported with an “E” as “J”.	Yes. The serial dilution associated for sample VC16-D21-24E was outside of the laboratory’s control limits for aluminum, manganese, and titanium. The analytes in the sample were J qualified as estimated.
Spot check ICS recoveries 80-120%. Contact lab if unacceptable.	Yes.
Spot check ICV 90-110%. Contact lab if unacceptable.	Yes.
Spot check CCV 90-110%. Contact lab if unacceptable.	Yes.
Spot check ICB/CCB +/- RL. Contact lab if unacceptable.	Yes.

	Antimony and tin were detected in ICB R1048066-2. The associated samples that were less than 5X the blank detection were U qualified as non-detect and the MDL was elevated to sample result. Antimony, strontium, titanium, and tin were detected in ICB R1048506-2. The associated results for antimony and tin in rinse sample RB-02102018 was U qualified as non-detect and the MDL was elevated to sample result. The associated result for tin in rinse sample RB-02112018 was U qualified as non-detect and the MDL was elevated to the sample result. Antimony and tin were detected in ICB R1049194-2. The associated samples were less than 5X the blank detection were U qualified as non-detect and the MDL was elevated to sample result. Iron was detected in CCBs R1048506-25 and R1048506-27. There were no associated sample results less than 5X the blank detection. Molybdenum was detected in CCB R1049194-17. The associated samples noted in Table 2A were less than 5X the blank detection were U qualified as non-detect and the MDL was elevated to sample result.
Are laboratory replicates within limits?	No. Laboratory replicates were outside of acceptance limit for cadmium, copper, potassium, tin and zinc for rinse sample RB-02102018. No qualifications were made.
Were any samples re-analyzed or diluted (see Table 6)? For any sample re-analysis and dilutions is only one reportable result by flagged?	Yes. All samples were diluted for metals at a 10X dilution and mercury at a 5X dilution. There is no impact to data usability.
Do field duplicate results show good precision for all compounds (see Table 6)?	Yes.

Total Organic Carbon - Lloyd Khan	
Description	**Notes and Qualifiers**
Are any compounds present in method and field blanks as noted on Table 2?	No.
For samples, if results are <5 times the blank then "U" flag data.	N/A
Is laboratory QC frequency one blank and LCS with each batch and one set of MS/MSD per 20 samples?	Yes.
Are MS/MSD within QC criteria (see Table 4)? QC limits are not applicable to sample results greater than 4 times spike amount.	Yes.
Is LCS and SRM within QC criteria (see Table 5)? If out, and the recovery high with no positive values, then no data qualification is required.	Yes.

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Total Organic Carbon - Lloyd Khan	
Description	**Notes and Qualifiers**
Is initial calibration r ≥ 0.995 and slope ± 10% the historical curves? Is initial calibration verification 80-120% of True Value?	Yes.
Is continuing calibration for 80-120% of True Value?	Yes.
Spot check CCB < reporting limit. Contact lab if unacceptable.	Yes.
Are laboratory duplicates within QC limits?	Yes.
Do field duplicate results show good precision for all compounds (see Table 7)?	Yes.

Summary of Findings

- 8270D: Dibenz(a,h)anthracene, naphthalene, indeno(1,2,3-cd)pyrene were detected in laboratory's method blank. The associated sample results that were less than 5X the blank detection were U qualified as non-detect and the MDL was elevated to sample result.

- 8270D: 2-Methylnaphthalene, acenaphthene, acenaphthylene, anthracene, benz(a)anthracene, benzo(b)fluoranthene, benzo(ghi)perylene, benzo(k)fluoranthene, chrysene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, and pyrene were detected in rinse blank RB-02102018 and RB-02112018. The associated samples that were less than 5X the blank detection were J qualified as estimated.

- 8081B: Retention time for dieldrin and 4,4'-DDT, 2,4-DDE, 2,4-DDD exceeded RPD limit for sample VC16-D15-18E. The retention time was not calculated for endrin aldehyde as one concentration showed to be non-detect. The concentration of endrin aldehyde was not calculated due to not being detected in column 1 but detected in column 2. The sample results were J qualified as estimated.

- 8081B: Endrin aldehyde and methoxyclor were recovered below the acceptance limit in the MS for sample VC16-D21-24E. The sample results were UJ qualified as estimated non-detect.

- 8082A: The opening and closing CCVs for Aroclor 1016 and Aroclor 1260 in channel A were outside of the acceptance criteria. The associated samples were UJ qualified as estimated non-detect.

- 6020A: Antimony and tin were detected in multiple laboratory's method blanks. The associated samples results in which were less than 5X the blank detection were U qualified as non-detect and the MDL was elevated to sample result. Two sample results for tin were U qualified as non-detect and the PQL was elevated to the sample result.

- 6020A: Barium, cadmium, calcium, copper, magnesium, nickel, potassium, sodium, strontium, and zinc were detected in rinse blank RB-02102018. The associated samples that were less than 5X the blank detection were J qualified as estimated.

- 6020A: Barium, calcium, chromium, magnesium, manganese, nickel, sodium, strontium, and zinc were detected in rinse blank RB-02112018. The associated samples that were less than 5X the blank detection were J qualified as estimated.

- 6020A: The serial dilution associated for sample VC16-D21-24E was outside of the laboratory's control limit for aluminum, manganese, and titanium. The associated sample results were J qualified as estimated.

- 6020A: Antimony and tin were detected in multiple laboratory's ICBs. The associated samples that were less than 5X the blank detection were U qualified as non-detect and the MDL was elevated to sample result.

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Summary of Findings

- 6020A: Stronium and titanium were detected in ICB R1048506-2. The associated samples were U qualified as non-detect and the MDL was elevated to sample result. One sample result was U qualified as non-detect and the PQL was elevated to the sample result.
- 6020A: Molybdenum was detected in CCB R1049194-17. The associated samples that were less than 5X the blank detection were U qualified as non-detect and the MDL was elevated to sample result.
- 6020A: For sample VC16-D21-24E antimony, potassium, silver, sodium, and titanium were recovered outside the control limit. The associated sample results were J qualified as estimated.
- 6020A: For sample VC40-D15-18E calcium and silver were recovered above the control limit. The samples result for calcium was J qualified as estimated and UJ qualified as estimated non-detect for silver.

Table 2 – List of Positive Results for Blank Samples

Method	Sample Name	Matrix	Analyte	Result	Qualifier	Units	MDL	QL
6020A	WG1089165-1	LB	ANTIMONY, TOTAL	0.321	J	mg/kg	0.135	1.6
6020A	WG1089165-1	LB	TIN, TOTAL	0.838	J	mg/kg	0.124	1.6
6020A	WG1089231-1	LB	TIN, TOTAL	0.132	J	mg/kg	0.124	1.2
6020A	WG1089511-1	LB	ANTIMONY, TOTAL	0.00081	J	mg/l	0.00042	0.004
6020A	WG1089511-1	LB	IRON, TOTAL	0.0323	J	mg/l	0.0191	0.05
6020A	WG1089511-1	LB	MANGANESE, TOTAL	0.00097	J	mg/l	0.00044	0.001
6020A	WG1089511-1	LB	TIN, TOTAL	0.00286	J	mg/l	0.00112	0.004
6020A	WG1089511-1	LB	TITANIUM, TOTAL	0.00023	J	mg/l	0.00007	0.0005
6020A	R1048066-2	ICB	ANTIMONY	0.909	J	ug/l		
6020A	R1048066-2	ICB	TIN	1.10	J	ug/l		
6020A	R1048506-2	ICB	ANTIMONY	1.99	J	ug/l		
6020A	R1048506-2	ICB	STRONIUM	0.0519	J	ug/l		
6020A	R1048506-2	ICB	TITANIUM	0.311	J	ug/l		
6020A	R1048506-2	ICB	TIN	3.46		ug/l		
6020A	R1048506-25	CCB	IRON	19.6	J	ug/l		
6020A	R1048506-27	CCB	IRON	20.8	J	ug/l		
6020A	R1049194-2	ICB	ANTIMONY	1.13	J	ug/l		
6020A	R1049194-2	ICB	TIN	2.54	J	ug/l		
6020A	R1049194-17	CCB	MOLYBDENUM	0.381	J	ug/l		
E7474/SW846	WG1088775-1	LB	MERCURY, TOTAL	0.00001	J	mg/l	0.00001	0.00005
SW8270DSIM	WG1088941-1	LB	DIBENZ(A,H)ANTHRACENE	0.446	J	ug/kg	0.412	4
SW8270DSIM	WG1088941-1	LB	NAPHTHALENE	0.389	J	ug/kg	0.358	4
SW8270DSIM	WG1088944-1	LB	2-CHLORONAPHTHALENE	0.352	J	ug/kg	0.313	4
SW8270DSIM	WG1088944-1	LB	2-METHYLNAPHTHALENE	0.548	J	ug/kg	0.496	4
SW8270DSIM	WG1088944-1	LB	NAPHTHALENE	1.09	J	ug/kg	0.358	4
SW8270DSIM	WG1089187-1	LB	DIBENZ(A,H)ANTHRACENE	0.809	J	ng/l	0.685	10
SW8270DSIM	WG1089187-1	LB	INDENO(1,2,3-CD)PYRENE	0.694	J	ng/l	0.533	10
6020A	RB-02102018	RB	ALUMINUM, TOTAL	0.138		mg/l	0.00327	0.01

