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Evaluation of Dredged Material Proposed for Ocean Disposal from Liberty Island Anchorage, New York

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Battelle Marine Sciences Laboratory Sequim, Washington

November 1996

Prepared for the U.S. Army Corps of Engineers - New York District under a Related Services Agreement with the U.S. Department of Energy under Contract DE-AC06-76RLO 1830

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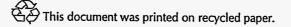
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EVALUATION OF DREDGED MATERIAL PROPOSED FOR OCEAN DISPOSAL FROM LIBERTY ISLAND ANCHORAGE, NEW YORK

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Summary

Sediment samples were collected from the Liberty Island Anchorage and Military Ocean Terminal in Bayonne, New Jersey (MOTBY), during a survey conducted from June 7 through June 10, 1994. Tests and analyses were conducted on Liberty Island Anchorage sediment core samples according to the manual developed by the U.S. Army Corps of Engineers (USACE) and the U.S. Environmental Protection Agency (EPA), Evaluation of Dredged Material Proposed for Ocean Disposal (Testing Manual), commonly referred to as the "Green Book," and the regional manual developed by the USACE-New York District (NYD) and EPA Region II, Guidance for Performing Tests on Dredged Material to be Disposed of in Ocean Waters. The evaluation of proposed dredged material from Liberty Island Anchorage consisted of bulk sediment chemical analyses, chemical analyses of site water and elutriate, and water-column and benthic acute toxicity tests. Individual sediment core samples collected from Liberty Island Anchorage were analyzed for grain size, moisture content, and total organic carbon (TOC). A composite sediment sample, representing the entire area proposed for dredging, was analyzed for bulk density, metals, chlorinated pesticides, polychlorinated biphenyl (PCB) congeners. polynuclear aromatic hydrocarbons (PAH), 1,4-dichlorobenzene, and dioxin/furan congeners. Site water and elutriate water, prepared from the suspended-particulate phase (SPP) of Liberty Island Anchorage sediment, were analyzed for metals, pesticides, and PCBs. Water-column or SPP toxicity tests were performed with three species, the mysid Mysidopsis bahia, the juvenile silverside Menidia beryllina, and larvae of the mussel Mytilus galloprovincialis. Benthic acute toxicity tests were performed with the amphipod, Ampelisca abdita, and the mysid M. bahia. The amphipod benthic toxicity test procedures followed EPA guidance for reduction of total ammonia concentrations in test systems prior to test initiation. A similar procedure was followed for the mysid toxicity test. Bioaccumulation tests were conducted with the burrowing, polychaete worm Nereis virens, the deposit-feeding bent-nose clam Macoma nasuta, and the suspension-feeding clam, Tapes japonica. Tissues from each species were archived for possible future analysis of metals, pesticides/PCB congeners, PAHs, 1,4-dichlorobenzene, or dioxin/furan congener analyses.

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Liberty Island Anchorage sediment core samples were predominantly black, silt-clay material with TOC concentrations ranging from 1.9% to 4.2%. The Liberty Island Anchorage sediment composite sample contained elevated levels of Cr, Cu, Pb, Zn, DDT and the family of DDT compounds, and had total PCB and PAH concentrations of 1738 μ g/kg and 10,060 μ g/kg respectively. Three dioxin/furan congeners were detected at levels at least one order of magnitude higher than the other congeners; these congeners were OCDD, 1,2,3,4,6,7,8-HpCDD, and OCDF.

In water-column toxicity tests, 100% SPP treatments of Liberty Island Anchorage sediments were not acutely toxic to *M. bahia*, *M. beryllina*, or *M. galloprovincialis*. The median lethal concentrations (LC_{50}) for these tests could not be calculated because there was not 50% mortality in any SPP treatment. The median effective concentration (EC_{50}) for *M. galloprovincialis* normal development, a more sensitive measure than survival, was 28.5% SPP.

Liberty Island Anchorage sediments were acutely toxicity to *A. abdita* (52%) relative to Mud Dump Reference Site sediments (94%). Liberty Island Anchorage sediments were not acutely toxic to *M. Bahia* (95%) relative to the Mud Dump Reference Site sediments (88%).

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1.0 Introduction

1.1 Project Objectives

The objective of the Liberty Island Anchorage project was to evaluate proposed dredged material from Liberty Island Anchorage located in the Upper Bay of New York Harbor to determine its suitability for unconfined ocean disposal at the Mud Dump Site. The Mud Dump Site is the present dredged material disposal site for the Ports of New York and New Jersey. It lies in the apex of the New York Bight about 6 miles east of Sandy Hook, New Jersey, and 12 miles south of Rockaway Point, New York.

A reference sediment, referred to as the Mud Dump Reference Site, was also collected and compared with the Liberty Island Anchorage sediments to evaluate potential impacts of disposal of these sediments. The Mud Dump Reference sediment is located approximately 2.6 miles southwest of the center of the Mud Dump Site.

Chemical analyses were conducted on 10 Liberty Island Anchorage sediment core samples, and chemical analysis and biological testing were performed on one Liberty Island Anchorage sediment composite (COMP LI), which is a homogenous mixture of all 10 field samples. These analyses were conducted according to the manual developed by the U.S. Army Corps of Engineers (USACE) and the U.S. Environmental Protection Agency (EPA), *Evaluation of Dredged Material Proposed for Ocean Disposal (Testing Manual)* (EPA/USACE 1991), commonly referred to as the "Green Book," and the regional manual developed by the USACE-New York District (NYD) and EPA Region II, *Guidance for Performing Tests on Dredged Material to be Disposed of in Ocean Waters* (USACE-NYD/EPA Region II 1992), hereinafter referred to as the "Regional Guidance Manual." The Regional Guidance Manual provides specifications for the use of local or appropriate test species in biological tests and identifies chemical contaminants of concern:

As required by the Regional Guidance Manual, the evaluation of proposed dredged material from the Liberty Island Anchorage consisted of bulk sediment chemical analyses, chemical analyses of site water and elutriate, water-column and benthic acute toxicity tests, and bioaccumulation studies. Individual sediment core samples collected from Liberty Island

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Anchorage were analyzed for grain size, moisture content, and total organic carbon (TOC). A composite sediment sample, representing the entire area proposed for dredging, was analyzed for grain size, bulk density, metals, chlorinated pesticides, polychlorinated biphenyl (PCB) congeners, polynuclear aromatic hydrocarbons (PAH), 1,4-dichlorobenzene, and dioxin/furan congeners. Site water and elutriate water, which was prepared from the suspended-particulate phase (SPP) of Liberty Island Anchorage sediment, were analyzed for metals, pesticides, and PCBs. Water-column or SPP toxicity tests were performed with three species, the mysid *Mysidopsis bahia*, the juvenile silverside *Menidia beryllina*, and larvae of the mussel *Mytilus galloprovincialis*. Benthic acute toxicity tests were performed with the amphipod *Ampelisca abdita* and the mysid *M. bahia*. Bioaccumulation tests were conducted with the burrowing worm *Nereis virens*, the surface-feeding clam *Macoma nasuta*, and the suspension feeding clam *Tapes japonica*.

1.2 Project Background

The proposed Liberty Island Anchorage project area is shown in (Figure 1.1). The project requires dredging and disposal of an estimated 30,000 cu yd of sediment. Project depth of the channel is -18 ft mean low water (MLW) plus 1 ft of overdepth. Liberty Island Anchorage was evaluated in parallel with sediments collected from the Military Ocean Terminal (MOTBY) in Bayonne, New Jersey. Sediment samples from these waterways were collected during a survey that took place from June 7 through June 10, 1994. This sampling and testing effort became known as New York-3 Program. Combining sample collection and evaluation of multiple dredged material projects was more cost-effective for the USACE-NYD, because the expense of reference site testing and quality control analyses could be shared between projects.

1.3 Organization of Report

Following this introduction, Section 2 presents the methods and materials used for sample collection, sample processing, sediment sample analysis of physical and chemical

parameters, and quality assurance. Results of all physical/chemical analyses and bioassays are presented in Section 3. A discussion of the results and conclusions are provided in Section 4. Section 5 lists the literature cited in this report. Appendix A contains tabulated quality control data for all physical and chemical sediment analyses. Appendix B contains results of replicate sample analyses and quality control data for site water and elutriate chemical parameters. Appendix C contains raw data associated with water-column toxicity tests, such as water quality measurements, test animal survival data, and results of reference toxicant tests. Similar data for benthic acute toxicity tests are provided in Appendix D. Appendix E contains water quality measurements, test animal survival data, and results of reference toxicant tests for the bioaccumulation tests.

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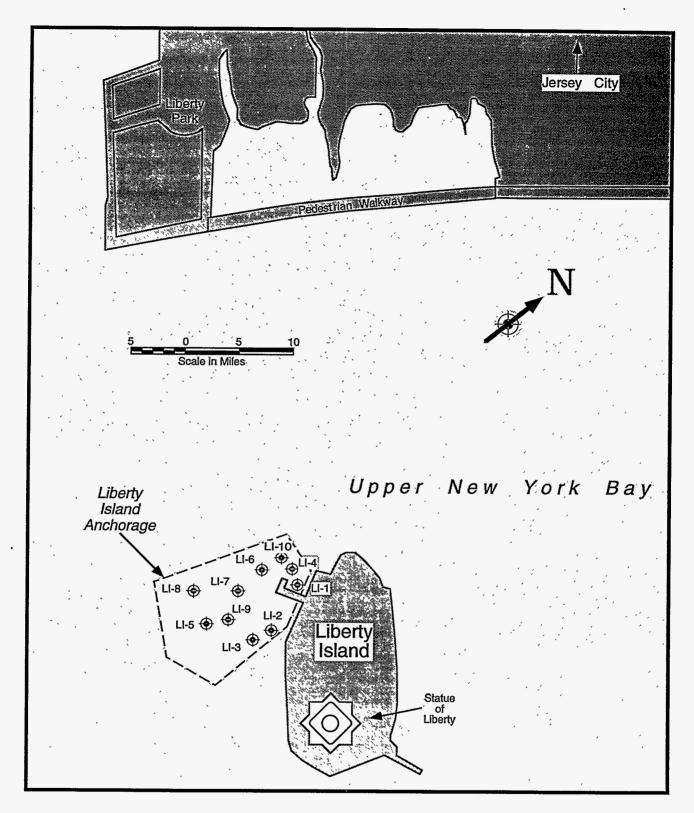


FIGURE 1.1. Location of Liberty Island Anchorage Project Area and Sample Collection Stations

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2.0 Materials and Methods

2.1 Sediment and Water Collection

Sediment samples were collected from 10 stations within the Liberty Island Anchorage. Sampling locations were selected by the USACE-NYD based on recent bathymetric surveys. The locations, their coordinates, and water and core sampling depths are presented with the sampling results in Section 3.0. Water samples were collected at a representative location in Liberty Island Anchorage and in the Mud Dump Site. Reference sediment was collected from the Mud Dump Reference Site. Liberty Island Anchorage samples were collected aboard the *M/V Hayward* (first 4 samples) and the *M/V Driftmaster* (remaining samples), which are owned and operated by USACE-NYD at Caven Point, New Jersey.

2.1.1 Test Sediment and Site Water Sampling

Test sediment core samples were collected using a vibracore sampler deployed from the *Hayward* and the *Driftmaster*. The approximate sampling locations were first determined by reference to landmarks, such as shoreline features or buoys, as well as by water depth. Then, a hand-held Magellan Global Positioning System (GPS) was used to identify and record (within 30 m) each sampling station. The vessel's LORAN was available as a backup system. Water depth at the time of sampling was measured by a fathometer on the ship. The actual water depth was corrected to MLW depth by correcting to the tide height at the time the depth was recorded. The difference between the MLW depth and the project depth, plus 1 ft overdepth, equalled the amount of core required.

Core samples were collected aboard the *Hayward* and the *Driftmaster* using a vibracore owned and operated by Ocean Surveys, Inc., Old Saybrook, Connecticut. The vibracore sampler consisted of a 4-in. outer diameter (OD), steel core barrel attached to an electric vibratory hammer. The vibratory hammer could be fitted to steel core barrels of various lengths, depending on the length of core needed. To collect a core sample, the core barrel was fitted with a 3.125-in. interior diameter (ID), steam-cleaned, Lexan polycarbonate tube. The vibracore was then suspended by the ship's crane. Once the coring apparatus was directly

above the sampling station, the core was lowered through the water to the sediment surface. At this point, the station coordinates were recorded from the Magellan GPS, and water depth was recorded from the ship's fathometer. The vibratory hammer was switched on until the corer penetrated through the sediment to the desired project depth. Adequate penetration was determined relative to marks on the outside of the core barrel and on the cable suspending the vibracore from the crane. The vibracore apparatus was then pulled out of the sediment and lowered onto the ship's deck. A cutter-head and core-catcher assembly prevented loss of the sediment through the bottom of the core liner. After each core was brought on board, the liner was pulled from the barrel and the length of cored sediment was measured from the mudline to determine whether the appropriate depth had been reached. If not, the liner was replaced and a second core sample was attempted. If the sediment core length achieved project depth plus 1 ft overdepth, it was capped, sealed with tape, and labeled. As cores were collected, they were shuttled back to the USACE Caven Point facility by USACE personnel in a small Boston Whaler. Cores were immediately placed in refrigerated storage (~4°C).

A surface-water sample for site water chemical analysis was collected at one station in Liberty Island Anchorage. Site water was also collected from the Mud Dump Site for chemical analysis and as dilution water in water-column toxicity tests. Water samples were collected using a clean, epoxy-coated steel bucket below the surface of the water. Water was then transferred to precleaned, 20-L polypropylene carboys. (Prior to the sampling survey, carboys were washed with hot water and detergent, acid-rinsed with dilute hydrochloric acid, then rinsed with distilled water, followed by acetone and methylene chloride). The carboys were rinsed with site water three times before filling. Water samples were labeled and stored at ambient temperature (in the shade) while on board the ship.

A log book was maintained containing records of each sample collected, including station designation, coordinates, replicate number, date, sampling time, water depth, core length, and number of core sections per core. Sample identification numbers were logged on chain-of-custody forms daily.

At the conclusion of the sample collection survey, sediment cores and water samples were shipped by refrigerated van thermostatically controlled to maintain approximately 4°C from Caven Point, New Jersey, to the Battelle Marine Sciences Laboratory (MSL) in Sequim, Washington. The shipment departed from Caven Point on June 10, 1994, and arrived at the MSL on June 14, 1994.

2.1.2 Reference and Control Sediment Sampling

Reference sediment for toxicity and bioaccumulation tests was collected from the Mud Dump Reference Site by USACE personnel aboard the M/V *Cleanwaters*, a vessel owned and operated by the EPA, Region II. Four 5-gal containers of surficial sediment were collected using a van Veen grab sampler. After recovery, the sediments were transferred to epoxycoated steel buckets. The buckets were covered, labeled, and stored at ambient temperature (in the shade) while aboard the ship, then were transferred to refrigerated storage at Caven Point at the end of the sampling day.

Records of reference sediment collection included coordinates, replicate number, date, sampling time, and water depth. At the conclusion of the sampling day, reference sediment samples were loaded into the refrigerated van at the staging area, and sample identification numbers were logged on chain-of-custody forms.

Control sediments were used in each toxicity and bioaccumulation test to validate test procedures. Control sediment used in *M. nasuta* and *M. bahia* tests was collected from Sequim Bay, Washington, using a Van Veen sampler deployed from a MSL research vessel. Native sediment for *A. abdita* and *N. virens* were supplied with the test organisms by their respective suppliers.

2.2 Test Organism Collection

Seven species of test organisms were used to evaluate sediment samples from the Liberty Island Anchorage project area:

- Ampelisca abdita, a tube-dwelling, surface detrital-feeding amphipod
- Mysidopsis bahia, a juvenile mysid shrimp
- Menidia beryllina, a juvenile silverside fish
- Mytilus galloprovincialis, the larval zooplankton stage of the mussel
- · Macoma nasuta, the bent-nose clam, a burrowing, surface-detrital feeder
- Tapes japonica, a suspension-feeding clam
- Nereis virens, a burrowing, deposit-feeding polychaete.