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Method	Sample Name	Matrix	Analyte	Result	Qualifier	Units	MDL	QL
6020A	RB-02102018	RB	ANTIMONY, TOTAL	0.00061	J	mg/l	0.00042	0.004
6020A	RB-02102018	RB	ARSENIC, TOTAL	0.0003	J	mg/l	0.00016	0.0005
6020A	RB-02102018	RB	BARIUM, TOTAL	0.01056		mg/l	0.00017	0.0005
6020A	RB-02102018	RB	CADMIUM, TOTAL	0.00093		mg/l	0.00005	0.0002
6020A	RB-02102018	RB	CALCIUM, TOTAL	2.05		mg/l	0.0394	0.1
6020A	RB-02102018	RB	CHROMIUM, TOTAL	0.00391		mg/l	0.00017	0.0005
6020A	RB-02102018	RB	COBALT, TOTAL	0.00038	J	mg/l	0.00016	0.0005
6020A	RB-02102018	RB	COPPER, TOTAL	0.0353		mg/l	0.00038	0.001
6020A	RB-02102018	RB	IRON, TOTAL	1.55		mg/l	0.0191	0.05
6020A	RB-02102018	RB	LEAD, TOTAL	0.00171		mg/l	0.00034	0.001
6020A	RB-02102018	RB	MAGNESIUM, TOTAL	1.36		mg/l	0.0242	0.07
6020A	RB-02102018	RB	MANGANESE, TOTAL	0.04676		mg/l	0.00044	0.001
6020A	RB-02102018	RB	NICKEL, TOTAL	0.00557		mg/l	0.00055	0.002
6020A	RB-02102018	RB	POTASSIUM, TOTAL	2.27		mg/l	0.0309	0.1
6020A	RB-02102018	RB	SODIUM, TOTAL	10.8		mg/l	0.0293	0.1
6020A	RB-02102018	RB	STRONTIUM, TOTAL	0.01288		mg/l	0.00003	0.0005
6020A	RB-02102018	RB	TIN, TOTAL	0.00477		mg/l	0.00112	0.004
6020A	RB-02102018	RB	TITANIUM, TOTAL	0.00768		mg/l	0.00007	0.0005
6020A	RB-02102018	RB	ZINC, TOTAL	0.0802		mg/l	0.00341	0.01
6020A	RB-02112018	RB	ALUMINUM, TOTAL	0.029		mg/l	0.00327	0.01
6020A	RB-02112018	RB	BARIUM, TOTAL	0.00243		mg/l	0.00017	0.0005
6020A	RB-02112018	RB	CADMIUM, TOTAL	0.00036		mg/l	0.00005	0.0002
6020A	RB-02112018	RB	CALCIUM, TOTAL	0.493		mg/l	0.0394	0.1
6020A	RB-02112018	RB	CHROMIUM, TOTAL	0.00231		mg/l	0.00017	0.0005
6020A	RB-02112018	RB	COBALT, TOTAL	0.00023	J	mg/l	0.00016	0.0005
6020A	RB-02112018	RB	COPPER, TOTAL	0.00248		mg/l	0.00038	0.001
6020A	RB-02112018	RB	IRON, TOTAL	1.56		mg/l	0.0191	0.05
6020A	RB-02112018	RB	LEAD, TOTAL	0.00045	J	mg/l	0.00034	0.001
6020A	RB-02112018	RB	MAGNESIUM, TOTAL	0.389		mg/l	0.0242	0.07

Method	Sample Name	Matrix	Analyte	Result	Qualifier	Units	MDL	QL
6020A	RB-02112018	RB	MANGANESE, TOTAL	0.02384		mg/l	0.00044	0.001
6020A	RB-02112018	RB	NICKEL, TOTAL	0.00277		mg/l	0.00055	0.002
6020A	RB-02112018	RB	POTASSIUM, TOTAL	0.104		mg/l	0.0309	0.1
6020A	RB-02112018	RB	SODIUM, TOTAL	3.54		mg/l	0.0293	0.1
6020A	RB-02112018	RB	STRONTIUM, TOTAL	0.0036		mg/l	0.00003	0.0005
6020A	RB-02112018	RB	TIN, TOTAL	0.00282	J	mg/l	0.00112	0.004
6020A	RB-02112018	RB	TITANIUM, TOTAL	0.00171		mg/l	0.00007	0.0005
6020A	RB-02112018	RB	ZINC, TOTAL	0.0148		mg/l	0.00341	0.01
SW8270DSIM	RB-02102018	RB	2-METHYLNAPHTHALENE	17.56		ng/mL	1.07	10.1
SW8270DSIM	RB-02102018	RB	ACENAPHTHENE	4.19	J	ng/l	1.63	10.1
SW8270DSIM	RB-02102018	RB	ACENAPHTHYLENE	2.17	J	ng/l	1.79	10.1
SW8270DSIM	RB-02102018	RB	ANTHRACENE	8.79	J	ng/l	1.96	10.1
SW8270DSIM	RB-02102018	RB	BENZ(A)ANTHRACENE	11.2		ng/l	1.75	10.1
SW8270DSIM	RB-02102018	RB	BENZO(A)PYRENE	6.52	J	ng/l	0.848	10.1
SW8270DSIM	RB-02102018	RB	BENZO(B)FLUORANTHENE	10.7		ng/l	1.49	10.1
SW8270DSIM	RB-02102018	RB	BENZO(GHI)PERYLENE	6.1	J	ng/l	1.32	10.1
SW8270DSIM	RB-02102018	RB	BENZO(K)FLUORANTHENE	8.58	J	ng/l	1.18	10.1
SW8270DSIM	RB-02102018	RB	CHRYSENE	14.6		ng/l	0.945	10.1
SW8270DSIM	RB-02102018	RB	DIBENZ(A,H)ANTHRACENE	2.33	J	ng/l	0.692	10.1
SW8270DSIM	RB-02102018	RB	FLUORANTHENE	42.5		ng/l	1.5	10.1
SW8270DSIM	RB-02102018	RB	FLUORENE	7.31	J	ng/l	1.75	10.1
SW8270DSIM	RB-02102018	RB	INDENO(1,2,3-CD)PYRENE	5.95	J	ng/l	0.538	10.1
SW8270DSIM	RB-02102018	RB	NAPHTHALENE	32.8		ng/l	1.79	10.1
SW8270DSIM	RB-02102018	RB	PHENANTHRENE	54.7		ng/l	1.91	10.1
SW8270DSIM	RB-02102018	RB	PYRENE	26.2		ng/l	1.54	10.1
SW8270DSIM	RB-02112018	RB	2-METHYLNAPHTHALENE	19.1		ng/l	1.07	10.1
SW8270DSIM	RB-02112018	RB	ACENAPHTHENE	3.09	J	ng/l	1.63	10.1
SW8270DSIM	RB-02112018	RB	ACENAPHTHYLENE	2.88	J	ng/l	1.79	10.1
SW8270DSIM	RB-02112018	RB	ANTHRACENE	3.36	J	ng/l	1.96	10.1

Method	Sample Name	Matrix	Analyte	Result	Qualifier	Units	MDL	QL
SW8270DSIM	RB-02112018	RB	BENZ(A)ANTHRACENE	3.14	J	ng/l	1.75	10.1
SW8270DSIM	RB-02112018	RB	BENZO(A)PYRENE	1.95	J	ng/l	0.848	10.1
SW8270DSIM	RB-02112018	RB	BENZO(B)FLUORANTHENE	3.15	J	ng/l	1.49	10.1
SW8270DSIM	RB-02112018	RB	BENZO(GHI)PERYLENE	2.19	J	ng/l	1.32	10.1
SW8270DSIM	RB-02112018	RB	BENZO(K)FLUORANTHENE	2.23	J	ng/l	1.18	10.1
SW8270DSIM	RB-02112018	RB	CHRYSENE	5.07	J	ng/l	0.945	10.1
SW8270DSIM	RB-02112018	RB	DIBENZ(A,H)ANTHRACENE	0.872	J	ng/l	0.692	10.1
SW8270DSIM	RB-02112018	RB	FLUORANTHENE	13.4		ng/l	1.5	10.1
SW8270DSIM	RB-02112018	RB	FLUORENE	3.12	J	ng/l	1.75	10.1
SW8270DSIM	RB-02112018	RB	INDENO(1,2,3-CD)PYRENE	1.94	J	ng/l	0.538	10.1
SW8270DSIM	RB-02112018	RB	NAPHTHALENE	34.2		ng/l	1.79	10.1
SW8270DSIM	RB-02112018	RB	PHENANTHRENE	21.1		ng/l	1.91	10.1
SW8270DSIM	RB-02112018	RB	PYRENE	9	J	ng/l	1.54	10.1

Table 2A – List of Samples Qualified for Blank Contamination

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qual.	Units	MDL	QL	Affected Sample	Sample Flag
6020A	R1048506-2	ICB	ANTIMONY	1.99	0.612	J	Ug/l	0.00042	0.004	RB-02102018	U Flag
6020A	R1049194-2	ICB	ANTIMONY	1.13	0.108	J	ug/l			VC38-D21-24E	U Flag
6020A	R1048066-2	ICB	ANTIMONY	0.909	0.436		ug/l	0.155	1.83	VC40-D15-18E	U Flag
6020A	R1048506-2	ICB	TIN	3.46	4.773		Ug/l	1.12	4	RB-02102018	U Flag
6020A	R1048506-2	ICB	TIN	3.46	2.82		Ug/l	1.12	4	RB-02112018	U Flag
6020A	R1049194-2	ICB	TIN	2.54	0.313	J	ug/l	0.134	2.17	VC38-D21-24E	U Flag
6020A	R1049194-2	ICB	TIN	2.54	0.095	J	ug/l	0.147	2.37	VC38-D24-26.1E	U Flag
6020A	R1049194-2	ICB	TIN	2.54	0.133	J	ug/l	0.156	2.52	VC41ALT-D12-15E	U Flag
6020A	R1049194-2	ICB	TIN	2.54	0.016	J	ug/l	0.156	2.51	VC41ALT-D15-18E	U Flag
6020A	R1049194-2	ICB	TIN	2.54	0.032	J	ug/l	0.149	2.41	VC41ALT-D18-19.4E	U Flag
6020A	R1049194-2	ICB	TIN	2.54	0.009	J	ug/l	0.157	2.53	DUP7-D15-18E	U Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qual.	Units	MDL	QL	Affected Sample	Sample Flag
6020A	R1049194-17	CCB	MOLYBDENUM	0.381	0.033	J	ug/l	0.117	0.868	VC38-D21-24E	U Flag
6020A	R1049194-17	CCB	MOLYBDENUM	0.381	0.02	J	ug/l	0.136	1.01	VC41ALT-D12-15E	U Flag
6020A	R1049194-17	CCB	MOLYBDENUM	0.381	0.009	J	ug/l	0.136	1	VC41ALT-D15-18E	U Flag
6020A	R1049194-17	CCB	MOLYBDENUM	0.381	-0.001	J	ug/l	0.13	0.964	VC41ALT-D18-19.4E	U Flag
6020A	R1049194-17	CCB	MOLYBDENUM	0.381	0.009	J	ug/l	0.137	1.01	DUP7-D15-18E	U Flag
SW8270DSIM	WG1088941-1	SE	DIBENZ(A,H)ANTHRACENE	0.446	2.14	J	ug/kg	0.695	6.75	VC16-D24-27.9E	U Flag
SW8270DSIM	WG1089187-1	WH	DIBENZ(A,H)ANTHRACENE	0.809	2.33	J	ng/l	0.692	10.1	RB-02102018	U Flag
SW8270DSIM	WG1089187-1	WH	DIBENZ(A,H)ANTHRACENE	0.446	0.872	J	ng/l	0.692	10.1	RB-02112018	U Flag
SW8270DSIM	WG1088941-1	SE	NAPHTHALENE	0.389	0.896	J	ug/kg	0.612	6.84	VC16-D21-24E	U Flag
SW8270DSIM	WG1088941-1	SE	NAPHTHALENE	0.389	0.91	J	ug/kg	0.604	6.75	VC16-D24-27.9E	U Flag
SW8270DSIM	WG1088941-1	SE	NAPHTHALENE	0.389	0.89	J	ug/kg	0.621	6.94	VC17-D15-18E	U Flag
SW8270DSIM	WG1088941-1	SE	NAPHTHALENE	0.389	0.806	J	ug/kg	0.655	7.32	VC17-D18-21E	U Flag
SW8270DSIM	WG1088941-1	SE	NAPHTHALENE	0.389	1.38	J	ug/kg	0.615	6.87	VC17-D21-24.1E	U Flag
SW8270DSIM	WG1088941-1	SE	NAPHTHALENE	0.389	0.471	J	ug/kg	0.383	4.28	VC39-D12-15E	U Flag
SW8270DSIM	WG1088941-1	SE	NAPHTHALENE	0.389	0.418	J	ug/kg	0.386	4.31	VC39-D15-18E	U Flag
SW8270DSIM	WG1088941-1	SE	NAPHTHALENE	0.389	0.504	J	ug/kg	0.377	4.21	VC38-D21-24E	U Flag
SW8270DSIM	WG1088944-1	SE	NAPHTHALENE	1.09	0.468	J	ug/kg	0.423	4.73	VC38-D15-18E	U Flag
SW8270DSIM	WG1088944-1	SE	NAPHTHALENE	1.09	0.715	J	ug/kg	0.41	4.59	VC38-D18-21E	U Flag
SW8270DSIM	WG1088944-1	SE	NAPHTHALENE	1.09	0.692	J	ug/kg	0.422	4.71	VC38-D24-26.1E	U Flag
SW8270DSIM	WG1088944-1	SE	NAPHTHALENE	1.09	0.682	J	ug/kg	0.383	4.28	VC40-D15-18E	U Flag
SW8270DSIM	WG1088944-1	SE	NAPHTHALENE	1.09	0.512	J	ug/kg	0.426	4.76	VC41ALT-D12-15E	U Flag
SW8270DSIM	WG1088944-1	SE	NAPHTHALENE	1.09	0.607	J	ug/kg	0.443	4.95	VC41ALT-D18-19.4E	U Flag
SW8270DSIM	WG1089187-1	WH	INDENO(1,2,3-CD)PYRENE	0.694	1.94	J	ng/l	0.538	10.1	RB-02112018	U Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	49.98		ug/L	0.381	5.41	DUP6-D18-21E	J Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	46.30		ug/L	0.37	5.25	VC16-D18-21E	J Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	40.07		ug/L	0.378	5.36	VC16-D21-24E	J Flag

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Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qual.	Units	MDL	QL	Affected Sample	Sample Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	45.85		ug/L	0.358	5.09	VC16-D24-27.9E	J Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	41.76		ug/L	0.379	5.38	VC17-D15-18E	J Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	40.39		ug/L	0.385	5.46	VC17-D18-21E	J Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	43.74		ug/L	0.377	5.35	VC17-D21-24.1E	J Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	12.07		ug/L	0.236	3.35	VC39-D12-15E	J Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	12.94		ug/L	0.242	3.44	VC39-D15-18E	J Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	18.01		ug/L	0.241	3.42	VC39-D18-21.2E	J Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	8.43		ug/L	0.242	3.44	VC40-D15-18E	J Flag
6020A	RB-02102018	SE	BARIUM, TOTAL	10.56	7.56		ug/L	0.241	3.42	VC40-D18-21.7E	J Flag
6020A	RB-02102018	SE	CADMIUM, TOTAL	0.93	0.387	J	ug/L	0.048	0.361	DUP6-D18-21E	J Flag
6020A	RB-02102018	SE	CADMIUM, TOTAL	0.93	2.16		ug/L	0.051	0.386	VC16-D15-18E	J Flag
6020A	RB-02102018	SE	CADMIUM, TOTAL	0.93	0.444	J	ug/L	0.046	0.35	VC16-D18-21E	J Flag
6020A	RB-02102018	SE	CADMIUM, TOTAL	0.93	0.148	J	ug/L	0.047	0.358	VC16-D21-24E	J Flag
6020A	RB-02102018	SE	CADMIUM, TOTAL	0.93	0.149	J	ug/L	0.045	0.339	VC16-D24-27.9E	J Flag
6020A	RB-02102018	SE	CADMIUM, TOTAL	0.93	0.161	J	ug/L	0.047	0.359	VC17-D15-18E	J Flag
6020A	RB-02102018	SE	CADMIUM, TOTAL	0.93	0.146	J	ug/L	0.048	0.364	VC17-D18-21E	J Flag
6020A	RB-02102018	SE	CADMIUM, TOTAL	0.93	0.156	J	ug/L	0.047	0.357	VC17-D21-24.1E	J Flag
6020A	RB-02102018	SE	CADMIUM, TOTAL	0.93	0.077	J	ug/L	0.03	0.229	VC40-D15-18E	J Flag
6020A	RB-02102018	SE	CADMIUM, TOTAL	0.93	0.092	J	ug/L	0.03	0.228	VC40-D18-21.7E	J Flag
6020A	RB-02102018	SE	CALCIUM, TOTAL	2050	557	J	ug/L	67.9	558	VC39-D12-15E	J Flag
6020A	RB-02102018	SE	CALCIUM, TOTAL	2050	559	J	ug/L	69.7	573	VC39-D15-18E	J Flag
6020A	RB-02102018	SE	CALCIUM, TOTAL	2050	457	J	ug/L	69.4	571	VC39-D18-21.2E	J Flag
6020A	RB-02102018	SE	CALCIUM, TOTAL	2050	370	J	ug/L	69.6	573	VC40-D15-18E	J Flag
6020A	RB-02102018	SE	CALCIUM, TOTAL	2050	529	J	ug/L	69.3	570	VC40-D18-21.7E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	37.13		ug/L	0.35	3.61	DUP6-D18-21E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	149		ug/L	0.374	3.86	VC16-D15-18E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	35.57		ug/L	0.34	3.5	VC16-D18-21E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	18.80		ug/L	0.347	3.58	VC16-D21-24E	J Flag