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The amphipod A. abdita was supplied by East Coast Amphipod, Kingston, Rhode Island. A. abdita and its native sediment were collected from Narragansett Bay, Rhode Island, by dragging a large dipnet along the sediment surface. Test organisms were carefully removed from their tubes for counting, and then placed in clean, native sediment for overnight transport to the MSL. Mysids were purchased from Aquatic Indicators, St. Augustine, Florida. Mysids that were less than 24-h old were shipped via overnight delivery in plastic bags containing oxygen-supersaturated seawater shipped with gel refrigerant packs. Silversides were also supplied by Aquatic Indicators and were shipped via overnight delivery in plastic bags containing oxygen-supersaturated seawater shipped with gel refrigerant packs. Mussels used for obtaining *M. galloprovincialis* larvae were purchased from the commercial supplier Marinus, Long Beach, California. Mussels were wrapped in moist paper towels and transported in a Styrofoam cooler packed with gel refrigerant packs. Clams (M. nasuta and T. japonica) were collected from intertidal zones in Discovery Bay, Washington, by Johnson and Gunstone. The clams were kept in large containers filled with sediment and seawater obtained from the collection site and transported to the MSL. Worms (*N. virens*) were purchased through Aquatic Research Organisms, and were collected from an intertidal region in Hampton, New Hampshire. The worms were packed in insulated boxes with mats of moist seaweed and shipped at ambient temperature to the MSL via overnight delivery.

All organisms were shipped or transported in native sediment or under conditions designed to ensure their viability. After arrival at the MSL, the test organisms were gradually acclimated to test conditions. Animals with abnormal behavior or appearance were not used in toxicological tests. All acclimation and animal care records are part of the raw data files for these projects.

2.3 Sediment Sample Preparation

Sediment samples for physical, chemical, and biological analysis were prepared from individual core samples, composites of a number of core samples, reference sediment, and control sediment. All sediment samples were assigned random, unique code numbers to ensure that samples are handled without bias by staff in the biology and chemistry laboratories.

Sediment for biological testing was used within the 6-week holding period specified in the Green Book. During this holding time, the sediment samples were received at the MSL; inventoried against chain-of-custody forms; processed and used for benthic and water-column toxicity tests, elutriate analysis, and bioaccumulation tests; and subsampled for sediment physical/chemical analyses. This section describes procedures followed for equipment preparation, safety considerations, and preparation of sediments for biological testing and chemical analyses.

2.3.1 Laboratory Preparation and Safety Considerations

All glassware, stainless-steel or titanium utensils, Nalgene, Teflon, and other laboratory containers and equipment underwent stringent cleaning procedures to avoid contamination of samples. Glassware (e.g., test containers, aquaria, sediment transfer dishes) was washed with hot water and detergent, rinsed with deionized water, then soaked in a 10% solution of reagent grade nitric acid for a minimum of 4 h and rinsed again with deionized water before it was allowed to air dry. Glassware was then rinsed with methylene chloride and allowed to dry under a fume hood. Polyvinyl chloride (PVC), Nalgene, and Teflon tools were treated in the same manner as glassware. Stainless-steel bowls, spoons, spatulas, and other utensils were washed with hot water and detergent, rinsed with deionized water, and allowed to air dry. They were then solvent-rinsed with methylene chloride and allowed to dry under a fume hood.

Neoprene stoppers and polyethylene sheets or other porous materials were washed with hot water and detergent and rinsed with deionized water. These items were then "seasoned" by continuous soaking in 0.45- μ m filtered seawater for at least 2 days prior to use. Large pieces of laboratory equipment, such as the epoxy-coated sediment mixer, were washed with a dilute solution of detergent, and thoroughly rinsed with tap water followed by deionized water. Equipment used for determining water quality, including the meters for pH, dissolved oxygen (DO), temperature, and salinity, were calibrated according to the manufacturers' specifications and internal MSL standard operating procedures (SOPs).

Because the potential toxicity of the Liberty Island Anchorage sediment was unknown, sediment processing and testing were segregated from other laboratory activities. Specific areas at the MSL were established for sample storage and for core-cutting, sediment mixing, and sediment sieving. Work areas were covered with plastic sheeting to contain any waste

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sediment. Wastewater generated during all operations was retained in 55-gal barrels and periodically pumped through activated charcoal filters and into the MSL's wastewater treatment system. These procedures minimized any potential for cross-contamination of sediment samples.

Laboratory staff members were protected by personal safety equipment such as Tyvek suits, plastic aprons, and rubber gloves. Those who were likely to have the most exposure to the potential volatile compounds in the bulk sediment (i.e., those responsible for opening, homogenizing, and compositing core samples) were also provided with half-mask respirators.

2.3.2 Preparation of Sediment for Benthic Testing and Bulk Sediment Analyses

Each Lexan core liner was opened by cutting the core longitudinally with a saw to expose the sediment. As each sediment core sample was opened, it was examined for physical characteristics (e.g., sediment type and consistency, color, odor). In particular, the presence of any strata in the cores was noted. All core observations were recorded in the sediment preparation log book. The sediment between the mudline and project depth was then transferred from the core liner to a clean, stainless-steel bowl by scooping the sediment from the core liner with a spoon or spatula. The sediment was mixed by hand with stainless-steel utensils until the color and consistency appeared homogenous, creating a sample representative of the individual sampling station. Sieving the test sediment was not necessary because no organisms were present in the sediment samples.

Aliquots of the homogenized sediment were then transferred to the appropriate sample jar(s) for physical or chemical analyses required on individual core samples. A portion of each homogenized core sample was also retained as an archive sample. The remainder of the homogenized sediment from the individual core stations was combined to create a composite sample representing the entire Liberty Island Anchorage project area, designated COMP LI. The composite sediment was homogenized in an epoxy-coated mixer. Aliquots of homogenized composite sediment were transferred to the appropriate sample jar(s) for physical or chemical analyses required on the composite sample. A portion of the homogenized composited sediment was also retained as an archive sample. The remainder was stored in

labeled epoxy-coated buckets, tightly covered, at 4°C±2°C until use for SPP/elutriate preparation or benthic toxicity and bioaccumulation tests.

The Mud Dump Reference Site sediment, *M. nasuta, T. japonica, and N. virens* native control sediments were also homogenized in the large, epoxy-coated mixer, but prior to mixing, these sediments were pressed through a 1-mm mesh to remove live organisms that might affect the outcome of toxicity tests. After mixing, aliquots for physical and chemical analyses were removed. Native control sediments for *A. abdita* were sieved through a 0.5-mm mesh to remove live organisms and then mixed in stainless-steel bowls. All reference and control sediments were stored at 4°C±2°C until used in benthic toxicity and bioaccumulation tests.

2.3.3 Preparation of Suspended Particulate Phase and Elutriate

Toxicological effects of dredged sediments dissolved and suspended in the watercolumn at an open-water disposal site were simulated in the laboratory by preparation of the SPP. To prepare the SPP, a sediment-water slurry was created and centrifuged at low speed. The centrifugation procedure replaced the 1-h settling procedure described for elutriate preparation in the Green Book. Low speed centrifugation provided a more timely SPP preparation and maintained consistency between projects. The supernatant was decanted and reserved for testing with water-column organisms. The elutriate phase was prepared by centrifuging the SPP at a higher speed and collecting the decanted supernatant. This liquid was analyzed for chemical constituents to identify potential water-soluble contaminants that could remain in the water-column after dredge and disposal operations.

The SPP was prepared by creating a 4:1 (volume:volume) filtered seawater-to-sediment slurry in 1–L glass jars with Teflon-lined lids. The jars were marked at 200 mL and 400 mL and filled to the 200-mL mark with 0.45- μ m-filtered Sequim Bay seawater. Sequim Bay seawater was used in place of dredging site water due to lower salinity of dredging site water and to maintain consistancy in salinity among the dredging projects tested. Homogenized COMP LI sediment was added until the water was displaced to the 400-mL mark. Each jar was then filled to 1 L with filtered seawater, placed on a shaker table, and agitated for 30 min at 120 to 150 cycles/min. The slurry was then transferred to 500-mL Teflon jars, tightly sealed, and centrifuged at approximately 1750 rpm for 10 min, at a relative centrifugal force of approximately 1000 g. Following centrifugation, the supernatant was poured into 4-L glass jars.

The Teflon jars were rinsed after each use and the above process continued until an adequate amount of SPP was produced from each composite. Between SPP preparations, all glass and Teflon containers were cleaned according to procedures described in Section 2.3.1. When all SPP for a treatment was prepared, portions were taken for elutriate preparation. The remaining SPP was either used immediately for biological tests or stored at 4°C±2°C and used within 24 h for testing. The 100% COMP LI SPP was mixed with Mud Dump Site water to yield three dilutions: 0%, 10%, and 50% SPP, for a total of four concentrations.

To prepare elutriate for chemistry analyses, a 1-L aliquot of the SPP was collected in an acid-washed Teflon bottle for trace metals analysis, and three 1-L aliquots were collected in EPA-certified amber glass bottles for analysis of organic compounds. The SPP for metals analysis was transferred to acid-washed polycarbonate centrifuge jars, and the SPP for analysis of organic compounds was transferred to Teflon centrifuge jars. Both were centrifuged at 2000 rpm for 30 min at a relative centrifugal force of approximately 1200 g. The decanted supernatant liquid was the elutriate phase. One liter of elutriate was submitted for triplicate trace metals analysis and three 1-L portions were submitted for triplicate analysis of organic compounds.

2.4 Physical and Chemical Analytical Procedures

Individual sediment cores, composite LI, water, and elutriate samples were analyzed for selected physical and chemical parameters. Table 2.1 is a list of the parameters measured, the method used for each analysis, and the target analytical detection limits. The following sections briefly describe the procedures used for physical and chemical analyses. Procedures followed those required by the Regional Guidance Manual unless otherwise noted.

Analyte	Methods	Sediment Detection Limit ^(a)	Water Detection Limit
• • • • • • • • • • • • • • • • • • • •			
PHYSICAL PARAMETERS		4.004	
Grain Size	Plumb (1981)	1.0%	
NA(a) Bulk Density	EM 1110-2-1906 (USACE 197	70)	N1A
Percent Moisture	Sediment: Plumb (1981)	1.0 %	NA NA
TOC	EPA (1986)	0.1%	NA
<u>METALS</u>			
Arsenic	EPA 200.2,3,8,9 ^(c)	0.1 μg/g	
Cadmium	EPA 200.2,3,8,9 ^(c)	0.01 μg/g	0.025 μg/L
Chromium	EPA 200.2,3,8,9 ^(c)	0.02 μg/g	0.020 μg/L 1.0 μg/L
Copper	EPA 200.2,3,8,9 ^(c)	0.02 μg/g 0.1 μg/g	0.35 μg/L
Lead	EPA 200.2,3,8,9 ^(c)	0.1 μg/g	0.35 μg/L
Mercury	EPA 245.5 (sed.)	0.02 μg/g	0.002 μg/L
Nickel	EPA 200.2,3,8,9 ^(c)	0.1 μg/g	0.30 μg/L
Silver	EPA 200.2,3,8,9 ^(c)	0.1 μg/g	0.25 μg/L
Zinc	EPA 200.2,3,8,9 ^(c)	0.1 μg/g	0.15 μg/L
PESTICIDES			
Aldrin	EPA 8080 (sediment)	1.0 ng/g	
	EPA 608 (water) ^(c)		0.004 μg/L
α-Chlordane	EPA 8080 (sediment)	1.0 ng/g	
trans-Nonachlor	EPA 608 (water) ^(c) EPA 8080 (sediment)	10	0.014 μg/L
	EPA 608 (water) ^(c)	1.0 ng/g	0.014
Dieldrin	EPA 8080 (sediment)	1.0 ng/g	0.014 μg/L
	EPA 608 (water) ^(c)	1.0 119/9	0.002 μg/L
4,4'-DDT	EPA 8080 (sediment)	1.0 ng/g	0.002 µg/c
	EPA 608 (water) (c)	00	0.012 μg/L
2,4'-DDT	EPA 8080 (sediment)	1.0 ng/g	
	EPA 608 (water) ^(c)		0.020 μg/L
4,4'-DDD	EPA 8080 (sediment)	1.0 ng/g	
	EPA 608 (water) ^(c)		0.011 μg/L
2,4'-DDD	EPA 8080 (sediment)	1.0 ng/g	
	EPA 608 (water) ^(c)		0.020 μg/L
4,4'-DDE	EPA 8080 (sediment)	1.0 ng/g	
	EPA 608 (water) ^(c)	1.0	0.004 μg/L
2,4'-DDE	EPA 8080 (sediment) EPA 608 (water) ^(c)	1.0 ng/g	0.000
			0.020 μg/L

TABLE 2.1. List of Analytes, Methods, and Target Detection Limits

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TABLE 2.1. (contd)

		Sediment	Water
Analyte	Method(s)	Detection Limit	Detection Limit
<u> </u>	Method(S)		
Endosulfan I	EPA 8080 (sediment)	1.0 ng/g	
	EPA 608 (water) (c)		0.014 μg/L
Endosulfan II	EPA 8080 (sediment) EPA 608 (water) ^(c)	1.0 ng/g	0.004 μg/L
Endosulfan sulfate	EPA 8080 (sediment) EPA 608 (water) ^(c)	1.0 ng/g	0.010 μg/L
Heptachlor	EPA 8080 (sediment) EPA 608 (water) ^(c)	1.0 ng/g	0.003 μg/L
Heptachlor epoxide	EPA 8080 (sediment) EPA 608 (water) ^(c)	1.0 ng/g	0.100 μg/L
<u>PCBs</u>			
CL2(8)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL3(18)	NYSDEC (1992) (c)	1.0 ng/g	0.0005 μg/L
CL3(28)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL4(44)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL4(49)	NYSDEC (1992) (6)	1.0 ng/g	0.0005 μg/L
CL4(52)	NYSDEC (1992) (c)	1.0 ng/g	0.0005 μg/L
CL4(66)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL5(87)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL5(101)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL5(105)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL5(118)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL6(128)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL6(138)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL6(153)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL7(170)	NYSDEC (1992) ⁽⁶⁾	1.0 ng/g	0.0005 μg/L
CL7(180)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL7(183)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL7(184)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL7(187)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL8(195)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL9(206)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L
CL10(209)	NYSDEC (1992) ^(c)	1.0 ng/g	0.0005 μg/L

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TABLE 2.1. (contd)

		Sediment	Water
Analyta		Detection	Detection
<u>Analyte</u>	<u>Method(s)</u>	<u>Limit</u>	<u>Limit</u>
<u>PAHs</u>			
Acenapthene	EPA 8270 ^(c)	10.0 ng/g	NA
Acenaphthylene	EPA 8270 ^(c)	10.0 ng/g	NA
Anthracene	EPA 8270 ^(c)	10.0 ng/g	NA
Fluorene	EPA 8270 ^(c)	10.0 ng/g	NA
Naphthalene	EPA 8270 ^(c)	10.0 ng/g	NA
Phenanthrene	EPA 8270 ^(c)	10.0 ng/g	NA
Benz[a]anthracene	EPA 8270 ^(c)	10.0 ng/g	NA
Benzo[a]pyrene	EPA 8270 ^(c)	10.0 ng/g	NA
Benzo[b]fluoranthene	EPA 8270 ^(c)	10.0 ng/g	NA
Benzo[ghi]perylene	EPA 8270 ^(c)	10.0 ng/g	NA
Benzo[k]fluoranthene	EPA 8270 ^(c)	10.0 ng/g	NA
Chrysene	EPA 8270 ^(c)	10.0 ng/g	NA
Dibenz[a,h]anthracene	EPA 8270 ^(c)	10.0 ng/g	NA
Fluoranthene	EPA 8270 ^(c)	10.0 ng/g	NA
Indeno[1,2,3- <i>cd</i>]pyrene	EPA 8270 ^(c)	10.0 ng/g	NA
Pyrene	EPA 8270 ^(c)	10.0 ng/g	NA
1,4-Dichlorobenzene	EPA 8270 ^(c)	1.0 ng/g	NA
Dioxin/furan			
2,3,7,8-TCDD	EPA 8290	1.0 pg/g	NA
1,2,3,7,8-PCDD	EPA 8920	1.0 pg/g 1.0 pg/g	NA
1,2,3,4,7,8-HxCDD	EPA 8290	2.5 pg/g	NA
1,2,3,6,7,8-HxCDD	EPA 8290	2.5 pg/g 2.5 pg/g	NA
1,2,3,7,8,9-HxCDD	EPA 8290	2.5 pg/g	NA
1,2,3,4,6,7,8-HpCDD	EPA 8290	2.5 pg/g 2.5 pg/g	NA
OCDD	EPA 8290	5.0 pg/g	NA
2,3,7,8-TCDF	EPA 8290	1.0 pg/g	NA
2,3,4,7,8-PCDF	EPA 8920	1.0 pg/g	NA
1,2,3,7,8-PCDF	EPA 8290	1.0 pg/g	NA
1,2,3,4,7,8-HxCDF	EPA 8290	2.5 pg/g	NA
1,2,3,6,7,8-HxCDF	EPA 8290	2.5 pg/g	NA
1,2,3,4,7,8-HxCDF	EPA 8290	2.5 pg/g	NA
2,3,4,6,7,8-HxCDF	EPA 8290	2.5 pg/g	NA
1,2,3,4,6,7,8-HpCDF	EPA 8290	2.5 pg/g	NA
1,2,3,4,7,8,9-HpCDF	EPA 8290	2.5 pg/g	NA
OCDF	EPA 8290	5.0 pg/g	NA

(a) Detection limits are in dry weight for all sediment parameters except Hg.
(b) Not applicable.
(c) Equivalent Battelle Ocean Sciences or MSL standard operating procedures were substituted for the methods cited.