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Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qual.	Units	MDL	QL	Affected Sample	Sample Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	21.28		ug/L	0.329	3.39	VC16-D24-27.9E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	23.25		ug/L	0.348	3.59	VC17-D15-18E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	20.09		ug/L	0.353	3.64	VC17-D18-21E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	22.22		ug/L	0.346	3.57	VC17-D21-24.1E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	12.79		ug/L	0.216	2.23	VC39-D12-15E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	16.15		ug/L	0.222	2.29	VC39-D15-18E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	14.53		ug/L	0.221	2.28	VC39-D18-21.2E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	16.00		ug/L	0.222	2.29	VC40-D15-18E	J Flag
6020A	RB-02102018	SE	COPPER, TOTAL	35.30	18.50		ug/L	0.221	2.28	VC40-D18-21.7E	J Flag
6020A	RB-02102018	SE	MAGNESIUM, TOTAL	1360	3162		ug/L	13.8	112	VC39-D12-15E	J Flag
6020A	RB-02102018	SE	MAGNESIUM, TOTAL	1360	3521		ug/L	14.1	115	VC39-D15-18E	J Flag
6020A	RB-02102018	SE	MAGNESIUM, TOTAL	1360	3179		ug/L	14.1	114	VC39-D18-21.2E	J Flag
6020A	RB-02102018	SE	MAGNESIUM, TOTAL	1360	4322		ug/L	14.1	114	VC40-D15-18E	J Flag
6020A	RB-02102018	SE	MAGNESIUM, TOTAL	1360	3676		ug/L	14	114	VC40-D18-21.7E	J Flag
6020A	RB-02102018	SE	NICKEL, TOTAL	5.57	20.99		ug/L	0.298	1.12	VC39-D12-15E	J Flag
6020A	RB-02102018	SE	NICKEL, TOTAL	5.57	24.97		ug/L	0.306	1.15	VC39-D15-18E	J Flag
6020A	RB-02102018	SE	NICKEL, TOTAL	5.57	21.53		ug/L	0.305	1.14	VC39-D18-21.2E	J Flag
6020A	RB-02102018	SE	NICKEL, TOTAL	5.57	24.74		ug/L	0.305	1.14	VC40-D18-21.7E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	5706		ug/L	28.6	180	DUP6-D18-21E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	4631		ug/L	30.6	193	VC16-D15-18E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	5750		ug/L	27.8	175	VC16-D18-21E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	5069		ug/L	28.4	179	VC16-D21-24E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	6185		ug/L	26.9	170	VC16-D24-27.9E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	5855		ug/L	28.5	179	VC17-D15-18E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	5648		ug/L	28.9	182	VC17-D18-21E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	6088		ug/L	28.3	178	VC17-D21-24.1E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	2487		ug/L	17.7	112	VC39-D12-15E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	2090		ug/L	18.2	115	VC39-D15-18E	J Flag