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2.4.1 Grain Size and Percentage of Moisture

Grain size was measured following two methods described by Plumb (1981). The wet sieve method was used to determine the size distribution of sand or coarser-grained particles larger than a U.S. No. 230 standard sieve ($62.5-\mu m$ mesh). The size distribution of particles smaller than a U.S. No. 230 sieve was determined using the pipet method. Grain size was reported as percentages within four general size classes:

gravel	>2.0 mm
sand	2.0 - 0.063 mm
silt	0.063 - 0.004 mm
clay	<0.004 mm.

Percentage of moisture was obtained using the Plumb (1981) method for determining total solids. The procedure involves drying a sediment sample at 100°C until a constant weight is obtained. Percentage of moisture was calculated by subtracting the percentage of total solids from 100.

2.4.2 Bulk Density and Specific Gravity

Bulk density, or unit weight, was determined according to EM 111-2-1906 (USACE 1970).

2.4.3 TOC

Samples were analyzed for TOC according to the EPA Edison, New Jersey, Laboratory Procedure (EPA 1986). Inorganic carbon was removed from the sediment sample by acidification. The sample was combusted and the evolved carbon dioxide was quantitated using a carbon-hydrogen-nitrogen (CHN) analyzer. TOC was reported as a percentage of the dry weight of the unacidified sample.

2.4.4 Metals

Sediment samples for analysis of As, Cd, Cr, Cu, Pb, Ni, and Zn were prepared according to an MSL SOP equivalent to EPA Method 200.2 (EPA 1991). Solid samples were first freeze-dried and blended in a Spex mixer mill. A 0.2- to 0.5-g aliquot of dried homogeneous sample was then digested using peroxide and nitric acid. Samples were heated in sealed Teflon bombs overnight at approximately 130°C. Sediment samples were analyzed

for As, Cd, Cr, Cu, Pb, Ni, and Zn using inductively coupled plasma/mass spectrometry (ICP/MS), following an MSL SOP based on EPA Method 200.8 (EPA 1991). Sediment samples were analyzed for Ag by graphite furnace atomic absorption (GFAA) according to an MSL SOP based on EPA Method 200.9 (EPA 1991). Sediments were analyzed for Hg by cold vapor atomic absorption (CVAA) according to an MSL procedure for total Hg determination equivalent to EPA Method 245.5 (EPA 1991).

Preparation and analysis of water samples for As, Cd, Cr, Cu, Pb, Ni, Ag, and Zn were conducted according to MSL SOPs equivalent to EPA Methods 200.2 and 200.9 (EPA 1991). Samples were chelated with 2% ammonium pyrrolidinedithiocarbamate (APDC), precipitated out of solution, and filtered. The filter was digested in concentrated nitric acid and the digestate was analyzed by GFAA spectroscopy for Cr and Zn, or by ICP/MS for Cd, Cu, Pb, Ni, and Ag. Water samples were analyzed for Hg directly by CVAA according to the method of Bloom and Crecelius (1983). This CVAF technique is based on emission of 254-nm radiation by excited elemental Hg atoms in an inert gas stream. Mercuric ions in an oxidized sample were reduced to elemental Hg with tin chloride (SnCl₂), then purged onto gold-coated sand traps to preconcentrate the Hg and remove interferences. Mercury vapor was thermally desorbed to a second "analytical" gold trap, and from that into the fluorescence cell. Fluorescence (indicated by peak area) is proportional to the quantity of Hg collected, and was quantified using a standard curve as a function of the quantity of the sample purged.

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2.4.5 Chlorinated Pesticides and PCBs

Sediment samples for pesticide and PCB analysis were extracted and analyzed according to an MSL procedure similar to EPA Method 8080 for pesticides and the New York State Department of Environmental Conservation (NYSDEC) Congener-Specific Method 91-11 (NYSDEC 1992). The method also uses techniques from the National Oceanic and Atmospheric Administration (NOAA 1993). A 20- to 50-g sample of homogenized sediment was first combined with sodium sulfate in a sample jar to remove water. Samples were extracted by adding successive portions of methylene chloride and agitating sample jars at ambient temperature using a roller technique. Extract volumes were reduced and solvent-exchanged to

hexane, followed by Florisil column chromatography cleanup. Interferences were removed using HPLC cleanup. Sample extracts were concentrated and analyzed using gas chromatography with electron capture detection (GC-ECD) by the internal standard technique.

The concentration of total PCB in each matrix was estimated by taking the sum of the 22 congeners (x) and using the following equation based on NOAA (1989) noted in the Regional Guidance Manual (USACE-NYD/EPA Region II 1992): Total PCB = 2.19(x) + 2.19.

Water samples were prepared and analyzed for chlorinated pesticides and PCBs according to an MSL procedure equivalent to EPA Method 8080 (EPA 1990), and incorporating techniques developed by the National Status and Trends "Mussel Watch" Program (NOAA 1993). Samples were extracted with methylene chloride. Extract volumes were reduced and solvent exchanged to hexane. The sample extracts underwent cleanup by alumina and silica column chromatography; further interferences were removed by an additional cleanup treatment using high-performance liquid chromatography (HPLC). Sample extracts were concentrated and analyzed using GC-ECD by the internal standard technique.

2.4.6 PAHs and 1,4-Dichlorobenzene

Sediment samples were prepared for the analysis of 16 PAHs and 1,4-dichlorobenzene (see Table 2.1) according to an MSL method based on the NOAA Mussel Watch procedure (NOAA 1993). A 20- to 50-g sample of homogenized sediment was first combined with sodium sulfate in a sample jar to remove water. Samples were extracted by adding successive portions of methylene chloride and agitating sample jars at ambient temperature using a shaker technique. Extract volumes were reduced and solvent-exchanged to hexane, followed by column chromatography cleanup. Interferences were removed using HPLC cleanup (Krahn et al. 1988).

Sample extracts were concentrated and analyzed using gas chromatography with mass spectrometry (GC/MS) in the selective ion monitoring (SIM) mode.

2.4.7 Dioxins and Furans

All dioxin/furan analyses followed EPA Method 8290 (EPA 1990) with modifications taken from EPA Method 1613. A 5- to 10-g aliquot of sediment was spiked with nine isotopically labeled PCDD/PCDF compounds. Sediment samples underwent a benzene

Soxhlet extraction for about 18 h. To evaluate recovery through cleanup procedures, 2,3,7,8-TCDD³⁷Cl₄ was added to the extracts prior to any cleanup steps. The sample extracts were subjected to an acid/base washing and dried with sodium sulfate and then processed through acid/base silica, alumina, and carbon AX-21/celite columns. After a concentration step, the extracts were spiked with internal standards: ¹³C₁₂-1,2,3,4,8,9-HxCDD for quantitation of hexa-, hepta, and octachlorinated dioxin and furan congeners. Sample extracts were analyzed by high-resolution gas chromatography/high-resolution mass spectroscopy (HRGC/HRMS) in the SIM mode on a DB-5 column.

2.5 Biological Testing Procedures

2.5.1 Water-Column Toxicity Tests

Water-column effects of open-water dredged-material disposal were evaluated by exposing three species of water-column organisms to the SPP of the Liberty Island Anchorage sediment composite. The three test species were juvenile *M. beryllina* (silverside), juvenile *M. bahia* (mysid), and larval *M. galloprovincialis* (mussel).

2.5.1.1 Water-Column Toxicity Test with Menidia beryllina

Upon receipt, the *M. beryllina* were placed in a 10-gal glass aquarium and gradually acclimated from 27.5‰ seawater to 30.0‰ Sequim Bay seawater over a 24-h period. *M. beryllina* were received and held at 20°C±2°C prior to testing and were fed concentrated brine shrimp nauplii daily. During acclimation and holding, 2% to 3% mortality of the silversides was observed.

Test containers for the water-column toxicity test with silversides were 500-mL glass jars, labeled with sediment treatment code, concentration, position number, and replicate number. Five replicates of each concentration were tested. The 300-mL test volume of SPP was placed in each of the five replicate test chambers. Each test chamber was then placed in a randomly assigned position on a water table at 20°C±2°C and allowed to equilibrate to test temperature for several hours. After the concentrations were prepared and placed on the water

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table, water quality parameters were measured and recorded for all replicates of all concentrations for each sediment treatment.

To initiate the test, *M. beryllina* were transferred from the holding tank to test chambers with a wide-bore pipet via small transfer cups. Ten individuals were introduced to each test chamber, creating a test population of 50 silversides per concentration for each treatment. Ten animals per test chamber were used, rather than the 20 animals per chamber as described in the Regional Guidance Manual, because it is not possible to make accurate daily observations of *M. beryllina* behavior when using 20 animals. Test initiation time and date were recorded. Following test initiation, water quality parameters were recorded in one replicate of each concentration daily. Because several treatments had DO levels lower than 40% saturation prior to test initiation, all test chambers were aerated to maintain consistency in handling DO concentration among test containers. Acceptable parameters for this test were as follows:

Temperature	20°C±2°C
DO	>40% saturation (>3.0 mg/L at 20°C, 30‰)
pН	7.8±0.5
Salinity	30.0‰±2.0‰.

The test was run under a 16-h light/8-h dark photoperiod, and silversides were fed brine shrimp nauplii daily during the test. Observations of the animals were performed at 2 h, 24 h, 48 h, and 72 h, and the number of live, dead, and missing organisms was recorded. At the end of the 96-h test period, water quality parameters were measured for all test chambers, and the number of live, dead, and missing silversides was recorded on termination forms. As a quality control check, a second observer confirmed surviving test organisms on at least 10% of the termination counts.

A 96-h, water-only, reference toxicant test was performed concurrently with the toxicity test with each population of *M. beryllina* to establish the health and expected response of the test organisms. The reference toxicant test was conducted in the same manner as the water-column toxicity test. *M. beryllina* were exposed to a seawater control plus four concentrations of copper sulfate: 16, 64, 160, and 400 μ g/L Cu, using three replicates of each concentration.

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2.5.1.2 Water-Column Toxicity Test with Mysidopsis bahia

Upon receipt, the *M. bahia* were placed in a 10-gal aquarium and gradually acclimated from 28.0‰ seawater to 30‰ Sequim Bay seawater over a 24-h period. Mysids were received and held at 20°C±2°C until testing and were fed concentrated brine shrimp nauplii twice daily prior to testing. Mortality of the *M. bahia* during holding was less than 1%.

The water-column toxicity test with the mysid was performed in 200 mL of test solution in 400-mL jars, labeled with sediment treatment code, concentration, position number, and replicate number. Five replicates of each concentration were tested. Each of the test chambers received 200 mL of test solution, then was placed randomly in a recirculating water bath and allowed to equilibrate to test temperature for several hours. Prior to test initiation, water quality parameters were measured in each replicate of each sediment treatment concentration. Acceptable water quality parameters for this test were as follows:

ration (>3.0 mg/L at 20°C, 30‰)
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To initiate the test, *M. bahia* were transferred from the holding tank to test chambers with a wide-bore pipet via small transfer cups. Ten individuals were introduced to each test chamber, creating a test population of 50 mysids per concentration (200 mysids per treatment). Ten animals per test chamber were used, rather than the 20 animals per chamber as described in the Regional Guidance Manual, because it is not possible to make accurate daily observations of *M. bahia* behavior when using 20 animals. Test initiation time and date were documented on data forms. Observations of test organisms were performed at 4 h, 24 h, 48 h, and 72 h, using a fluorescent light table to enhance visibility of the *M. bahia*. After test initiation, water quality parameters were measured daily in one replicate of each concentration for each sediment treatment. During the 96-h exposure, *M. bahia* were fed <24-h-old brine shrimp daily. Excess food was removed daily with a small pipet, taking care not to disturb test animals.

Prior to test termination, water quality parameters were measured in all replicates. At 96 h, the number of live versus dead animals was recorded for each test container. An animal

was considered dead if it did not respond to gentle probing. As a quality control check, a second observer confirmed surviving test organisms on at least 10% of the termination counts.

A 96-h, water-only, reference toxicant test was performed concurrently with the toxicity test with each batch of *M. bahia* to establish the health and expected response of the test organisms. The reference toxicant test was conducted in the same manner as the water-column toxicity test. *M. bahia* were exposed to a seawater control plus four concentrations of copper sulfate: 100, 150, 200, and 300 μ g/L Cu, using two replicates of each concentration.

2.5.1.3 Water-Column Toxicity Test with Mytilus galloprovincialis Larvae

Prior to testing, adult *M. galloprovincialis* were held in flowing, unfiltered Sequim Bay seawater at ambient temperatures for approximately 5 days. Chambers for the bivalve larvae test were 500-mL glass jars labeled with sediment treatment code, concentration, position number, and replicate number. Dilutions of COMP LI SPP (0%, 10%, 50%, and 100%) were prepared with Mud Dump Site water in a 2000-mL graduated cylinder, then 300 mL of test solution was transferred into each test chamber. Test chambers were placed in random positions on a water table and allowed to equilibrate to test temperature for several hours. Initial water quality parameters were measured in all replicates once test chambers reached testing temperatures (16°C±2°C).

Spawning was induced by placing *M. galloprovincialis* into 15°C, filtered Sequim Bay seawater and rapidly raising the holding water temperature to 20°C. Spawning generally occurs within 1 h of temperature elevation; however, on the first day of spawning, gametes were shed after 3 h to 4 h. For this group of mussels, the water bath was changed when DO levels fell below 3.0 mg/L. When spawning began, males and females were identified and isolated in individual jars containing filtered Sequim Bay seawater and allowed to shed gametes for approximately 45 min. Eggs from each female were filtered through a 75- μ m Nytex screen into separate jars to remove feces, detritus, and byssal fibers. Sperm from at least three males were pooled and 10 mL of sperm solution was then added to each of the egg stocks. Egg-sperm solutions were gently mixed every 10 min with a perforated plunger. Fertilization proceeded for 1 h, then fertilization rate (percentage of fertilized eggs) was determined by removing a subsample and observing the number of multicell-stage embryos. Fertilization was considered successful if greater than 90% of the embryos were in the multicell stage. Egg

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stocks with greater than 90% fertilization were combined and rinsed on a 20-µm Nytex screen to remove excess sperm. Stock embryo solution density was estimated by removing a 0.1-mL subsample and counting all multicell embryos, then multiplying by 10 to yield embryo density (embryos/mL). Stock solution was diluted or concentrated to yield 7500 to 9000 embryos/mL. The test was initiated by introducing 1 mL of stock solution into each test chamber, to produce embryo densities of 25 to 30 embryos/mL. Test initiation date and time were recorded on data sheets. Following initiation, 10-mL stocking-density subsamples were removed from each container and preserved in 5% formaldehyde to determine actual stocking density later.

Water quality parameters were measured in one replicate of each concentration per treatment daily throughout the test. Acceptable ranges for water quality parameters were as follows:

Temperature	16°C±2°C
DO	>60% saturation (>4.9 mg/L at 16°C, 30‰)
pН	7.8±0.5
Salinity	30.0‰±3.0‰.

Because several treatments had DO levels below the acceptable level of 40% saturation, each chamber was provided with gentle aeration to maintain consistency in handling DO concentration among test containers. The bivalve test was terminated after 72 h when greater than 80% of the larvae in the controls had reached the D-cell stage. Final water quality parameters were recorded for all replicates. The contents of each chamber were then homogenized with a perforated plunger, and a 10-mL subsample was removed and placed into a 20-mL scintillation vial. The subsample was then fixed with 1 mL of 50% solution of formaldehyde in seawater. Samples were scored for the appearance of normal and abnormal D-shaped larvae, blastula larvae, and total number of larvae. At least 10% of the counts were confirmed by a second observer.

A 72-h reference toxicant test was conducted to establish the health and expected response of the test organisms. The reference toxicant test was set up and conducted in the same manner as the liquid-phase tests. *M. galloprovincialis* larvae were exposed to a filtered Sequim Bay seawater control plus copper sulfate concentrations of 1, 4, 16, and 64 μ g/L copper, with three replicates per concentration.

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2.5.2 Benthic Acute Toxicity Tests

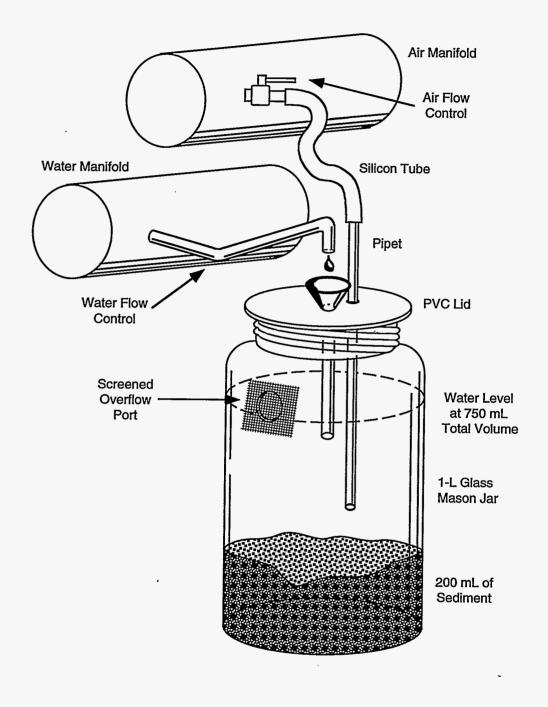
Acute toxicity of COMP LI sediments was evaluated using the amphipod *A. abdita* and the mysid *M. bahia*.

2.5.2.1 Static Renewal Test with Ampelisca abdita

Upon receipt, the *A. abdita* were placed in a tub of clean sand from their collection area and gradually acclimated with flowing Sequim Bay seawater from 28‰ to 30.5‰ salinity, over a period of 2 days. *A. abdita* were received at approximately 11°C and acclimated to 20°C±2°C over 4 days. They were held at 20°C±2°C for one day and were not fed prior to testing.

The amphipod test was performed in 1-L glass jars modified for use as static-renewal test chambers. The test chambers were fitted with funneled lids and screened outflow and overflow ports (Figure 2.1). The flow-through system was calibrated prior to test initiation and thereafter turned on periodically, long enough to deliver the seawater at a rate of two chamber exchanges per day. Five replicates of COMP LI, Mud Dump Reference Site, and native test animal control treatments were tested.

Concentrations of ammonia have been encountered in the pore water of sediment core samples from New York/New Jersey waterways at concentrations high enough to affect survival of amphipods in benthic toxicity tests (Barrows et al. 1996). Therefore, the amphipod test was conducted according to the ammonia protocols issued by EPA and the USACE (EPA/USACE 1993). This guidance requires postponing test initiation (exposure of test animals) until pore water total ammonia concentrations are <30 mg/L for *A. abdita*. During this "purging" period, test chambers were set up and maintained under test conditions, and the overlying water was exchanged twice daily until the pore water ammonia concentrations reached the level appropriate for the particular amphipod. Pore water ammonia measurements were made on "dummy" containers that were set up and maintained in the same manner as the actual test containers but without animals added to them. The pore water was obtained by siphoning off the overlying water in the dummy jar and centrifuging the sediment in a Teflon jar for at least 20 min at approximately 3000 rpm. Salinity, temperature, and pH were also determined in the pore water samples.





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The amphipod toxicity test was initiated by the addition of 20 organisms to each test chamber for a test population of 100 amphipods per sediment treatment. Amphipods were gently sieved from their native sediment in holding tanks and transferred to shallow baking dishes. For each test chamber, five animals were counted and transferred by pipet into each of four small, plastic cups. The animals in each transfer cup were recounted by a second observer. The animals were placed in the test chamber by dipping the cup below the surface of the water to release the amphipods.

Salinity, temperature, DO, and pH were measured in all replicates prior to test initiation, in at least one replicate per treatment daily, and in all replicates at test termination. Measurements of total ammonia levels in the overlying water and pore water also continued during testing. Overlying water ammonia was measured in all replicates prior to test initiation (Day 0), in at least one replicate per treatment daily, and in all replicates at test termination (Day 10). Pore water ammonia was measured on Day 0 and Day 10. The following were the acceptable ranges for water quality parameters during the amphipod tests:

20°C±2°C
>60% saturation(4.6 mg/L@20°C, 30‰)
7.8±0.5
30‰±2‰
≤30 mg/L
2 exchanges/day.

Gentle aeration was provided throughout the test, and the amphipods were not fed during testing. At the end of the 10-day period, the contents of each chamber were gently sieved through 0.5-mm mesh, and the number of live, dead, and missing amphipods was recorded on termination forms. An animal was considered dead if it did not respond to gentle probing. As a quality control check, a second observer confirmed surviving test organisms on at least 10% of the termination counts.

Reference toxicant tests with cadmium chloride were performed concurrently with testing. The reference toxicant tests were 96-h, water-only exposures that were otherwise conducted following the same procedures as for the static-renewal tests with sediment. *A. abdita* were exposed to nominal concentrations of 0, 0.19, 0.38, 0.75, and 1.5 mg/L Cd.

2.5.2.2 Static Renewal Test With Mysidopsis bahia

Upon receipt at the laboratory, *M. bahia* were placed in 10-gal aquaria and gradually acclimated from 28‰ seawater to 30‰ with Sequim Bay seawater over a 24-h period. Mysids were received and held for 4 days at 20°C±2°C until testing and were fed concentrated brine shrimp nauplii twice daily prior to testing. Mortality of the *M. bahia* during holding was less than 1%.

The 10-day static-renewal benthic acute toxicity test with *M. bahia* was performed in 1-L glass jars. The test chambers were the same ones previously described for the amphipod test. The flow-through system was turned on periodically, long enough to deliver the seawater at a rate of two chamber exchanges per day. To prepare each test container, 200 mL of clean seawater was placed in each jar. Sediment was added until water was displaced up to the 400-mL mark, then seawater was added up to the 750-mL mark. Five replicates of COMP LI sediment, Mud Dump Reference Site sediment, and native test animal control sediment were tested.

Concentrations of ammonia have been encountered in the pore water of sediment core samples from New York/New Jersey waterways at concentrations high enough to affect survival of amphipods in benthic toxicity tests (Barrows et al. 1996). Therefore, the mysid test was conducted according to the ammonia protocols issued by EPA and the USACE (EPA/USACE 1993). This guidance requires postponing test initiation (exposure of test animals) until pore water total ammonia concentrations are < 20 mg/L for *M. bahia*. During this "purging" period, test chambers were set up and maintained under test conditions, and the overlying water was exchanged twice daily until the pore water ammonia concentrations reached the level appropriate for the particular amphipod. Pore water ammonia measurements were made on "dummy" containers that were set up and maintained in the same manner as the actual test containers but without animals added to them. The pore water was obtained by siphoning off the overlying water in the dummy jar and centrifuging the sediment in a Teflon jar for at least 20 min at approximately 3000 rpm. Salinity, temperature, and pH were also determined in the pore water samples.

The mysid benthic toxicity test was initiated by the addition of 20 organisms to each test chamber for a test population of 100 mysids per sediment treatment. Mysids were transferred

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from holding tanks to shallow glass dishes. For each test chamber, five animals were counted and transferred by pipet into each of four small, plastic cups. The animals in each transfer cup were recounted by a second analyst. The animals were placed in the test chamber by dipping the cup below the surface of the water to release the mysids.

Salinity, temperature, DO, pH, and total ammonia in overlying water were measured in all replicates prior to test initiation, in at least one replicate per treatment daily, and in all replicates at test termination. The following were the acceptable ranges for water quality parameters during the *M. bahia* benthic test:

Temperature	20°C±2°C
DO	>40% saturation(3.0 mg/L @ 20°C, 30‰)
рН	7.8±0.5
Salinity	30‰±2‰
Ammonia	<20 mg/L
Renewal rate	2 exchanges/day.

Gentle aeration was provided to all test chambers during the test to maintain consistency in handling DO concentration among test containers. At the end of the 10-day period, the contents of each chamber were gently sieved through 0.5-mm mesh, and the number of live and dead or missing mysids was recorded on termination forms. An animal was considered dead if it did not respond to gentle prodding. As a quality control check, a second observer confirmed surviving test organisms on at least 10% of the termination counts.

Two different mysid populations were used for the benthic test and the water-column test, a 96-h, water-only reference toxicant test with copper sulfate (0, 50, 100, 150, 200, and $300 \ \mu g/L$ Cu) was performed concurrently with each test.

2.5.3 Bioaccumulation Testing

The polychaete *N. virens* and the bivalves *M. nasuta* and *T. japonica* were used to evaluate the potential bioaccumulation of contaminants from dredged material. The bioaccumulation tests were 28-day flow-through exposures to sediment followed by a 24-h depuration period that allowed the organisms to void their digestive tracts of sediment. *N. virens*, *M. nasuta*, and *T. japonica* were tested in separate 10-gal flow-through aquaria because *N. virens* are natural predators of bivalves. Animals were exposed to five replicates of COMP LI, Mud Dump Reference Site sediment, and native control sediment. Each chamber contained

25 test organisms. Water quality parameters (temperature, DO, pH, and salinity) were measured in all replicates at test initiation, in at least one replicate per treatment daily, and in all replicates at test termination. Flow rates were measured daily in all chambers.

	<u>M. nasuta / T. japonica</u>	<u>N. virens</u>
Temperature	14°C±2°C	20°C±2°C
DO	> 60% saturation	> 60% saturation
pН	7.8±0.5	7.8±0.5
Salinity	30‰±2‰	30‰±2‰
Flow Rate	125±10 mL/min	125±10 mL/min.

Aeration was provided to all test chambers to maintain consistency in handling DO concentrations among test chambers. Water quality, organism behavior (e.g., burrowing activity, feeding) and organism mortality were recorded daily. Dead organisms were removed daily. At the end of the 28-day testing period, *M. nasuta, T. japonica* and *N. virens* were placed in clean, flowing seawater for 24 h, after which the tissues were transferred into the appropriate chemistry jars, frozen immediately and stored for archive purposes at <-20°C.

Water-only reference toxicant tests (96-h) were also performed using copper sulfate in six geometrically increasing concentrations. The exposures were conducted using a test volume of 5 L in static 9.5-L (2.5-gal) aquaria. Three replicates of each concentration were tested, each containing 10 organisms. Water quality parameters were monitored at the same frequency and maintained within the same limits as the 28-day test, except that there were no flow rates. The *M. nasuta/T. japonica* reference toxicant tests were conducted with treatments of 0, 0.25, 0.50, 0.75, 1.0, 1.5 and 2.0 mg/L Cu; the *N. virens* test was conducted with treatments of 0, 0.05, 0.075, 0.15, 0.20, 0.25, and 0.30 mg/L Cu.

2.6 Data Analysis and Interpretation Procedures

Statistical analyses were conducted to determine the magnitude and significance of toxicity in test treatments relative to the reference treatment. Each statistical test was based on a completely random design that allowed unbiased comparison between treatments.

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2.6.1 Randomization

All water-column and benthic toxicity tests were designed as completely random tests. Organisms were randomly allocated to treatments, and treatments were randomly positioned on water tables. To determine randomization, a random-number table was generated for each test using the discrete random-number generator in Microsoft *Excel* spreadsheet software.

2.6.2 Statistical Analysis of Water-Column Tests

Two statistical tests are presented in the Green Book for the interpretation of SPP (water-column) tests. The first is a one-sided t-test between survival in control test replicates and survival in the 100% SPP test replicates. This test is to be performed only when survival in the 100% SPP is less than the control (0% SPP) survival, and when control survival is >90% for nonlarval tests and >70% for larval tests (including test validity). Prior to conducting the t-test, angular transformation (arcsine of the square root) of the proportion survival in test replicates was performed to reduce possible heterogeneity of variance between mean survival of test organisms in the control and in the 100% SPP. The second test required by the Green Book is an LC_{50} or EC_{50} calculation, the concentration of SPP that is lethal to (LC_{50}) or affects (EC_{50}) 50% of the organisms tested. The LC_{50} or EC_{50} values for these tests were calculated using the trimmed Spearman-Karber method (Finney 1971). The Spearman-Karber estimator is appropriate only if there was increasing mortality (or effect) with increasing concentration, and if \geq 50% mortality (or effect) did not occur in the 100% SPP concentrations for any treatments, then LC_{50} or EC_{50} values were reported as >100% SPP.

2.6.3 Statistical Analysis of Benthic Toxicity Tests

Benthic toxicity of all sediment treatments was compared by analysis of variance. (ANOVA) on the arcsine square root of the proportion of organisms surviving the test. The arcsine square root transformation stabilizes the within-class variances to help meet assumptions of the ANOVA. The Green Book recommends Dunnett's test (Dunnett 1964) for comparing test treatments with a single reference treatment. All treatments were compared using Dunnett's test for comparison of all test treatments with the reference site using an experiment-wise error of alpha = 0.05.

2.7 Quality Assurance/Quality Control Procedures

The quality assurance/quality control (QA/QC) procedures for the Liberty Island Anchorage project were consistent with the Regional Guidance Manual and the Green Book, and were documented in the Work/Quality Assurance Project Plan, *Evaluation of Dredged Material Proposed for Ocean Disposal from Federal Projects at Liberty Island Anchorage and MOTBY*, New York prepared by the MSL and submitted to the USACE-NYD for this program. This document describes all QA/QC procedures that were followed for sample collection, sample tracking and storage, and physical/chemical analyses. A member of Pacific Northwest National Laboratory's (PNNL) quality engineering staff was present throughout all phases of this program to observe procedures, review and audit data, and ensure that accepted protocols were followed. Laboratory notebooks or data accumulation notebooks were assigned to each portion of these studies and served as records of day-to-day project activities.

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3.0 Results

This section presents results of sample collection and processing, physical and chemical analyses, and biological testing were conducted on sediment samples collected from the proposed Liberty Island Anchorage dredging area.

3.1 Sample Collection and Processing

Sediment core samples were collected from the Liberty Island Anchorage project area on June 7 through June 10, 1994. Table 3.1 lists each sampling station within the Liberty Island Anchorage project area, collection date, sampling coordinates, length of core required for testing, and length of core actually collected. Liberty Island Anchorage sediments were collected aboard the M/V *Hayward* (first 4 samples) and the M/V *Driftmaster* (remaining samples). Nine of 10 core samples were collected to project depth, plus 1 ft of overdepth, one was collected to project depth.

Upon delivery of the sediment core samples to the MSL on June 14, 1994, samples were prepared for the physical and chemical analyses according to the procedures described in Section 2. Individual sediment core samples were analyzed for grain size, moisture content, and TOC. One composited sediment sample representing the entire Liberty Island Anchorage project area (COMP LI) was analyzed for bulk density, metals, chlorinated pesticides, PCBs, PAHs, 1,4-dichlorobenzene, and dioxin/furan congeners.