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Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qual.	Units	MDL	QL	Affected Sample	Sample Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	1394		ug/L	18.1	114	VC39-D18-21.2E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	893		ug/L	18.2	114	VC40-D15-18E	J Flag
6020A	RB-02102018	SE	POTASSIUM, TOTAL	2270	916		ug/L	18.1	114	VC40-D18-21.7E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	9488		ug/L	21.1	271	DUP6-D18-21E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	12123		ug/L	22.6	289	VC16-D15-18E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	9374		ug/L	20.5	263	VC16-D18-21E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	7486		ug/L	20.9	268	VC16-D21-24E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	7878		ug/L	19.9	254	VC16-D24-27.9E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	7885		ug/L	21	269	VC17-D15-18E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	6140		ug/L	21.3	273	VC17-D18-21E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	5097		ug/L	20.9	268	VC17-D21-24.1E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	2198		ug/L	13.1	167	VC39-D12-15E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	2172		ug/L	13.4	172	VC39-D15-18E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	2280		ug/L	13.4	171	VC39-D18-21.2E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	2045		ug/L	13.4	172	VC40-D15-18E	J Flag
6020A	RB-02102018	SE	SODIUM, TOTAL	10800	1875		ug/L	13.4	171	VC40-D18-21.7E	J Flag
6020A	RB-02102018	SE	STRONTIUM, TOTAL	12.88	62.60		ug/L	0.438	1.79	VC16-D21-24E	J Flag
6020A	RB-02102018	SE	STRONTIUM, TOTAL	12.88	63.28		ug/L	0.416	1.7	VC16-D24-27.9E	J Flag
6020A	RB-02102018	SE	STRONTIUM, TOTAL	12.88	13.06		ug/L	0.274	1.12	VC39-D12-15E	J Flag
6020A	RB-02102018	SE	STRONTIUM, TOTAL	12.88	12.68		ug/L	0.281	1.15	VC39-D15-18E	J Flag
6020A	RB-02102018	SE	STRONTIUM, TOTAL	12.88	14.75		ug/L	0.28	1.14	VC39-D18-21.2E	J Flag
6020A	RB-02102018	SE	STRONTIUM, TOTAL	12.88	6.32		ug/L	0.281	1.14	VC40-D15-18E	J Flag
6020A	RB-02102018	SE	STRONTIUM, TOTAL	12.88	7.34		ug/L	0.28	1.14	VC40-D18-21.7E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	130		ug/L	4.69	18	DUP6-D18-21E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	298		ug/L	5.01	19.3	VC16-D15-18E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	130		ug/L	4.55	17.5	VC16-D18-21E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	101		ug/L	4.65	17.9	VC16-D21-24E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	113		ug/L	4.41	17	VC16-D24-27.9E	J Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qual.	Units	MDL	QL	Affected Sample	Sample Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	108		ug/L	4.66	17.9	VC17-D15-18E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	102		ug/L	4.74	18.2	VC17-D18-21E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	111		ug/L	4.64	17.8	VC17-D21-24.1E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	46.45		ug/L	2.9	11.2	VC39-D12-15E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	60.18		ug/L	2.98	11.5	VC39-D15-18E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	52.24		ug/L	2.97	11.4	VC39-D18-21.2E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	46.70		ug/L	2.98	11.4	VC40-D15-18E	J Flag
6020A	RB-02102018	SE	ZINC, TOTAL	80	38.38		ug/L	2.96	11.4	VC40-D18-21.7E	J Flag
SW8270DSIM	RB-02102018	SE	2-METHYLNAPHTHALENE	17.56	6.95	J	ng/mL	0.87	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	2-METHYLNAPHTHALENE	17.56	57.98		ng/mL	0.94	7.58	VC16-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	2-METHYLNAPHTHALENE	17.56	8.15	J	ng/mL	0.882	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	ACENAPHTHENE	4.15	3.15	J	ng/mL	0.786	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	ACENAPHTHENE	4.15	18.23		ng/mL	0.85	7.58	VC16-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	ACENAPHTHENE	4.15	2.80	J	ng/mL	0.796	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	ACENAPHTHYLENE	2.15	5.19	J	ng/mL	0.47	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	ACENAPHTHYLENE	2.15	5.25	J	ng/mL	0.476	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	ANTHRACENE	8.70	13.20		ng/mL	0.87	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	ANTHRACENE	8.70	12.97		ng/mL	0.882	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	ANTHRACENE	8.70	1.79	J	ng/mL	0.848	6.84	VC16-D21-24E	J Flag
SW8270DSIM	RB-02102018	SE	ANTHRACENE	8.70	1.76	J	ng/mL	0.837	6.75	VC16-D24-27.9E	J Flag
SW8270DSIM	RB-02102018	SE	ANTHRACENE	8.70	1.49	J	ng/mL	0.852	6.87	VC17-D21-24.1E	J Flag
SW8270DSIM	RB-02102018	SE	BENZ(A)ANTHRACENE	11.11	36.31		ng/mL	1.87	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	BENZ(A)ANTHRACENE	11.11	29.68		ng/mL	1.9	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(B)FLUORANTHENE	10.55	36.84		ng/mL	0.73	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(B)FLUORANTHENE	10.55	31.66		ng/mL	0.74	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(B)FLUORANTHENE	10.55	2.05	J	ng/mL	0.711	6.84	VC16-D21-24E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(B)FLUORANTHENE	10.55	2.64	J	ng/mL	0.702	6.75	VC16-D24-27.9E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(B)FLUORANTHENE	10.55	2.57	J	ng/mL	0.722	6.94	VC17-D15-18E	J Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qual.	Units	MDL	QL	Affected Sample	Sample Flag
SW8270DSIM	RB-02102018	SE	BENZO(B)FLUORANTHENE	10.55	1.68	J	ng/mL	0.761	7.32	VC17-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(B)FLUORANTHENE	10.55	3.14	J	ng/mL	0.715	6.87	VC17-D21-24.1E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(GHI)PERYLENE	6.04	25.74		ng/mL	0.578	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(GHI)PERYLENE	6.04	24.10		ng/mL	0.586	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(GHI)PERYLENE	6.04	1.79	J	ng/mL	0.563	6.84	VC16-D21-24E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(GHI)PERYLENE	6.04	3.16	J	ng/mL	0.556	6.75	VC16-D24-27.9E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(GHI)PERYLENE	6.04	2.83	J	ng/mL	0.572	6.94	VC17-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(GHI)PERYLENE	6.04	1.59	J	ng/mL	0.603	7.32	VC17-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(GHI)PERYLENE	6.04	2.11	J	ng/mL	0.566	6.87	VC17-D21-24.1E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(GHI)PERYLENE	6.04	1.69	J	ng/mL	0.353	4.28	VC40-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(GHI)PERYLENE	6.04	1.93	J	ng/mL	0.356	4.32	VC40-D18-21.7E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(K)FLUORANTHENE	8.49	24.39		ng/mL	0.723	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(K)FLUORANTHENE	8.49	21.00		ng/mL	0.732	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(K)FLUORANTHENE	8.49	1.31	J	ng/mL	0.695	6.75	VC16-D24-27.9E	J Flag
SW8270DSIM	RB-02102018	SE	BENZO(K)FLUORANTHENE	8.49	1.26	J	ng/mL	0.708	6.87	VC17-D21-24.1E	J Flag
SW8270DSIM	RB-02102018	SE	CHRYSENE	14.47	38.60		ng/mL	0.617	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	CHRYSENE	14.47	31.23		ng/mL	0.625	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	CHRYSENE	14.47	1.99	J	ng/mL	0.601	6.84	VC16-D21-24E	J Flag
SW8270DSIM	RB-02102018	SE	CHRYSENE	14.47	2.23	J	ng/mL	0.593	6.75	VC16-D24-27.9E	J Flag
SW8270DSIM	RB-02102018	SE	CHRYSENE	14.47	1.52	J	ng/mL	0.61	6.94	VC17-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	CHRYSENE	14.47	1.16	J	ng/mL	0.644	7.32	VC17-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	CHRYSENE	14.47	2.68	J	ng/mL	0.604	6.87	VC17-D21-24.1E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORANTHENE	42.07	53.80		ng/mL	1.28	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORANTHENE	42.07	54.26		ng/mL	1.3	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORANTHENE	42.07	3.48	J	ng/mL	1.25	6.84	VC16-D21-24E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORANTHENE	42.07	4.21	J	ng/mL	1.23	6.75	VC16-D24-27.9E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORANTHENE	42.07	3.12	J	ng/mL	1.27	6.94	VC17-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORANTHENE	42.07	2.62	J	ng/mL	1.34	7.32	VC17-D18-21E	J Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qual.	Units	MDL	QL	Affected Sample	Sample Flag
SW8270DSIM	RB-02102018	SE	FLUORANTHENE	42.07	4.28	J	ng/mL	1.26	6.87	VC17-D21-24.1E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORENE	7.23	9.02	J	ng/mL	0.469	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORENE	7.23	8.88	J	ng/mL	0.475	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORENE	7.23	3.21	J	ng/mL	0.457	6.84	VC16-D21-24E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORENE	7.23	3.33	J	ng/mL	0.451	6.75	VC16-D24-27.9E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORENE	7.23	2.38	J	ng/mL	0.464	6.94	VC17-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORENE	7.23	2.03	J	ng/mL	0.489	7.32	VC17-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	FLUORENE	7.23	3.95	J	ng/mL	0.459	6.87	VC17-D21-24.1E	J Flag
SW8270DSIM	RB-02102018	SE	INDENO(1,2,3-CD)PYRENE	5.89	24.14		ng/mL	2	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	INDENO(1,2,3-CD)PYRENE	5.89	22.49		ng/mL	2.03	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	NAPHTHALENE	32.43	10.05		ng/mL	0.628	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	NAPHTHALENE	32.43	91.49		ng/mL	0.679	7.58	VC16-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	NAPHTHALENE	32.43	13.98		ng/mL	0.636	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	PHENANTHRENE	54.16	27.32		ng/mL	0.828	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	PHENANTHRENE	54.16	172		ng/mL	0.895	7.58	VC16-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	PHENANTHRENE	54.16	27.35		ng/mL	0.839	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	PHENANTHRENE	54.16	3.83	J	ng/mL	0.807	6.84	VC16-D21-24E	J Flag
SW8270DSIM	RB-02102018	SE	PHENANTHRENE	54.16	4.20	J	ng/mL	0.796	6.75	VC16-D24-27.9E	J Flag
SW8270DSIM	RB-02102018	SE	PHENANTHRENE	54.16	3.37	J	ng/mL	0.819	6.94	VC17-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	PHENANTHRENE	54.16	3.21	J	ng/mL	0.864	7.32	VC17-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	PHENANTHRENE	54.16	4.82	J	ng/mL	0.811	6.87	VC17-D21-24.1E	J Flag
SW8270DSIM	RB-02102018	SE	PYRENE	25.98	57.75		ng/mL	0.716	7.02	DUP6-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	PYRENE	25.98	47.23		ng/mL	0.725	7.11	VC16-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	PYRENE	25.98	2.12	J	ng/mL	0.697	6.84	VC16-D21-24E	J Flag
SW8270DSIM	RB-02102018	SE	PYRENE	25.98	2.47	J	ng/mL	0.688	6.75	VC16-D24-27.9E	J Flag
SW8270DSIM	RB-02102018	SE	PYRENE	25.98	1.95	J	ng/mL	0.708	6.94	VC17-D15-18E	J Flag
SW8270DSIM	RB-02102018	SE	PYRENE	25.98	1.44	J	ng/mL	0.747	7.32	VC17-D18-21E	J Flag
SW8270DSIM	RB-02102018	SE	PYRENE	25.98	2.35	J	ng/mL	0.701	6.87	VC17-D21-24.1E	J Flag

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: June 15, 2018	Completed by: Eridania Marte