3.2 Physical and Chemical Analyses

3.2.1 Sediment Core Sample Description

Table 3.2 lists physical characteristics of each intact sediment core sample that was examined.

Station	Collection	<u>Station</u>	Coordinates Longitude W	Core Length Required (ft)	Core Length Collected (ft)	Depth _ <u>(ft)</u> _
			<u>Longitudo II</u>	<u>Inequired (inj</u>	<u>Concered (it)</u>	<u></u>
LI-1	6/7/94	40°41.40	74°02.87	8.9	9.7	10.1
LI-2	6/10/94	40°41.34	74.02.88	3.0	2.5	16.0
LI-3	6/7/94	40°41.30	74°02.84	11.2	11.0	8.8
LI-4	6/7/94	40°41.40	74°02.88	6.0	8.0	13.0
L1-5	6/7/94	40°41.30	74°02.85	5.5	8.0	13.5
LI-6	6/7/94	40°41.36	74°02.93	4.4	7.0	14.5
LI-7	6/7/94	40°41.33	74°02.88	4.0	10.0	15.0
LI-8	6/7/94	40°41.28	74°02.89	4.7	9.3	14.3
LI-9	6/10/94	40°41.26	74°02.89	6.0	6.5	13.0
LI-10	6/10/94	40°40.38	74°02.92	4.0	6.5	15.0

TABLE 3.1. Summary of Sediment Sampling Data for Liberty Island Anchorage

TABLE 3.2. Liberty Island Anchorage Sediment Core Descriptions

<u>Station</u> LI-1	<u>Core Top</u> 10.1	<u>Core Bottom</u> 19.0	Project <u>Depth (ft)</u> 19.0	Description of Observations Black, silty-clayey material with streaks of lighter grey silt. At approximately 15.0 ft, denser black silty-clay material, remaining core (approximately 2 ft) brown sand with rocks and a sulfide odor.
LI-2	16.0	19.0	19.0	Uniform black silty-clayey material, below project depth orange-brown sand with rocks.
LI-3	8.8	19.0	19.0	Uniform black silty-clayey material which becomes more dense towards project depth. At approximately 18 ft, red clay is encountered.
LI-4	13.0	19.0	19.0	Soft black, silty-clayey material. Between 17 ft and 18 ft a band of brown sand with rocks is encountered. The remaining core is black silty-clay material.
LI-5	13.5	19.0	19.0	Very soft black, silty material.
LI-6	14.6	19.0	19.0	Between 14.6 ft and 17 ft Very soft and wet black silty-clay material, oil sheen in standing water. At 17 ft a 4-in. band of gray clay is encountered followed by a 3-in. band of gray sand, and a 10-in. band of gray clay. The remaining core is gray sand.
LI-7	15.0	19.0	19.0	Uniform black silty-clayey material.
LI-8	14.3	19.0	19.0	Top of core is 5-in. of grayish silt. The rest of core is uniform black silty-clayey material becoming more dense towards project depth.
LI-9	13.0	19.0	19.0	Uniform black silty-clayey material that is loose and wet. Bottom 6-in. is gray clay with shell hash.
LI-10	15.0	19.0	19.0	Uniform black silty-clayey material. Bottom 6-in. is lighter silty clayey material with some sand mixed in.

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3.2.2 Grain Size and TOC

Table 3.3 shows the results of the analysis of Liberty Island Anchorage sediment samples for grain size and percentage of moisture. A quality control summary and associated quality control data for grain size and TOC measurements are provided in Appendix A. · · · · · ·

Liberty Island Anchorage sediments were predominantly silt and clay. Percentages of sand ranged from 3% to 42%; silt ranged from 8% to 53%; and clay ranged from 17% to 59%. The moisture content ranged from 42% to 58%. The COMP LI had an average dry bulk density of 39 lbs/ft³. TOC measurements for Liberty Island Anchorage sediments ranged from 1.9% to 4.2%.

	Total Percent (dry weight)					
	Gravel	Sand	Silt	Clay	Percent	Percent
<u>Station</u>	<u>>2.0 mm</u>	<u>2.0 - 0.063 mm</u>	<u>0.063- 0.004 mm</u>	<u><0.004 mm</u>	of TOC	of Moisture
COMP LI	2	22	41	35	3.1	50
						56
LI-1	4	26	38	32	2.6	53
LI-1A ^(a)	13	65	19	3	NA ^(b)	22
L1-2	13	40	30	17	1.9	42
LI-3	0	5	53	42	3.1	58
LI-3A ^(c)	0	5	70	25	NA	33
LI-4	8	25	8	59	3.6	53
LI-5	0	5	53	42	3.9	57
LI-6	1	42	34	23	2.1	47
LI-6A ^(d)	0	97	2	1	NA	20
LI-7	0	7	53	40	3.8	54
LI-8	0	3	52	45	3.9	55
LI-9	0	8	51	41	4.2	53
LI-10	0	22	43	35	3.5	58

<u>TABLE 3.3</u>. Results of Analysis of Liberty Island Anchorage Sediment Samples for Grain Size, Percentage of Moisture, and TOC

(a) Extra grain size sample taken from a 2 ft band of brown sand with rocks.

(b) NA Not applicable, sample not measured for this parameter.

(c) Extra grain size sample taken from a 1 ft band of red clay.

(d) Extra grain size sample taken from a 1 ft band of gray sand.

3.2.3 Metals

Table 3.4 shows the results of the analysis of COMP LI sediment samples for metals. A quality control sample summary and quality control data associated with the metals analysis are provided in Appendix A.

All nine metals were detected in the Liberty Island Anchorage sediment composite. Cr, Cu, Pb, and Zn were detected at concentrations greater than 200 ppm.

3.2.4 Chlorinated Pesticides

Table 3.5 shows the results of the analysis of Liberty Island Anchorage sediment composite for chlorinated pesticides. A quality control sample summary and associated quality control data are provided in Appendix A.

The COMP LI sediment contained detectable concentrations of eight pesticides. The predominant pesticides found in COMP LI were the DDT family of compounds (74.5 μ g/kg total DDTs), followed by aldrin, dieldrin, α -chlordane, and *trans*-nonachlor.

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Analyte	COMP LI (mg/kg dry weight)
Ag	8.57
As	15.6
Cd	4.12
Cr	214
Cu	203
Hg	2.88
Ni	43.0
Pb	228
Zn	258
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TABLE 3.4. Results of Analysis of Liberty Island Anchorage Sediment Samples for Metals

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<u>TABLE 3.5</u>. Results of Analysis of Liberty Island Anchorage Sediment for Chlorinated Pesticides

Analyte	COMP LI (<u>µg/kg dry weight)</u> ^(a)
2,4-DDD	12.4
2,4-DDT	0.22 U ^(b)
4,4-DDD	14.1
4,4-DDE	39.2
4,4-DDT	8.41
Aldrin	9.19
α-Chlordane	4.79
Dieldrin	6.35
Endosulfan I	0.33 U
Endosulfan II	0.33 U
Endosulfan sulfate	0.33 U
Heptachlor	0.06 U
Heptachlor epoxide	0.29 U
trans-Nonachlor	3.17 J ^(c)
Total Estimated DDT ^(d)	98.4
Total Detected DDT	97.6

(a) Mean of replicated sample.

(b) U Undetected at or above the given concentration.

(c) J Analyte detected is below established method detection limit (MDL).

(d) Sum of 2,4-DDD, 2,4-DDE, 2,4-DDT, 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT; one-half of the detection limit used in summation when analyte was undetected.

3.2.5 PCBs

Table 3.6 shows the results of the analysis of the Liberty Island Anchorage sediment for PCBs. A quality control sample summary and associated quality control data are provided in Appendix A.

Twenty-one of the 22 PCB congeners analyzed were detected in COMP LI sediment. The total PCB concentration calculated for COMP LI was 1738 μ g/kg.

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Analyte	COMP LI <u>(µg/kg dry weight)</u> ^(a)
PCB 8	27.9
PCB 18	82.2
PCB 28	131
PCB 44	60.1
PCB 49	67.0
PCB 52	77.1
PCB 66	95.9
PCB 87	13.5
PCB 101	43.8
PCB 105	17.8
PCB 118	44.1
PCB 128	5.89
PCB 138	32.2
PCB 153	33.1
PCB 170	10.1
PCB 180	19.3
PCB 183	5.66
PCB 184	0.26 U ^(b)
PCB 187	12.3
PCB 195	2.38
PCB 206	5.58
PCB 209	5.58
Total Estimated PCB ^(c)	1590
Total Detected PCB	792

TABLE 3.6. Results of Analysis of Liberty Island Anchorage Sediment for PCBs

(a) Mean of replicated sample.

(b) U Undetected at or above the given concentration.

(c) Total PCB = 2(x), where x = sum of all PCB congeners detected; one-half of the detection limit used in summation when analyte was undetected.

3.2.6 PAHs and 1,4-Dichlorobenzene

Table 3.7 shows the results of the analysis of the Liberty Island Anchorage sediments for PAHs. A quality control sample summary and associated quality control data are provided in Appendix A.

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All 16 PAHs analyzed were detected in COMP LI sediment. Low-molecular-weight PAH (LPAH) made up approximately 24% of the total PAH concentration, whereas high-molecular-weight PAH (HPAH) made up 76% of the total. The four PAHs found at the highest concentrations were fluoranthene, pyrene, phenanthrene, and benzo(b)fluoranthene. The COMP LI concentration of 1,4-dichlorobenzene was detected at 204 μ g/kg.

TABLE 3.7. Results of Analysis of Liberty Island Anchorage Sediment for PAHs and 1,4-Dichlorobenzene

Analyte	COMP LI (µg/kg dry weight) ^(a)
naphthalene	380
acenaphthylene	68.0
acenaphthene	234
fluorene	275
phenanthrene	1030
anthracene	428
Total LPAH	2420
fluoranthene	1570
pyrene	1550
benz[a]anthracene	721
chrysene	797
benzo[b]fluoranthene	975
benzo[k]fluoranthene	343
benzo[a]pyrene	733
indeno[1,2,3-c,d]pyrene	451
dibenz[a,h]anthracene	114
benzo[g,h,i]perylene	391
Total HPAH	7650
Total PAH	10,100
1,4-Dichlorobenzene	204

(a) Mean of replicated sample.

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3.2.7 Dioxins and Furans

Table 3.8 shows the results of the analysis of the Liberty Island Anchorage sediment for dioxins/furans. A quality control sample summary and associated quality control data are provided in Appendix A.

Fifteen dioxin/furan congeners were detected in the sediment from COMP LI. Three compounds, OCDD, OCDF, and 1,2,3,4,6,7,8-HpCDD, were detected in COMP LI sediment at concentrations at levels greater than 100 pg/g wet weight.

TABLE 3.8. Results of Analysis of Liberty Island Anchorage Sediment for Dioxins and Furans

	COMP LI
<u>Analyte</u>	<u>(pg/q wet weight)^(a)</u>
2,3,7,8-TCDD	11.8
1,2,3,7,8-PeCDD	0.2 U ^(b)
1,2,3,4,7,8-HxCDD	1.97
1,2,3,6,7,8-HxCDD	12.5
1,2,3,7,8,9-HxCDD	8.86
1,2,3,4,6,7,8-HpCDD	231
OCDD	1741
2,3,7,8-TCDF	10.2
1,2,3,7,8-PeCDF	2.97
2,3,4,7,8-PeCDF	3.94
1,2,3,4,7,8-HxCDF	13.8
1,2,3,6,7,8-HxCDF	3.0
1,2,3,7,8,9-HxCDF	0.2 U
2,3,4,6,7,8-HxCDF	2.24
1,2,3,4,6,7,8-HpCDD	49.9
1,2,3,4,7,8,9-HpCDF	2.66
OCDF	110

(a) Mean of replicated sample.

(b) U Undetected at or above the given concentration; value is calculated using one-half the detection limit for each replicate.

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3.3 Site Water and Elutriate Analyses

Metals, chlorinated pesticides, and PCBs were analyzed in dredging site water collected from Liberty Island Anchorage and in elutriate samples prepared from control seawater (Sequim Bay) and the Liberty Island Anchorage sediment composite. Sequim Bay seawater was used in place of dredging site water due to lower salinity of dredging site water and to maintain consistancy in salinity among the dredging projects tested. Water and elutriate samples were analyzed in triplicate. Mean results of the triplicate analyses are presented and discussed in the following sections. Complete results of site water and elutriate samples, as well as a quality control summary and associated quality control data, are provided in Appendix B.

3.3.1 Metals

Results of analysis of Sequim Bay control water, Liberty Island Anchorage site water, and Liberty Island Anchorage elutriate are shown in Table 3.9. As expected, the Sequim Bay Control water had the lowest concentrations of metals relative to the Liberty Island Anchorage site water of elutriate water samples. Concentrations of Zn, Cu, and Ni were above 0.330 μ g/L for the Sequim Bay control water.

		Concentration in µg/L ^(a)			
<u>Analyte</u>	Sequim Bay Control Water	Liberty Island Anchorage <u>Site Water</u>	Liberty Island Anchorage <u>Elutriate Water</u>		
Ag	0.0090 Q ^(b)	0.0507	0.0133		
Cd	0.0617	0.0867	0.0293		
Cr	0.251	1.06	1.13		
Cu	0.428	2.54	0.799		
Hg	0.0002	0.0131	0.0064		
Ni	0.330	1.01	2.01		
Pb	0.0433	1.71	0.878		
Zn	0.777	15.5	2.37		

<u>TABLE 3.9</u>. Results of Analysis of Liberty Island Anchorage Site Water and Elutriate for Metals

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(a) Values shown are the means of triplicate analyses.

(b) Q Not detected at or above twice the given concentration.

Liberty Island Anchorage Site water had elevated levels of metals that were at least two times higher than in either the elutriate or control water. Concentrations of Hg, Pb, and Zn were 65, 40, and 20 times higher respectively in the Liberty Island Anchorage Site water than in the Sequim Bay control water.

Liberty Island Anchorage elutriate concentrations of metals were more similar to the concentrations found in the Sequim Bay control water.

3.3.2 Chlorinated Pesticides and PCBs

Results of analysis of Sequim Bay control water, Liberty Island Anchorage site water, and Liberty Island Anchorage elutriate are shown in Table 3.10.

Pesticides and PCB congeners were not detected in the Sequim Bay control water. Measurable amounts of all of the pesticides and PCB congeners were found in Liberty Island site water. However, these concentrations were similar to the detection limits for each analyte, except for PCB (44), which was detected at 11.1 ng/L. Three pesticides were detected in Liberty Island Elutriate water sample, two of which (dieldrin and 4,4-DDE) were detected at levels at least six times greater than the Liberty Island Anchorage site water sample. Most of the PCB congeners were detected in the Liberty Island Anchorage elutriate water sample. Eight of these were detected at levels at least five times greater than levels found in the site water.