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qual.	Units	MDL	QL	Affected Sample	Sample Flag
6020A	RB-02112018	SE	BARIUM, TOTAL	2.43	7.78		ug/L	0.247	3.51	VC38-D15-18E	J Flag
6020A	RB-02112018	SE	CALCIUM, TOTAL	493	454	J	ug/L	71.1	585	VC38-D15-18E	J Flag
6020A	RB-02112018	SE	CALCIUM, TOTAL	493	336	J	ug/L	70.5	580	VC38-D18-21E	J Flag
6020A	RB-02112018	SE	CALCIUM, TOTAL	493	354	J	ug/L	66	542	VC38-D21-24E	J Flag
6020A	RB-02112018	SE	CALCIUM, TOTAL	493	370	J	ug/L	72	592	VC38-D24-26.1E	J Flag
6020A	RB-02112018	SE	CHROMIUM, TOTAL	2.31	11.09		ug/L	0.555	2.37	VC38-D24-26.1E	J Flag
6020A	RB-02112018	SE	MAGNESIUM, TOTAL	389	1541		ug/L	14.4	117	VC38-D15-18E	J Flag
6020A	RB-02112018	SE	MAGNESIUM, TOTAL	389	1902		ug/L	14.3	116	VC38-D18-21E	J Flag
6020A	RB-02112018	SE	MANGANESE, TOTAL	23.84	95.90		ug/L	0.519	2.34	VC38-D15-18E	J Flag
6020A	RB-02112018	SE	MANGANESE, TOTAL	23.84	95.48		ug/L	0.526	2.37	VC38-D24-26.1E	J Flag
6020A	RB-02112018	SE	MANGANESE, TOTAL	23.84	115		ug/L	0.56	2.52	VC41-ALT-D12-15E	J Flag
6020A	RB-02112018	SE	MANGANESE, TOTAL	23.84	107		ug/L	0.558	2.51	VC41-ALT-D15-18E	J Flag
6020A	RB-02112018	SE	MANGANESE, TOTAL	23.84	102.92		ug/L	0.535	2.41	VC41-ALT-D18-19.4E	J Flag
6020A	RB-02112018	SE	NICKEL, TOTAL	2.77	12.17		ug/L	0.338	1.27	DUP7-D15-18E	J Flag
6020A	RB-02112018	SE	NICKEL, TOTAL	2.77	12.50		ug/L	0.31	1.16	VC38-D18-21E	J Flag
6020A	RB-02112018	SE	NICKEL, TOTAL	2.77	13.10		ug/L	0.317	1.18	VC38-D24-26.1E	J Flag
6020A	RB-02112018	SE	NICKEL, TOTAL	2.77	9.83		ug/L	0.337	1.26	VC41-ALT-D12-15E	J Flag
6020A	RB-02112018	SE	NICKEL, TOTAL	2.77	10.30		ug/L	0.336	1.26	VC41-ALT-D15-18E	J Flag
6020A	RB-02112018	SE	NICKEL, TOTAL	2.77	11.09		ug/L	0.322	1.2	VC41-ALT-D18-19.4E	J Flag
6020A	RB-02112018	SE	SODIUM, TOTAL	3540	6416		ug/L	14.8	190	DUP7-D15-18E	J Flag
6020A	RB-02112018	SE	SODIUM, TOTAL	3540	2602		ug/L	13.7	175	VC38-D15-18E	J Flag
6020A	RB-02112018	SE	SODIUM, TOTAL	3540	2628		ug/L	13.6	174	VC38-D18-21E	J Flag
6020A	RB-02112018	SE	SODIUM, TOTAL	3540	1832		ug/L	12.7	163	VC38-D21-24E	J Flag

Method	Method Blank	Matrix	Analyte	Blank Result	Sample Result	Lab Qual.	Units	MDL	QL	Affected Sample	Sample Flag
6020A	RB-02112018	SE	SODIUM, TOTAL	3540	3080		ug/L	13.9	178	VC38-D24-26.1E	J Flag
6020A	RB-02112018	SE	SODIUM, TOTAL	3540	5504		ug/L	14.8	189	VC41-ALT-D12-15E	J Flag
6020A	RB-02112018	SE	SODIUM, TOTAL	3540	6381		ug/L	14.7	188	VC41-ALT-D15-18E	J Flag
6020A	RB-02112018	SE	SODIUM, TOTAL	3540	5295		ug/L	14.1	181	VC41-ALT-D18-19.4E	J Flag
6020A	RB-02112018	SE	STRONTIUM, TOTAL	3.60	5.86		ug/L	0.287	1.17	VC38-D15-18E	J Flag
6020A	RB-02112018	SE	STRONTIUM, TOTAL	3.60	11.76		ug/L	0.284	1.16	VC38-D18-21E	J Flag
6020A	RB-02112018	SE	STRONTIUM, TOTAL	3.60	13.89		ug/L	0.266	1.08	VC38-D21-24E	J Flag
6020A	RB-02112018	SE	STRONTIUM, TOTAL	3.60	10.46		ug/L	0.29	1.18	VC38-D24-26.1E	J Flag
6020A	RB-02112018	SE	STRONTIUM, TOTAL	3.60	16.40		ug/L	0.309	1.26	VC41-ALT-D12-15E	J Flag
6020A	RB-02112018	SE	STRONTIUM, TOTAL	3.60	16.42		ug/L	0.308	1.26	VC41-ALT-D15-18E	J Flag
6020A	RB-02112018	SE	ZINC, TOTAL	14.80	30.83		ug/L	3.29	12.7	DUP7-D15-18E	J Flag
6020A	RB-02112018	SE	ZINC, TOTAL	14.80	27.33		ug/L	3.04	11.7	VC38-D15-18E	J Flag
6020A	RB-02112018	SE	ZINC, TOTAL	14.80	27.25		ug/L	3.01	11.6	VC38-D18-21E	J Flag
6020A	RB-02112018	SE	ZINC, TOTAL	14.80	47.49		ug/L	2.82	10.8	VC38-D21-24E	J Flag
6020A	RB-02112018	SE	ZINC, TOTAL	14.80	33.16		ug/L	3.08	11.8	VC38-D24-26.1E	J Flag
6020A	RB-02112018	SE	ZINC, TOTAL	14.80	24.93		ug/L	3.28	12.6	VC41-ALT-D12-15E	J Flag
6020A	RB-02112018	SE	ZINC, TOTAL	14.80	24.35	J	ug/L	3.26	12.6	VC41-ALT-D15-18E	J Flag
6020A	RB-02112018	SE	ZINC, TOTAL	14.80	24.40	J	ug/L	3.13	12	VC41-ALT-D18-19.4E	J Flag

Table 3 – List of Samples with Surrogates outside Control Limits

None.

Table 4 - List MS/MSD Recoveries outside Control Limits

Method	Parent Sample	Analyte	Dil Fac	Unit	MS	MSD	Low Limit	High Limit	Qualifier
6020A	VC16-D21-24E	ALUMINUM, TOTAL	10	mg/kg	888	895	75	125	None - 4X
6020A	VC16-D21-24E	ANTIMONY, TOTAL	10	mg/kg	70	72	75	125	J Flag
6020A	VC16-D21-24E	CALCIUM, TOTAL	10	mg/kg	222	303	75	125	None - 4X
6020A	VC16-D21-24E	IRON, TOTAL	10	mg/kg	852	1290	75	125	None - 4X
6020A	VC16-D21-24E	MAGNESIUM, TOTAL	10	mg/kg	142	150	75	125	None - 4X
6020A	VC16-D21-24E	MANGANESE, TOTAL	10	mg/kg	145	146	75	125	None - 4X
6020A	VC16-D21-24E	POTASSIUM, TOTAL	10	mg/kg	139	147	75	125	J Flag
6020A	VC16-D21-24E	SILVER, TOTAL	10	mg/kg	17	17	75	125	J Flag
6020A	VC16-D21-24E	SODIUM, TOTAL	10	mg/kg	129	134	75	125	J Flag
6020A	VC16-D21-24E	TITANIUM, TOTAL	10	mg/kg	156	158	75	125	J Flag
6020A	VC40-D15-18E	ALUMINUM, TOTAL	10	mg/kg	352	306	75	125	None - 4X
6020A	VC40-D15-18E	CALCIUM, TOTAL	10	mg/kg	134	135	75	125	J Flag
6020A	VC40-D15-18E	IRON, TOTAL	10	mg/kg	352	742	75	125	None - 4X
6020A	VC40-D15-18E	SILVER, TOTAL	10	mg/kg	17	16	75	125	UJ Flag
SW8081B	VC16-D21-24E	ENDRIN ALDEHYDE	1	ug/kg	24.7	--	40	140	UJ Flag
SW8081B	VC16-D21-24E	METHOXYCHLOR	1	ug/kg	33.4	--	40	140	UJ Flag

Table 4A – List RPDs outside Control Limits

Method	Parent Sample	Analyte	Dil Fac	Unit	RPD	RPD Limit	Qualifier	Sample Type
LLOYDKAHN	VC16-D21-24E	TOTAL ORGANIC CARBON (REP2)	1	mg/kg	26	25	None	MSD
SW8081B	VC16-D21-24E	ENDRIN ALDEHYDE	1	ug/kg	60	50	UJ Flag	MSD

Table 4B – List of Laboratory Replicate outside Control Limits

Method	Parent Sample	Sample ID	Sample Type	Analyte	Dil Fac	Units	RPD	RPD Limit	Exceeds RPD	Qualifier
6020A	RB-02102018	WG1089511-4	LR	CADMIUM, TOTAL	1	mg/l	107	20	High	None
6020A	RB-02102018	WG1089511-4	LR	COPPER, TOTAL	1	mg/l	139	20	High	None
6020A	RB-02102018	WG1089511-4	LR	LEAD, TOTAL	1	mg/l	26	20	High	None
6020A	RB-02102018	WG1089511-4	LR	POTASSIUM, TOTAL	1	mg/l	44	20	High	None

Method	Parent Sample	Sample ID	Sample Type	Analyte	Dil Fac	Units	RPD	RPD Limit	Exceeds RPD	Qualifier
6020A	RB-02102018	WG1089511-4	LR	TIN, TOTAL	1	mg/l	43	20	High	None
6020A	RB-02102018	WG1089511-4	LR	ZINC, TOTAL	1	mg/l	41	20	High	None

Table 5 - List LCS Recoveries outside Control Limits
None.