	Concentration in ng/L ^(a)			
A market a	Sequim Bay	Liberty Island Anchorage	Liberty Island Anchorage	
<u>Analyte</u>	Control Water	<u>Site Water</u>	Elutriate Water	
Pesticides:				
2,4'-DDD	0.49 Q ^(b)	0.51 Q	- 1.27	
2,4'-DDE	0.37 Q	0.38 Q	0.39 Q	
2,4'-DDT	0.44 Q	0.46 Q	0.47 Q	
4,4'-DDD	0.14 Q	0.14 Q	0.15 Q	
4,4'-DDE	0.42 Q	0.44 Q	7.56	
4,4'-DDT	0.25 Q	0.26 Q	0.27 Q	
Total Estimated DDT ⁽	°) 2.11 Q·	2.18 Q	10.1	
Total Detected DDT	0.00	0.00	8.8	

<u>TABLE 3.10</u> .	Results of Analysis of Liberty Island Anchorage Site Water and Elutriate for
-	Chlorinated Pesticides and PCBs

<u>TABLE 3.10</u> .	(continued)
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	Concentration in ng/L ^(a)			
	Sequim Bay	Liberty Island Anchorage	Liberty Island Anchorage	
<u>Analyte</u>	Control Water	Site Water	Elutriate Water	
Pesticides (continue	۹.			
Aldrin	0.38 Q	0.39 Q	0.40 Q	
alpha-Chlordane	0.97 Q	0.99 Q	1.01 Q	
Dieldrin	0.18 Q	0.19 Q	5.17	
Endosulfan I	0.66 Q	0.68 Q	0.69 Q	
Endosulfan II	0.66 Q	0.68 Q	0.69 Q	
Endosulfan Sulfate	0.66 Q	0.68 Q	0.69 Q	
Heptachlor	0.51 Q	0.53 Q	0.54 Q	
Heptachlor Epoxide	1.07 Q	1.10 Q	1.12 Q	
trans-Nonachlor	0.29 Q	0.29 Q	0.30 Q	
PCBs:				
PCB 8	0.58 Q	0.60 Q	0.61 Q	
PCB 18	0.38 Q	0.39 Q	18.4	
PCB 28	0.16 Q	0.61	8.63	
PCB 44	0.28 Q	3.59	11.8	
PCB 49	0.20 Q	0.41	5.76	
PCB 52	0.20 Q	0.21 Q	7.93	
PCB 66	0.21 Q	0.22 Q	8.66	
PCB 87 PCB 101	0.18 Q	0.18 Q	0.19 Q	
PCB 101	0.10 Q 0.09 Q	0.10 Q	3.58	
PCB 105	0.12 Q	0.09 Q 0.12 Q	0.09 Q	
PCB 128	0.12 Q 0.15 Q	0.12 Q 0.15 Q	2.60 0.75	
PCB 138	0.09 Q	0.10 Q	3.29	
PCB 153	0.09 Q	0.09 Q	2.45	
PCB 170	0.18 Q	0.19 Q	0.58	
PCB 180	0.16 Q	0.17 Q	1.95	
PCB 183	0.20 Q	0.21 Q	0.21 Q	
PCB 184	0.20 Q	0.71	0.61	
PCB 187	0.18 Q	0.19 Q	0.19 Q	
PCB 195	0.56 Q	0.58 Q	0.59 Q	
PCB 206	0.12 Q	0.12 Q	0.66	
PCB 209	0.12 Q	0.12 Q	0.13 Q	
Total Estimated PCB ^{(c}	¹⁾ 8.99	18.2	` 159	
Total Detected PCB	0.00	5.32	77.6	
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(a) Values shown are a mean of triplicate analyses.

(b) Q Undetected at or above twice the given concentration.

(c) Sum of 2,4-DDD, 2,4-DDE, 2,4-DDT, 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT; one-half of the detection limit used in summation when analyte was undetected.

(d) Total Estimated PCB = 2(x), where x = sum of all PCB congeners detected; one half of the detection limit used in summation when analyte was undetected.

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3.4 Water-Column Toxicity Testing

Water-column tests were performed on four concentrations of an SPP preparation made from the Liberty Island Anchorage composite. SPP tests were conducted with the silverside *M. beryllina*, the mysid *M. bahia*, and larvae of the bivalve *M. galloprovincialis*. This section discusses the results of all water-column and reference toxicant testing. Complete test results, water quality measurements, and the results of the reference toxicant tests are presented in Appendix C. Throughout this section, the term "significant difference" is used to express *statistically* significant differences only. Tests for statistical significance between test treatment and control treatment were performed following methods outlined in Section 2.6.

3.4.1 Water-Column Toxicity Test with Menidia beryllina

Results of the *M. beryllina* water-column toxicity test are summarized in Table 3.11. Complete test results, and water quality data are presented in Appendix C. Control survival was 98%, validating this test. Survival in the 100% SPP preparation was 88% and was not significantly lower than the control. The *M. beryllina* LC_{50} of the Liberty Island Anchorage composite could not be calculated because 50% mortality did not occur in any treatment.

All water quality parameters were within acceptable ranges throughout the test. The Cu reference toxicant test produced an LC₅₀ of 100.0 μ g/L Cu, which was within the control limits established at the MSL (71 μ g/L to 136 μ g/L Cu).

3.4.2 Water-Column Toxicity Test with Mysidopsis bahia

Results of the *M. bahia* water-column toxicity test are summarized in Table 3.11. Complete test results and water quality data are presented in Appendix C. This test was validated by a control survival of 98%. Survival in the 100% SPP preparation was 94% and was not significantly lower than the controls. The *M. bahia* LC_{50} for the Liberty Island Anchorage composite could not be calculated because 50% mortality did not occur.

All water quality parameters were within acceptable ranges throughout the test. The Cu reference toxicant test produced an LC₅₀ of 273.6 μ g/L Cu, which is above the control limits established at the MSL (116 μ g/L to 229 μ g/L) suggesting that a less sensitive population of organisms could have been used for testing.

3.4.3 Water-Column Toxicity Test with Mytilus galloprovincialis

Results of the *M. galloprovincialis* water-column toxicity test are summarized in Table 3.11. Complete test results and water quality data are presented in Appendix C. This test was validated by 91% survival in the control. Survival was 93% in the 100% SPP preparation and was not significantly lower than the control. The LC_{50} could not be calculated because 50% mortality did not occur in any concentration of SPP. Normal development, considered a more sensitive indicator of toxicity, was significantly reduced in the 100% SPP, with 20% normal prodissoconch in this treatment. The EC₅₀ was 28.5% SPP.

All water quality parameters were within acceptable ranges throughout the test. The Cu reference toxicant test revealed an LC₅₀ of 10.26 μ g/L Cu and an EC₅₀ of 3.29 μ g/L Cu, which were within the control limits for the LC₅₀ (LC₅₀: 5.8 μ g/L to 35 μ g/L Cu) but slightly below the control EC₅₀ (EC₅₀ limits for the: 5.7 μ g/L to 21 μ g/L Cu) established at the MSL.

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<u>Test Organism</u>	Percent Survival/Normal Development in 0% SPP	Percent Survival/Normal Development in 100% SPP	Significant <u>Difference</u>	LC ₅₀ /EC ₅₀ (<u>%SPP)</u>
Menidia beryllina	98	88	No	>100
Mysidopsis bahia	98	94	No	>100
<i>Mytilus galloprovincialis</i> (Survival)	s 91	93	No	>100
<i>Mytilus galloprovincialis</i> (Normal development)	86	20	Yes	28.5 ^(a)

<u>TABLE 3.11</u>. Summary of Water-Column Toxicity Tests Performed with Liberty Island Anchorage Sediment

(a) Median-effective concentration (EC₅₀) based on normal development to the D-shaped, prodissoconch stage.

3.5 Benthic Acute Toxicity Tests

Benthic acute toxicity tests were performed on the Liberty Island Anchorage composite and Mud Dump Reference Site sediment. Benthic tests were conducted with the amphipod *A. abdita*, and the mysid *M. bahia*. This section discusses the results of all benthic and reference toxicant testing. Complete test results, water quality measurements, and the results of the reference toxicant tests are presented in Appendix D. Throughout this section the term "acutely toxic" is used to express *statistically* significant differences and greater than 10% (mysid) or 20% (amphipod) decreases in survival from the reference sediment. Tests for statistical significance between the treatments and reference treatment were performed following methods outlined in Section 2.6.

3.5.1 Static Renewal Test with Ampelisca abdita

Results of the benthic toxicity test with *A. abdita* are summarized in Table 3.12. Complete test results and water quality data are presented in Appendix D. Survival in the *A. abdita* control sediment was 88%. Survival in the Liberty Island Anchorage composite was 52% and was acutely toxic compared to the Mud Dump Reference Site sediment (94% survival).

Water quality parameters were within acceptable ranges throughout the test. The Cd reference toxicant test produced an LC_{50} of 0.54 mg/L Cd, which was within the control limits established at the MSL (0.5 mg/L to 1.4 mg/L Cd). Prior to test setup, total ammonia concentration measured in the Liberty Island Anchorage bulk sediment composite was about 228 mg/L. Pore water ammonia was measured in "dummy" jars every few days until concentrations were 30 mg/L or less. The test was initiated after 11 days when the pore water ammonia concentration was 21.9 mg/L. Ammonia concentrations were less than 2.0 mg/L in the overlying water during the 10-day test, and were 13.9 mg/L in the porewater at test termination.

<u>Test Organism</u>	Mean % <u>Survival</u>	0	
A. abdita	52%	Yes	Yes
M. bahia	95%	No	No

<u>TABLE 3.12</u>. Summary of Benthic Acute Toxicity Tests Performed with Liberty Island Anchorage Sediment

(a) MDRS Mud Dump Reference Site.

(b) Acutely Toxic = Statistically significant mortality in the test treatment that is 20% greater than the reference for amphipods and 10% greater than the reference for mysids.

3.5.2 Static Renewal Test with Mysidopsis bahia

Results of the static renewal benthic toxicity test with *M. bahia* are summarized in Table 3.12. Complete test results and water quality data are presented in Appendix D. The static-renewal toxicity test with the mysid was conducted twice: once on June 26, 1994 and again on July 25, 1994 due to unacceptable control survival from the first test. The results from the second test were used to assess benthic effects of COMP LI to the mysid. Control survival was 93%, validating the test. Survival in the Liberty Island Anchorage composite was 95% and was not acutely toxic compared to the Mud Dump Reference Site sediment (88% survival).

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All water quality parameters were within acceptable ranges throughout the test. The Cu reference toxicant test produced an LC₅₀ of 265.6 μ g/L Cu, which is slightly above the control limits previously established at the MSL (116 μ g/L to 230 μ g/L Cu). As described in Section 2.5.2.2, the ammonia purging procedures were employed to reduce ammonia in the overlying water to nontoxic concentrations. Test chambers containing sediment and overlying water were set up and maintained under test temperatures with aeration during the ammonia purging period. Overlying water was exchanged two times daily for 6 days. The test was initiated when the overlying-water ammonia concentration was <1.0 mg/L. Ammonia concentrations in overlying water of the Liberty Island Anchorage composite treatments ranged from <1.0 mg/L to 6.4 mg/L.

3.6 Bioaccumulation Testing

Bioaccumulation tests with *M. nasuta, T. japonica*, and *N. virens* were conducted using the Liberty Island Anchorage composite, the Mud Dump Reference Site and appropriate control sediments. All organisms were exposed for 28 days under flow-through conditions. Survival of each species relative to the Liberty Island Anchorage composite was 98% in the *M. nasuta* test, 100% survival in the *T. japonica* test, and 97% in the *N. virens* test. All tests were validated by control survival of \ge 94%.

A Cu reference toxicant test was conducted on all three species. The LC₅₀ values for *M. nasuta* and *T. japonica* could not be calculated because greater than 50% mortality did not occur even at the highest concentration of 2.5 mg/L. An LC₅₀ of 0.17 mg/L was calculated for *N. virens*. Complete test results and water quality data are presented in Appendix E. The tissues of the exposed organisms have been frozen and archived at <-20°C should analysis be requested.

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4.0 Discussion and Conclusions

In this section, physical and chemical analyses, and bioassays performed on the Liberty Island Anchorage sediment composite are evaluated relative to the Mud Dump Reference Site sediment by the guidelines of the Green Book Tier III. Tier III evaluations include water-column toxicity tests, benthic toxicity tests, and whole-sediment bioaccumulation studies. Tier III evaluations assess the impact of contaminants in the dredged material on marine organisms to determine whether there is potential for the material to have an unacceptable environmental effect during ocean disposal. The Green Book provides the following guidance for determining whether the proposed dredged material is unacceptable for ocean disposal based on the Tier III test:

- <u>Water-Column Toxicity</u>. The limiting permissible concentration (LPC) of dissolved plus suspended contaminants cannot exceed 0.01 of the acutely toxic concentration at the boundaries of the disposal site within the first 4 h after disposal, or at any point in the marine environment after the first 4 h. The acutely toxic concentration in this case is taken to be the median lethal concentration (LC_{50}); therefore, acute toxicity in SPP tests would require at least 50% mortality in an SPP treatment to be evaluated according to the Green Book. A numerical mixing model should be used to predict whether concentrations greater than 0.01 of the acutely toxic SPP concentrations are likely to occur beyond the boundaries of the disposal site within the first 4 h after disposal.
- <u>Benthic Acute Toxicity</u>. The proposed dredged material does not meet the LPC for benthic toxicity when organism survival in the test sediment and the reference site sediment is statistically significant, and the decrease in survival is at least 20% for *A. abdita* or at least 10% for *M. bahia*.

Sections 4.1 through 4.4 discuss the proposed Liberty Island Anchorage dredged material in terms of sediment characterization and Tier III evaluations. The matrix in Figure 4.1 summarizes water-column and benthic acute toxicity for the Liberty Island Channel sediment composites.

4.1 Sediment Physical and Chemical Characterization

Liberty Island Anchorage sediment core samples were predominantly black, silty-clayey material. Percentages of silt ranged from 8% to 53%, and clay ranged from 17% to 59%. Sediment moisture contents varied from 42% to 58% in individual cores. Detectable levels of all metals were present in the Liberty Island Anchorage composite; Cr, Cu, Pb, and Zn were found at concentrations greater than 200 ppm. The dominant pesticides found were those in the DDD/DDE/DDT group of compounds. Twenty-one of the 22 PCB congeners analyzed were detected in Liberty Island Anchorage sediment, with a total PCB concentration of 1738 μ g/kg, dry weight. All 16 PAHs analyzed were detected in Liberty Island Anchorage sediment. Total PAH was 10,060 μ g/kg, dry weight; 24% of the total was LPAH; 76% of the total was HPAH. The concentration of 1,4-dichlorobenzene was 204 μ g/kg, dry weight. Fifteen dioxin/furan congeners were present in the Liberty Island Anchorage sediment composite, three of which were found at concentrations at least one order of magnitude above the remaining congeners.

4.2 Site Water and Elutriate Chemical Characterization

Sequim Bay control water had the lowest concentrations of metals, when compared with Liberty Island Anchorage Site water or Liberty Island Anchorage elutriate water. Metals concentrations were predominantly highest in the Liberty Island Anchorage site water. Pesticides were undetected in the Sequim Bay control water, and were detected in the site water and elutriate water at concentrations near the detection limit for each analyte. Measurable amounts of the PCB congener CL4(44) were found in Liberty Island Anchorage site water . Liberty Island Anchorage elutriate water concentrations for several PCBs were at least five times greater than those found in the site water.

4.3 Toxicity

In water-column toxicity tests, 100% SPP treatments were not acutely toxic to *M. beryllina M. bahia* or *M. galloprovincialis* survival. The EC₅₀ for *M. galloprovincialis* normal development, a more sensitive measure than survival, was 26.9% SPP. The LPC for water-

column effects outside of the disposal site boundaries after 4 h is 0.27% SPP for Liberty Island Anchorage sediment. A projection of SPP concentrations exceeding this value after 4 h at the Mud Dump Site boundary would be unacceptable.

The sediment composite LI was acutely toxic in mortality over the reference sediment in the static renewal test with *A. abdita*. Sediment from this composite was not acutely toxic to *M. bahia*. Therefore, Liberty Island Anchorage sediment did not meet the LPC for benthic toxicity to *A. abdita* if the observed effects are due to persistent contaminants.

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4.4 Bioaccumulation

Bioaccumulation tests were conducted and the survival results were discussed in Section 3.6. However, the tissues were not analyzed at the request of the New York District, USACE; and are currently being archived in the event that analysis is requested.

		Sediment Treatment Liberty Island
ity	A. abdita Benthic Static-Renewal Test	AT ^(a)
Acute Toxicity	M. bahia Benthic Static-Renewal Test	- ^(b)
Ĕ	M. beryllina SPP Test	-
ute	M. bahia SPP Test	-
Ac Ac	M. galloprovinciallis SPP Test	-

(a) AT Acutely toxic; significantly different from reference and mortality 20% (10% for mysids) greater than reference.

(b) - Not Acutely Toxic/No Significant Difference.

FIGURE 4.1 Summary Matrix of Liberty Island Channel Toxicity

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5.0 References

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Appendix A.