Table 5A – List RPDs outside Control Limits
None.

Table 5B – List SRM Recoveries outside Control Limits
None.

Table 6 –Samples that were Reanalyzed/Diluted

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
DUP6-D18-21E	SE	6020A	FD	Y	26	10	INITIAL	
DUP6-D18-21E	SE	E7474/SW846	FD	Y	1	5	INITIAL	
DUP7-D15-18E	SE	6020A	FD	N	5	10	INITIAL	Antimony, cadmium, selenium, silver, and thallium results non-detect and reported with elevated reporting limits.
DUP7-D15-18E	SE	6020A	FD	Y	21	10	INITIAL	
DUP7-D15-18E	SE	E7474/SW846	FD	N	1	5	INITIAL	
VC16-D15-18E	SE	6020A	N	Y	26	10	INITIAL	
VC16-D15-18E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC16-D18-21E	SE	6020A	N	Y	26	10	INITIAL	
VC16-D18-21E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC16-D21-24E	SE	6020A	N	Y	26	10	INITIAL	
VC16-D21-24E	SE	E7474/SW846	N	N	1	5	INITIAL	
VC16-D24-27.9E	SE	6020A	N	Y	26	10	INITIAL	
VC16-D24-27.9E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC17-D15-18E	SE	6020A	N	Y	26	10	INITIAL	

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
VC17-D15-18E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC17-D18-21E	SE	6020A	N	Y	26	10	INITIAL	
VC17-D18-21E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC17-D21-24.1E	SE	6020A	N	Y	26	10	INITIAL	
VC17-D21-24.1E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC38-D15-18E	SE	6020A	N	N	5	10	INITIAL	Antimony, cadmium, selenium, silver, and thallium results non-detect and reported with elevated reporting limits.
VC38-D15-18E	SE	6020A	N	Y	21	10	INITIAL	
VC38-D15-18E	SE	E7474/SW846	N	N	1	5	INITIAL	
VC38-D18-21E	SE	6020A	N	N	3	10	INITIAL	Cadmium, selenium, and thallium results non-detect and reported with elevated reporting limits.
VC38-D18-21E	SE	6020A	N	Y	23	10	INITIAL	
VC38-D18-21E	SE	E7474/SW846	N	N	1	5	INITIAL	
VC38-D21-24E	SE	6020A	N	N	4	10	INITIAL	Cadmium, selenium, silver, and thallium results non-detect and reported with elevated reporting limits.
VC38-D21-24E	SE	6020A	N	Y	22	10	INITIAL	
VC38-D21-24E	SE	E7474/SW846	N	N	1	5	INITIAL	
VC38-D24-26.1E	SE	6020A	N	N	6	10	INITIAL	Antimony, cadmium, molybdenum, selenium, silver, and thallium results non-detect and reported with elevated reporting limits.
VC38-D24-26.1E	SE	6020A	N	Y	20	10	INITIAL	
VC38-D24-26.1E	SE	E7474/SW846	N	N	1	5	INITIAL	
VC39-D12-15E	SE	6020A	N	N	5	10	INITIAL	Antimony, cadmium, selenium, silver, and thallium results non-detect and reported with elevated reporting limits.
VC39-D12-15E	SE	6020A	N	Y	21	10	INITIAL	
VC39-D12-15E	SE	E7474/SW846	N	N	1	5	INITIAL	

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
VC39-D15-18E	SE	6020A	N	N	5	10	INITIAL	Antimony, cadmium, selenium, silver, and thallium results non-detect and reported with elevated reporting limits.
VC39-D15-18E	SE	6020A	N	Y	21	10	INITIAL	
VC39-D15-18E	SE	E7474/SW846	N	Y	1	5	INITIAL	
VC39-D18-21.2E	SE	6020A	N	N	4	10	INITIAL	Antimony, cadmium, silver, and thallium results non-detect and reported with elevated reporting limits.
VC39-D18-21.2E	SE	6020A	N	Y	22	10	INITIAL	
VC39-D18-21.2E	SE	E7474/SW846	N	N	1	5	INITIAL	
VC40-D15-18E	SE	6020A	N	N	1	10	INITIAL	Silver results non-detect and reported with elevated reporting limits.
VC40-D15-18E	SE	6020A	N	Y	25	10	INITIAL	
VC40-D15-18E	SE	E7474/SW846	N	N	1	5	INITIAL	
VC40-D18-21.7E	SE	6020A	N	N	4	10	INITIAL	Antimony, selenium, silver, and thallium results non-detect and reported with elevated reporting limits.
VC40-D18-21.7E	SE	6020A	N	Y	22	10	INITIAL	
VC40-D18-21.7E	SE	E7474/SW846	N	N	1	5	INITIAL	
VC41ALT-D12-15E	SE	6020A	N	N	5	10	INITIAL	Antimony, cadmium, selenium, silver, and thallium results non-detect and reported with elevated reporting limits.
VC41ALT-D12-15E	SE	6020A	N	Y	21	10	INITIAL	
VC41ALT-D12-15E	SE	E7474/SW846	N	N	1	5	INITIAL	
VC41ALT-D15-18E	SE	6020A	N	N	5	10	INITIAL	Antimony, cadmium, selenium, silver, and thallium results non-detect and reported with elevated reporting limits.
VC41ALT-D15-18E	SE	6020A	N	Y	21	10	INITIAL	
VC41ALT-D15-18E	SE	E7474/SW846	N	N	1	5	INITIAL	

Sample Name	Matrix	Method	Sample Type	Detect Flag	Number of Chemicals	Dilution Factor	Test Type	Notes
VC41ALT-D18-19.4E	SE	6020A	N	N	5	10	INITIAL	Antimony, cadmium, selenium, silver, and thallium results non-detect and reported with elevated reporting limits.
VC41ALT-D18-19.4E	SE	6020A	N	Y	21	10	INITIAL	
VC41ALT-D18-19.4E	SE	E7474/SW846	N	N	1	5	INITIAL	

Table 7 – Summary of Field Duplicate Results

Method	Analyte	Unit	Matrix	PQL	VC16-D18-21E	DUP6-D18-21E	RPD	RPD Rating	Sample Qual
SW8270DSIM	2-METHYLNAPHTHALENE	ug/kg	SE	7.11	5.8	4.88	17.2%	Good	None
SW8081B	4,4'-DDD	ug/kg	SE	0.343	ND	0.499	NC	--	None
SW8270DSIM	ACENAPHTHENE	ug/kg	SE	7.11	1.99	2.21	10.5%	Good	None
SW8270DSIM	ACENAPHTHYLENE	ug/kg	SE	7.11	3.73	3.64	2.4%	Good	None
6020A	ALUMINUM, TOTAL	mg/kg	SE	175	15600	15800	1.3%	Good	None
SW8270DSIM	ANTHRACENE	ug/kg	SE	7.11	9.22	9.26	0.4%	Good	None
6020A	ANTIMONY, TOTAL	mg/kg	SE	2.8	0.461	0.392	16.2%	Good	None
6020A	ARSENIC, TOTAL	mg/kg	SE	0.875	12.6	13	3.1%	Good	None
6020A	BARIUM, TOTAL	mg/kg	SE	5.25	32.4	36.1	10.8%	Good	None
SW8270DSIM	BENZ(A)ANTHRACENE	ug/kg	SE	7.11	21.1	25.5	18.9%	Good	None
SW8270DSIM	BENZO(A)PYRENE	ug/kg	SE	7.11	24.6	27.2	10.0%	Good	None
SW8270DSIM	BENZO(B)FLUORANTHENE	ug/kg	SE	7.11	22.5	25.9	14.0%	Good	None
SW8270DSIM	BENZO(GHI)PERYLENE	ug/kg	SE	7.11	17.1	18.1	5.7%	Good	None
SW8270DSIM	BENZO(K)FLUORANTHENE	ug/kg	SE	7.11	14.9	17.1	13.8%	Good	None
6020A	BERYLLIUM, TOTAL	mg/kg	SE	0.525	0.939	0.951	1.3%	Good	None
6020A	CADMIUM, TOTAL	mg/kg	SE	0.35	0.311	0.28	10.5%	Good	None
6020A	CALCIUM, TOTAL	mg/kg	SE	875	8730	9240	5.7%	Good	None
6020A	CHROMIUM, TOTAL	mg/kg	SE	3.5	45.1	47.3	4.8%	Good	None
SW8270DSIM	CHRYSENE	ug/kg	SE	7.11	22.2	27.1	19.9%	Good	None
6020A	COBALT, TOTAL	mg/kg	SE	0.875	10.2	10.4	1.9%	Good	None
6020A	COPPER, TOTAL	mg/kg	SE	3.5	24.9	26.8	7.4%	Good	None
SW8270DSIM	DIBENZ(A,H)ANTHRACENE	ug/kg	SE	7.11	3.91	4.83	21.1%	Good	None

Method	Analyte	Unit	Matrix	PQL	VC16-D18-21E	DUP6-D18-21E	RPD	RPD Rating	Sample Qual
SW8270DSIM	FLUORANTHENE	ug/kg	SE	7.11	38.6	37.8	2.1%	Good	None
SW8270DSIM	FLUORENE	ug/kg	SE	7.11	6.31	6.33	0.3%	Good	None
SW8270DSIM	INDENO(1,2,3-CD)PYRENE	ug/kg	SE	7.11	16	16.9	5.5%	Good	None
6020A	IRON, TOTAL	mg/kg	SE	350	34700	35000	0.9%	Good	None
6020A	LEAD, TOTAL	mg/kg	SE	1.05	24.5	26.3	7.1%	Good	None
6020A	MAGNESIUM, TOTAL	mg/kg	SE	175	9010	9190	2.0%	Good	None
6020A	MANGANESE, TOTAL	mg/kg	SE	3.5	415	444	6.8%	Good	None
E7474/SW846	MERCURY, TOTAL	mg/kg	SE	0.026	0.219	0.275	22.7%	Good	None
6020A	MOLYBDENUM, TOTAL	mg/kg	SE	1.4	1.53	1.61	5.1%	Good	None
SW8270DSIM	NAPHTHALENE	ug/kg	SE	7.11	9.94	7.05	34.0%	Good	None
6020A	NICKEL, TOTAL	mg/kg	SE	1.75	28.3	28.2	0.4%	Good	None
SW8270DSIM	PHENANTHRENE	ug/kg	SE	7.11	19.4	19.2	1.0%	Good	None
6020A	POTASSIUM, TOTAL	mg/kg	SE	175	4030	4120	2.2%	Good	None
SW8270DSIM	PYRENE	ug/kg	SE	7.11	33.6	40.5	18.6%	Good	None
6020A	SELENIUM, TOTAL	mg/kg	SE	3.5	3.1	3.57	14.1%	Good	None
6020A	SILVER, TOTAL	mg/kg	SE	0.875	0.517	0.572	10.1%	Good	None
6020A	SODIUM, TOTAL	mg/kg	SE	263	6560	6850	4.3%	Good	None
A2540G	SOLIDS, TOTAL	percent	SE	0.1	54.5	53.7	1.5%	Good	None
6020A	STRONTIUM, TOTAL	mg/kg	SE	1.75	50.6	58.9	15.2%	Good	None
6020A	THALLIUM, TOTAL	mg/kg	SE	0.35	0.147	0.143	2.8%	Good	None
6020A	TIN, TOTAL	mg/kg	SE	2.8	2.82	2.58	8.9%	Good	None
6020A	TITANIUM, TOTAL	mg/kg	SE	0.875	330	344	4.2%	Good	None
LLOYDKAHN	TOTAL ORGANIC CARBON (REP1)	mg/kg	SE	500	16100	16100	0.0%	Good	None
LLOYDKAHN	TOTAL ORGANIC CARBON (REP2)	mg/kg	SE	500	16900	16900	0.0%	Good	None
6020A	VANADIUM, TOTAL	mg/kg	SE	1.75	41.8	44	5.1%	Good	None
6020A	ZINC, TOTAL	mg/kg	SE	17.5	91.4	93.6	2.4%	Good	None

Method	Analyte	Unit	Matrix	PQL	VC41-ALT-D15-18E	DUP7-D15-18E	RPD	RPD Rating	Sample Qual
6020A	ALUMINUM, TOTAL	mg/kg	SE	126	2310	2880	22.0%	Good	None
6020A	ARSENIC, TOTAL	mg/kg	SE	0.628	1.93	2.27	16.2%	Good	None
6020A	BARIUM, TOTAL	mg/kg	SE	3.77	6.75	8.09	18.1%	Good	None

Method	Analyte	Unit	Matrix	PQL	VC41-ALT-D15-18E	DUP7-D15-18E	RPD	RPD Rating	Sample Qual
6020A	BERYLLIUM, TOTAL	mg/kg	SE	0.377	0.154	0.158	2.6%	Good	None
6020A	CALCIUM, TOTAL	mg/kg	SE	628	1420	1620	13.2%	Good	None
6020A	CHROMIUM, TOTAL	mg/kg	SE	2.51	6.76	8.4	21.6%	Good	None
6020A	COBALT, TOTAL	mg/kg	SE	0.628	1.87	2.33	21.9%	Good	None
6020A	COPPER, TOTAL	mg/kg	SE	2.51	2.02	2.48	20.4%	Good	None
6020A	IRON, TOTAL	mg/kg	SE	251	5240	6280	18.1%	Good	None
6020A	LEAD, TOTAL	mg/kg	SE	0.754	2.15	2.63	20.1%	Good	None
6020A	MAGNESIUM, TOTAL	mg/kg	SE	126	1680	2020	18.4%	Good	None
6020A	MANGANESE, TOTAL	mg/kg	SE	2.51	53.8	66.1	20.5%	Good	None
6020A	MOLYBDENUM, TOTAL	mg/kg	SE	1	0.402	0.431	7.0%	Good	None
6020A	NICKEL, TOTAL	mg/kg	SE	1.26	5.19	6.17	17.3%	Good	None
6020A	POTASSIUM, TOTAL	mg/kg	SE	126	835	958	13.7%	Good	None
6020A	SODIUM, TOTAL	mg/kg	SE	188	3200	3250	1.6%	Good	None
A2540G	SOLIDS, TOTAL	percent	SE	0.1	75.4	75.3	0.1%	Good	None
6020A	STRONTIUM, TOTAL	mg/kg	SE	1.26	8.25	9.78	17.0%	Good	None
6020A	TIN, TOTAL	mg/kg	SE	2.51	0.25	0.269	7.3%	Good	None
6020A	TITANIUM, TOTAL	mg/kg	SE	0.628	104	120	14.3%	Good	None
LLOYDKAHN	TOTAL ORGANIC CARBON (REP1)	mg/kg	SE	500	1110	1070	3.7%	Good	None
LLOYDKAHN	TOTAL ORGANIC CARBON (REP2)	mg/kg	SE	500	1190	1040	13.5%	Good	None
6020A	VANADIUM, TOTAL	mg/kg	SE	1.26	7.75	9.03	15.3%	Good	None
6020A	ZINC, TOTAL	mg/kg	SE	12.6	12.2	15.6	24.5%	Good	None

Acronym List and Table Key:

BD = blank spike duplicate
BS = blank spike
COC = chain of custody
CVAA = cold vapor atomic absorption
DUSR = data usability summary report
FD = field duplicate sample
GC/ECD = gas chromatography / electron capture detection

Data Usability Summary Report	Project: NESE Offshore Sampling
Date Completed: June 15, 2018	Completed by: Eridania Marte

Acronym List and Table Key:

GC/MS	=	gas chromatography / mass spectrometry
ICP/MS	=	Inductively coupled / mass spectrometry
LB	=	laboratory blank
LCS	=	laboratory control sample
LCSD	=	laboratory control sample duplicate
LR	=	laboratory replicate
MB	=	method blank
MS	=	matrix spike
MSD	=	matrix spike duplicate
N	=	normal field sample
NC	=	not calculated
ND	=	not detected
NJDEP	=	New Jersey Department of Environmental Protection
NYSDEC	=	New York State Department of Environmental Conservation
PCB	=	polychlorinated biphenyl
PQL	=	practical quantitation limit
QA	=	quality assurance
QAPP	=	quality assurance project plan
QC	=	quality control
RB	=	rinsate blank sample
RPD	=	relative percent difference
SDG	=	sample delivery group
SRM	=	standard reference material
SVOC	=	semi-volatile organic compounds
TB	=	trip blank sample
VOC	=	volatile organic compound