Quality Assurance/Quality Control Data for Sediment Physical/Chemical Analyses, Liberty Island Anchorage

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PROGRAM: New York/New Jersey Federal Projects 3

PARAMETER: Grain Size

LABORATORY: Soil Technology, Bainbridge Island, Washington

MATRIX: Sediment

QA/QC DATA QUALITY OBJECTIVES

Reference Method	Range of <u>Recovery</u>	Relative <u>Precision</u>	Detection <u>Limit (µg/L)</u>	
Plumb 1981	Not applicable	≤20% for fractions greater than 5%	1.0%	
METHOD	Four grain size fractions were determined by a combination of sieve and pipet techniques. An additional measurement for salt content was performed and each grain size fraction was corrected for this salt measurement.			
HOLDING TIMES	The holding time of 6 months was met for all grain size analyses.			
DETECTION LIMITS	Target detection limits	of 0.1% were met for all sa	amples.	
METHOD BLANKS	Not Applicable.			
MATRIX SPIKES	Not Applicable.			
REPLICATES	One sample was analyzed in triplicate. Precision for triplicate analyses is reported by calculating the relative standard deviation (RSD) between the replicate results. The RSDs were within the QC limits of ≤20%.			
SRMS	Not Applicable.			

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PROGRAM:	New York/New Jersey Federal Projects 3			
PARAMETER:	Total Organic Carbon (TOC)			
LABORATORY:	Applied Marine Sciences, Inc., College Station, Texas			
MATRIX:	Sediment			
QA/QC DATA QUALIT	YOBJECTIVES			
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Reference	Range of	Relative	Detection
<u>Method</u>	<u>Recovery</u>	<u>Precision</u>	<u>Limit (μg/L)</u>
EPA 1986	≤20%	≤10%	0.1%

METHOD Total organic carbon is the amount of non-volatile, partially volatile, volatile, and particulate organic carbon compounds in a sample. Each sample was dried and ball milled to a fine powder. Before combustion, inorganic carbon in the sample was removed by acidification. The TOC was then determined by measuring the carbon dioxide released during combustion of the sample.

- **DETECTION LIMITS** Calculated method detection limit (MDL) for the coulometer was 0.1%, which meets the target detection limit.
- **HOLDING TIMES** The holding time of 6 months was met for all TOC analyses.
- METHOD BLANKS Not Applicable.

REPLICATES One sample was analyzed in triplicate with the samples. Precision for triplicate analyses is reported by calculating the relative standard deviation (RSD) between the replicate results. The calculated RSD was 1%, which is within the QC limits of ≤10%.

STANDARD REFERENCE MATERIAL

One SRM, NIST 1941a, was analyzed with each batch of samples. The values obtained for each SRM were within 30% of the certified value, indicating acceptable accuracy of the method.

REFERENCES

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PROGRAM: New York/New Jersey Federal Projects 3

PARAMETER: Metals

LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington

MATRIX: Sediment

QA/QC DATA QUALITY OBJECTIVES

	Reference	Range of	SRM	Relative	Detection
	Method	<u>Recovery</u>	<u>Accuracy</u>	Precision	Limit (dry wt)
Arsenic Cadmium Chromium Copper Lead Mercury Nickel Silver Zinc	ICP/MS ICP/MS ICP/MS ICP/MS ICP/MS ICP/MS ICP/MS ICP/MS	75-125% 75-125% 75-125% 75-125% 75-125% 75-125% 75-125% 75-125% 75-125%	≤20% ≤20% ≤20% ≤20% ≤20% ≤20% ≤20% ≤20%	≤20% ≤20% ≤20% ≤20% ≤20% ≤20% ≤20% ≤20%	0.572 0.020 0.401 0.525 0.136 0.001 0.849 0.119 2.55

METHOD

A total of nine metals was analyzed: silver (Ag), arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), nickel (Ni), lead (Pb) and zinc (Zn). Hg was analyzed using cold-vapor atomic absorption spectroscopy (CVAA) according to the method of Bloom and Crecelius (1983). The remaining metals were analyzed by inductively coupled plasma mass spectrometry (ICP/MS) following EPA Method 200.8 (EPA 1991).

Achieved

To prepare sediment samples for analysis, samples were freeze-dried and blended in a Spex mixer-mill. Approximately 5 g of mixed sample was ground in a ceramic ball mill. For ICP/MS and CVAA analyses, 0.2- to 0.5-g aliquots of dried homogenous sample were digested using a mixture of nitric and hydrochloric acids and hydrogen peroxide following EPA Method 200.2 (EPA 1991).

HOLDING TIMES One sample was received in good condition, frozen to -80°C, and freeze-dried. The following list summarizes all analysis dates:

<u>Task</u>	Date Performed
Sample Digestion ICP-MS CVAA	10/4/94 10/24/94 10/12/94

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QA/QC SUMMARY/METALS (contd)

- **DETECTION LIMITS** Target detection limits were exceeded for some metals; however metals were detected above the MDLs in all samples with the exception of Ag in one sample. MDLs were determined by multiplying the standard deviation of the results of 4 replicate low level sediment spikes by 3.5.
- METHOD BLANKS One method blank was analyzed. Only Cr was detected in the method blank. The value was less than 3 times the MDL and all sample values were detected at levels greater than 5 times the blank concentrations, therefore the data were blank corrected.
- MATRIX SPIKES One sample was spiked with all nine metals. Native concentrations in the sample were higher than the spike concentrations at the low level so recoveries were not very representative of the accuracy of the method. In some cases, no recoveries could be reported due to the excessive native levels. Many recoveries for the higher level spike, however, were still within the QC limits of 75% to 125%. Recoveries of Ag in the high spike were low due to precipitation of Ag as AgCL. Recoveries of Ag in both spikes were low due to precipitation of Ag as AgCl in when high levels of Ag are present. Since the samples were spiked with 25 ppm of Ag, this is most likely a result of the high Ag spike, rather than a problem with the sample itself.
- **REPLICATES** The sample was digested and analyzed in triplicate. Precision for triplicate analyses is reported by calculating the relative standard deviation (RSD) between the replicate results. RSD values ranged from 1 to 7%, within the QC limits of ±20%, indicating acceptable precision.
- SRM SRM 1646a (estuarine sediment from the National Institute of Standards and Technology, NIST), was analyzed for all metals. Only results for Hg were within ±20 % of the certified value (Ag is not certified). Values for the remaining metals were low because the digestion method used is not as strong as the method (perchloric acid) used to certify the SRM, thus the results for this analysis should not be expected to match the SRM certified values. Therefore, no corrective actions were taken.

REFERENCES

Bloom, N. S., and E.A. Crecelius. 1983. *Determination of Mercury in Seawater at Sub-Nanogram per Liter Levels*. <u>Mar. Chem.</u> 14:49-59.

EPA (U.S. Environmental Protection Agency). 1991. Methods for the Determination of Metals in Environmental Samples. EPA-600/4-91-010. Environmental Services Division, Monitoring Management Branch, Washington D.C.

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PROGRAM: New York/New Jersey Federal Projects 3

PARAMETER: Chlorinated Pesticides

LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington

MATRIX: Sediment

QA/QC DATA QUALITY OBJECTIVES

Reference	Surrogate	MS	Relative	Detection
Method	<u>Recovery</u>	<u>Recovery</u>	Precision	<u>Limit</u>
GC/ECD	30-150%	50-120%	≤30%	2-20 ng/L

SAMPLE CUSTODY One sample was received on 8/17/94 in good condition. The sample was logged into Battelle's log-in system and stored at approximately 4°C until extraction.

- METHOD Sediment samples were extracted with methylene chloride using a roller under ambient conditions following a MSL procedure based on EPA Method 3510 and 8080 (EPA (1986) and National Oceanic and Atmospheric Administration for the Status and Trends Program (Krahn et al. 1988). Samples were then cleaned using silica/alumina (5% deactivated) chromatography followed by HPLC cleanup (Krahn et al. 1988). Extracts were analyzed using Gas Chromatography/Electron Capture Detection (GC/ECD) following a procedure based on EPA method 8080 (EPA 1986). The column used was a J&W DB-17 and the confirmatory column was a DB-1701, both capillary columns (30-m x 0.25-mm I.D.).
- HOLDING TIMES Samples were stored at approximately -20°C until extraction. Samples were extracted on 9/9/94. Extracts were analyzed by GC/ECD on 9/20/94.
- **DETECTION LIMITS** Target detection limits were met for all samples. Actual method detection limits were determined from multiplying the standard deviation of 7 spiked sediment replicates by the student t value.
- METHOD BLANKS One method blank was extracted with each extraction batch. No pesticides or PCBS were detected in the blanks with the exception of PCB 28 at a level less than 3 times the MDL.

QA/QC SUMMARY/CHLORINATED PESTICIDES/PCBs (contd)

- SURROGATES Two compounds, PCB congeners 103 and 198, were added to all samples prior to extraction to assess the efficiency of the analysis. These compounds are also used to correct all sample results and are considered surrogate internal standards (SIS). Sample surrogate recoveries were all within the QC guidelines of 30-150% with the exception of PCB 198 in the blank and the SRM. The recovery was above the internal warning limit of 20% in both cases.
- MATRIX SPIKES One sample was spiked with a subset of 6 pesticides and 5 PCB congeners. For all compounds except Endosulfan II, the amount spiked was less than the amount in the native sample. For this reason, many spike recoveries were outside of the control limits.
- **REPLICATES** One sample was extracted in triplicate. Precision was measured by calculating the relative standard deviation (RSD) between the replicate results. RSDs for all compounds, except 4,4'-DDT were within the control limits of ≤30%.

SRMs One SRM, 1941 a marine sediment obtained by the National Institute of Science and Technology was analyzed with the samples. Four pesticides and 15 PCB congeners are certified. The recovery of 3 of the 4 pesticides were within the control limits ≤30%. 2,4'-DDE is certified at a level just above the MDL and was not detected. Only 2 of the 15 PCB congeners were detected at levels significantly outside the recovery range ≥30%. This is probably due to apparent interferences present in the SRM that could not be resolved by our GC system. The remaining congeners were all within 33% of the certified values.

MISCELLANEOUS

All pesticide detections were confirmed on a second, dissimilar column. The RPD criteria is set at 190% due to many coelutions between pesticides and PCB congeners. The confirmation, therefore, was primarily qualitative. When a known congener coeluted on the primary column, the value taken for that compound came for the rear, confirmation column. Compounds quantified from the confirmation column include 4,4'-DDE, beta BHC, and endosulfan II.

REFERENCES

Krahn, M.M., C.A. Wigren, R.W. Pearce, L.K. Moore, R.G. Bogar, W.D. MacLeod, Jr., S.L. Chan, and D.W. Brown. 1988 "New HPLC Cleanup and Revised Extraction Procedures for Organic Contaminants," NOAA Technical Memorandum NMFS F/NWC-153. NOAA/NMFS/NWFSC, Seattle, Washington.

U.S. Environmental Protection Agency (EPA). 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SW-846. U.S. Document No. 955-001-00000, U.S. Environmental Protection Agency, Washington D. C.

QA/QC SUMMARY

PROGRAM: New York/New Jersey Federal Projects 3

PARAMETER: Polynuclear Aromatic Hydrocarbons (PAH) and 1,4-Dichlorobenzene

LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington

MATRIX: Sediment

QA/QC DATA QUALITY OBJECTIVES

Reference	MS	Surrogate	SRM	Relative	Detection
Method	<u>Recovery</u>	<u>Recovery</u>	<u>Accuracy</u>	<u>Precision</u>	<u>Limit (wet wt)</u>
GC/MS/SIM	50-120%	30-150%	≤30%	≤30%	4 ng/g

SAMPLE CUSTODY One sediment sample was received on 8/17/94 in good condition. The sample was logged into Battelle's log-in system and stored frozen until extraction.

METHOD The sediment sample was extracted with methylene chloride using a roller under ambient conditions following a procedure that is based on methods used by the National Oceanic and Atmospheric Administration for its Status and Trends Program (Krahn et al. 1988). Samples were then cleaned using Silica/Alumina (5% deactivated) chromatography followed by HPLC cleanup.

Extracts were quantified using gas chromatography/mass spectrometry (GC/MS) in the selected ion mode (SIM) following a procedure based on EPA method 8270 (EPA 1986).

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HOLDING TIMES The same was extracted on 9/9/94. All extracts were analyzed by GC/MS/SIM on 9/23/94.

DETECTION LIMITS Target detection limits of 4 ng/g wet weight were met for all PAH compounds except for fluoranthene and pyrene, which had method detection limits (MDLs) of between 4 and 6 ng/g wet wt. MDLs were determined by multiplying the standard deviation of 7 spiked replicates of a background clam sample by the student t value. All PAHs were detected well above the MDLs in the sample.

METHOD BLANKS One method blank was extracted with the extraction batch. Naphthalene, phenanthrene, and pyrene were detected in the blank. All blank levels were less than 3 times the target MDL of 4 ng/g wet wt and all sample concentrations were well above 5 times the blank concentration, therefore, no data were flagged.

QA/QC SUMMARY/PAHs (contd)

SURROGATES Five isotopically labelled compounds were added prior to extraction to assess the efficiency of the method. These were d8-naphthalene, d10-acenaphthene, d12-chrysene, d14-dibenzo(a,h)anthracene and d4-1,4 dichlorobenzene. Recoveries of all surrogates were outside of the quality control limits of 30-150% for the blank and the SRM. In addition, recoveries for d4-1,4 dichlorobenzene were low in all samples and recoveries of d8-naphthalene were low in rep-1 of NY3-ORG-05. Overall, surrogate recoveries for the other compounds, though above 30%, were low, ranging from 34 to 58%. These low recoveries are due to extra sample processing as a result of an additional copper clean up needed to remove sulfur from the sample. This additional handling resulted in the losses evidenced by the low surrogate recoveries. All sample results are surrogate corrected and because the SRM results were generally acceptable, indicating acceptable accuracy and the triplicate RSDs were good, indicating acceptable precision, no data were flagged. MATRIX SPIKES One sample from each batch was spiked with all PAH compounds. Matrix spike recoveries were outside of the QC limits of 50-120% due to high native levels, relative to spiking levels. Spike concentrations were from 3 to 60 times lower than native concentrations. REPLICATES One sample from each batch was extracted and analyzed in triplicate. Precision was measured by calculating the relative standard deviation (RSD) between the replicate results. RSDs ranged from 1 to 6 %, within $\pm 30\%$, indicating acceptable precision. SRMs One SRM,1941, a marine sediment obtained by the National Institute for Science and Technology was analyzed with the samples. Fourteen of the 16 PAH compounds analyzed are certified. Ten of the 14 PAHs were detected within 30% of the certified mean. Three

compounds; chrysene, benzo(b)fluoranthene and dibenzo(a,h)anthracene were recovered above the certified range at recoveries ranging from 138 to 165%. One compound, benzo(ghi)perylene, was recovered at 67%, below the lower QC limit.

MISCELLANEOUS

Some of the compounds are flagged with "*" to indicate that the ion ratio for that compound was outside of the QC range. This is due primarily to low levels of the compound of interest. Because the confirmation ion is present at only a fraction of the level of the parent ion, when the native level of the compound is low, the amount of error in the concentration measurement of the confirmation ion goes up. The compound is actually quantified from the parent ion only so most likely this will not effect the quality of the data. For sample values that are relatively high (>5 times the MDL) it may be an indication of some sort of interference.

REFERENCES

Krahn, M.M., C.A. Wigren, R.W. Pearce, L.K. Moore, R.G. Bogar, W.D. MacLeod, Jr., S.L. Chan, and D.W. Brown. 1988 "New HPLC Cleanup and Revised Extraction Procedures for Organic Contaminants," NOAA Technical Memorandum NMFS F/NWC-153. NOAA/NMFS/NWFSC, Seattle, Washington.

EPA (U.S. Environmental Protection Agency). 1986. <u>Test Methods for Evaluating Solid Waste:</u> <u>Physical/Chemical Methods.</u> SW-846. U.S. Document No. 955-001-00000, U.S. EnvironmentalProtection Agency, Washington D.C.

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QA/QC SUMMARY

PROGRAM: New York/New Jersey Federal Projects 3

PARAMETER: Dioxins

LABORATORY: Battelle, Columbus, Ohio

MATRIX: Sediment

QA/QC DATA QUALITY OBJECTIVES

Reference	MS	Surrogate	SRM	Relative Detection
Method	<u>Recovery</u>	<u>Recovery</u>	<u>Accuracy</u>	Precision Limit (dry wt)
EPA 8290	50% - 120%	40 - 135%	≤30%	≤25% 1 pg/g - 5 pg/g

METHOD General procedures in EPA Method 8290 were followed for the analysis of 2,3,7,8-TCDD/TCDF. Samples were solvent extracted and processed through acid/base silica, alumina, and carbon column cleanup procedures. Samples were analyzed by gas chromatography/high resolution mass spectrometry (GC/HRMS) on a DB-Dioxin column. The DB-Dioxin column provided separation of 2,3,7,8-TCDD/TCDF from all other tetra isomers so that no second column confirmation analysis is necessary.

- HOLDING TIMES Samples were held frozen until extraction. Samples were collected in the field from June 7 through June 10, 1994, extracted on July 21, 1994 and analyzed from August 4 through August 9, 1994.
- DETECTION LIMITS Target detection limits were met for all analytes except 12378 PeCDD for COMP LI Rep. 1.
- METHOD BLANKS One method blank was analyzed with the sediment samples. Six dioxin/furan congeners were detected in the blank at concentrations ranging from 0.34 pg/g to 3.60 pg/g. No corrective action was taken because concentrations were less than three times the MDL for each analyte.
- **REPLICATES** COMP LI was analyzed in triplicate. Precision was measured by calculating the relative standard deviation (RSD) between the replicate results. RSDs ranged from 10.1% to 38.1% with three of the RSDs exceeding the QA/QC limit of <25% for this analyte. Note that the three analytes with the outlying RSDs were present in the samples at less than 10 times the MDL level.
- MATRIX SPIKE COMP LI was spiked with all 17 dioxin/furan congeners analyzed for this project. Accuracy of the spiking method was measured by calculating a percent recovery for each analyte. Only three analytes, TCDD, HpCDD, and OCDD were outside of the QA/QC limit of 50% to 120% established for these compounds. These outlying results are likely due to the large amount of native analytes present in the samples relative to the spike level.

QA/QC SUMMARY FOR DIOXIN/FURANS (contd)

SURROGATESAll samples were spiked with labeled internal standards to assess
method performance. Recoveries of the labeled compounds within
40% to 135% except for one analyte in the standard reference material.SRMA fortified reference material was used as an SRM. This reference
material is not certified but is used routinely by the analytical laboratory
to assess accuracy. All recoveries are within ±30% of the in-house
target values.

REFERENCES

EPA (U.S. Environmental Protection Agency). 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SW-846. U.S. Document No. 955-001-00000, U.S. Environmental Protection Agency, Washington D.C.

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Sediment		Gravel	Sand	Silt	Clay	Percent
Treatment	Batch	> 2.0 mm	2.0 - 0.063 mm	0.063 - 0.004 mm	< 0.004 mm	Moisture
LI-1	· 1	4	26	38	32	53
LI-1A ^(a)	1	13	65	19	3	12
LI-2	1	13	40	30	17	42
LI-3	1	0	5	53	42	58
LI-3A ^(b)	1	0	5	70	25	23
LI-4	1	8	25	8	59	53
LI-5	1	0	5	53	42	67
LI-6	1	1	42	34	23	47
LI-6A ^(c)	1	0	97	2	1	20
LI-7	1	0	7	53	40	64
LI-8	1	0	3	52	45	65
LI-9	1	0	8	51	41	63
LI-10	1	0	22	43	35	58
Comp LI, Replicate 1	1	1	23	41	35	56
Comp LI, Replicate 2	1	2	21	43	34	55
Comp LI, Replicate 3	1	3	23	39	35	56
RSD ^(d)			5%	5%	2%	2%
					_	
Mud Dump Reference Site	1	1	98	<i>.</i> 0	1	17
Ampelisca Control	1	0	6	69	25	71
Nereis Control	1	0	71	15	14	33
Mysid, Macoma and Tapes Control	1	1	15	53	31	72

Table A.1. Grain Size Analysis of Liberty Island Anchorage Sediments

(a) Extra grain size sample taken from a 2 ft band of brown sand with rocks.

(b) Extra grain size sample taken from a 1 ft band of red clay.

(c) Extra grain size sample taken from a 1 ft band of gray sand.

(d) RSD Relative standard deviation.

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Sediment			TOC
Treatment	Replicate	Batch	(% dry weight)
LI-1		2	2.63
LI-2		2	1.86
LI-3		2	3.13
LI-4		2	3.63
LI-5		2	3.88
LI-6		2	2.07
LI-7		2	3.78
LI-8		2	3.93
LI-9		2	4.16
LI-10		2	3.46
Comp L1		1	3.06
Mud Dump Reference		1	0.006
Ampelisca Control		1	3.10
Nereis Control		1	1.34
Mysid/Macoma Control		1	2.42
Quality Control Data Standard Reference Material			
Certified Value			4.80 ±1.2%
NIST 1941a		1	4.74
Percent Difference			1%
NIST 1941a		2	4.66
Percent Difference			3%
Triplicate Analysis			
MT-A-5	1	1	2.01
MT-A-5	2	1	2.00
MT-A-5	3	1	1.99
RSD ^(a)			0%
QC Sample (LI-3)	1	2	3.11
QC Sample (LI-3)	2	2	3.17
QC Sample (LI-3)	3	2	3.11
RSD			1%

TABLE A.2. Sediment Total Organic Carbon (TOC), Liberty Island Anchorage

(a) RSD Relative standard deviation.

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Sediment						Metals	(µg/g dry wt.)		
Treatment	Batch	Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Target DL ^(a) Achieved DL		0.1 0.119	0.1 0.572	0.01 0.020	0.02 0.401	0.1 0.525	0.1 0.002	0.1 0.849	0.1 0.136	0.1 2.55
Method Blank										
Blank	1	0.119 U ^(D)	0.57 U	0.02 U	0.675	0.53 U	0.002 U	0.85 U	0.†4 U	2.55 U
Matrix Spike										
COMP LI ^(c) COMP LI, MS Concentration Recovered Amount Spiked Percent Recovery	1 1	8.57 10.7 2.13 2.00 107%	15.5 17.3 1.77 2.00 88%	4.12 6.02 1.90 2.00 95%	214 208 0 2.00 NR ^(¤)	204 204 0 2.00 NR	2.88 4.62 1.74 2.00 87%	43.0 44.4 1.43 2.00 72%	228 222 0 2.00 NR	258 254 0 2.00 NR
COMP LI ^(c) COMP LI, MSD Concentration Recovered Amount Spiked Percent Recovery	1 1	8.57 19.2 10.6 25.0 43% ⁽¹⁾	15.5 39.3 23.8 25.0 95%	4.12 28.4 24.3 25.0 97%	214 231 17.0 25.0 68% ⁽¹⁾	204 223 19.0 25.0 76%	NS ^(e) NS NS NS NS	43.0 66.6 23.6 25.0 95%	228 243 15.0 25.0 60% ⁽¹⁾	258 273 15.0 25.0 60% ⁽¹⁾
RPD ⁽⁹⁾ I-Stat ⁽¹⁾		86% 0.43	7% 0.04	2% 0.01	NA ⁽ⁿ⁾ NA	NA NA	NA NA	28% 0.14	NA NA	NA NA
Standard Reference Materia	al					,				
Certified value Range		NC ^(I) NC	11.6 ±1.3	0.36 ±0.07	76 ±3	18 ±3	0.063 ±0.012	32 ±3	28.2 ±1.8	138 ±6
SRM 1646 PD [⊮]	1	0.078 NA	5.89 49% ^{\v}	0.190 47% ^{ייי}	35.9 53%	10.3 43% ^ሠ	0.060 5%	17.6 45% ^w	15.8 44% ^w	55.8 60% ⁽¹⁾

TABLE A.3. Quality Control Data for Metals, Dry Weight, in Liberty Island Anchorage Sediment

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TABLE A.3. (contd)

Sediment		Metals (μg/g dry wt.)								
Treatment	Batch	Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Analytical Replicates										
COMP LI, Replicate 1 COMP LI, Replicate 2 COMP LI, Replicate 3	1 1 1	7.91 9.00 8.80	15.4 15.8 15.4	4.16 4.15 4.06	212 219 210	205 201 206	3.09 2.76 2.78	42.5 42.7 43.7	234 227 223	259 255 260
RSD ^(m)		7%	1%	1%	2%	1%	6%	1%	2%	1%

(a) DL Detection limit.

(b) U Undetected at or above the given concentration.

(c) Value is mean of the replicates.

(d) NR Not recoverable; spike concentration below native concentration.

(e) NS Not spiked.

(f) Outside quality control criteria (75-125%) for matrix spike recoveries.
(g) RPD - Relative percent difference.

(h) NA Not applicable.

(i) I-Stat.

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(j) NC Not certified.

(k) PD Percent difference.

(I) Outside quality control criteria (±20%) for SRMs.
 (m) RSD Relative standard deviation.

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	Analytical Replicates (ng/g dry)				
	Method	COMP LI	COMP LI	COMP LI	
Analyte	Blank	REP 1	REP 2	REP 3	RSD% ^(a)
Handaafilan					
Heptachlor	0.06 U ^(b)	0.06 U	0.06 U	0.06 U	NA ^(c)
Aldrin	0.20 U	9.05	9.07	9.46	3
Heptachlor Epoxide	0.29 U	0.29 U	0.29 U	0.29 U	NA
2,4'-DDE	0.62 U	0.62 U	0.62 U	0.62 U	NA
Endosulfan I a-Chlordane	0.33 U 0.47 U	0.33 U 4.38	0.33 U 4.92	0.33 U 5.06	NA
Trans Nonachlor	0.47 U 0.21 U	4.38 3.08	4.92 3.25	3.19	8 3
4,4'-DDE	0.13 U	37.8	38.9	41.0	4
Dieldrin	0.19 U	6.31	6.21	6.52	2
2,4'-DDD	0.19 U	12.5	12.3	12.4	1
2,4'-DDT	0.22 U	0.22 U	0.22 U	0.22 U	NA
4,4'-DDD	0.24 U	13.8	14.4	14.2	2
Endosulfan II	0.33 U	0.33 U	0.33 U	0.33 U	NĂ
4,4'-DDT	0.69 U	5.15	15.4	4.69	72 ^(d)
Endosulfan Sulfate	0.33 U	0.33 U	0.33 U	0.33 U	NA
PCB 8	0.54 U	28.4	27.4	27.8	2
PCB 18	0.18 U	83.5	81.4	81.8	1
PCB 28	0.36	132	130	131	1
PCB 44	0.04 U	59.2	58.5	62.6	4
PCB 49	0.26 U	69.0	64.9	67.2	3
PCB 52	0.18 U	80.4	74.9	75.9	4
PCB 66	0.12 U	97.1	94.5	96.0	1
PCB 87	0.13 U	13.4	13.5	13.7	1
PCB 101	0.13 U	45.4	42.9	43.2	З
PCB 105	0.10 U	18.5	17.4	17.6	3
PCB 118	1.74 U	46.6	42.4	43.3	5
PCB 128	0.12 U	6.24	5.64	5.80	5
PCB 138	0.26 U	33.7	31.2	31.7	4
PCB 153	0.09 U	35.4	31.7	32.3	6
PCB 170	0.13 U	10.7	9.71	9.76	6
PCB 180	0.08 U	19.5	19.1	19.2	1
PCB 183	0.26 U	5.96	5.50	5.52	5
PCB 184	0.26 U	0.26 U	0.26 U	0.26 U	NA
PCB 187	0.06 U	13.0 2.47	12.0 2.28	11.9 2.40	5 4
PCB 195 PCB 206	0.08 U 0.05 U	2.47 6.36	2.28 4.89	2.40 5.50	13
PCB 209	0.03 U	6.61	4.03	5.36	17
	0.00 0	0.01	4.11	5.50	.,
Surrogate Recoveries (%)					
PCB 103 (SIS)	32	47	66	52	NA
PCB 198 (SIS)	23	53	91	69	NA

Table A.4. Quality Control Data (Method Blank and Triplicate Analysis) for Pesticides and PCB Congeners, Dry Weight, in Liberty Island Anchorage Sediment

(a) RSD Relative standard deviation.
(b) U Undetected at or above the given concentration.
(c) NA Not applicable.

(d) Exceeds quality control criteria (±30%) for precision.

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AnalyteCOMP LIAmountMS % RecoveryHeptachlor0.06U ^(b) NS (c)NSNSAldrin9.1910.93.6547 (a)Heptachlor Epoxide0.29UNSNSNS2,4'-DDE0.62UNSNSNSEndosulfan I0.33UNSNSNSa-Chlordane4.79NSNSNSTrans Nonachlor3.17NSNSNS4,4'-DDE39.241.23.6554Dieldrin6.358.873.65692,4'-DDD12.4NSNSNS4,4'-DDD14.116.93.6576Endosulfan II0.33U2.473.65594,4'-DDT8.418.133.65NR (a)Endosulfan Sulfate0.33UNSNSNS			N	/latrix Spike (ng/	g dry wt.)	
Heptachlor 0.06 $U^{(b)}$ NS (c)NSNSAldrin9.1910.93.6547(d)Heptachlor Epoxide0.29UNSNSNS2,4'-DDE0.62UNSNSNSEndosulfan I0.33UNSNSNSa-Chlordane4.79NSNSNSNSTrans Nonachlor3.17NSNSNS4,4'-DDE39.241.23.6554Dieldrin6.358.873.65692,4'-DDD12.4NSNSNS4,4'-DDD14.116.93.6576Endosulfan II0.33U2.473.65594,4'-DDT8.418.133.65NR (c)				•	Amount	MS %
Aldrin 9.19 10.9 3.65 47 47 Heptachlor Epoxide 0.29 U NS NS NS 2,4'-DDE 0.62 U NS NS NS Endosulfan I 0.33 U NS NS NS a-Chlordane 4.79 NS NS NS Trans Nonachlor 3.17 NS NS NS 4,4'-DDE 39.2 41.2 3.65 54 Dieldrin 6.35 8.87 3.65 69 2,4'-DDD 12.4 NS NS NS 2,4'-DDT 0.22 U NS NS NS 4,4'-DDD 14.1 16.9 3.65 76 Endosulfan II 0.33 U 2.47 3.65 59 4,4'-DDT 8.41 8.13 3.65 NR ^(e) 16 16 16 16 16 16 16 16 16 16 16 16 <t< td=""><td>Analyte</td><td>COMP LI^(a)</td><td></td><td>COMP LI, MS</td><td>Spiked</td><td>Recovery</td></t<>	Analyte	COMP LI ^(a)		COMP LI, MS	Spiked	Recovery
Aldrin 9.19 10.9 3.65 47 47 Heptachlor Epoxide 0.29 U NS NS NS 2,4'-DDE 0.62 U NS NS NS Endosulfan I 0.33 U NS NS NS a-Chlordane 4.79 NS NS NS Trans Nonachlor 3.17 NS NS NS 4,4'-DDE 39.2 41.2 3.65 54 Dieldrin 6.35 8.87 3.65 69 2,4'-DDD 12.4 NS NS NS 2,4'-DDT 0.22 U NS NS NS 4,4'-DDD 14.1 16.9 3.65 76 Endosulfan II 0.33 U 2.47 3.65 59 4,4'-DDT 8.41 8.13 3.65 NR ^(e) 16 16 16 16 16 16 16 16 16 16 16 16 <t< td=""><td>Hentachlor</td><td>0.06</td><td>1 I(p)</td><td></td><td>MQ</td><td>MQ</td></t<>	Hentachlor	0.06	1 I(p)		MQ	MQ
Heptachlor Epoxide 0.29 UNSNSNS $2,4'$ -DDE 0.62 UNSNSNSEndosulfan I 0.33 UNSNSNS a -Chlordane 4.79 NSNSNSTrans Nonachlor 3.17 NSNSNS $4,4'$ -DDE 39.2 41.2 3.65 54 Dieldrin 6.35 8.87 3.65 69 $2,4'$ -DDD 12.4 NSNSNS $2,4'$ -DDT 0.22 UNSNSNS $4,4'$ -DDD 14.1 16.9 3.65 76 Endosulfan II 0.33 U 2.47 3.65 59 $4,4'$ -DDT 8.41 8.13 3.65 NR (6)	•		0		-	
2,4'-DDE 0.62 U NS NS NS Endosulfan I 0.33 U NS NS NS a-Chlordane 4.79 NS NS NS NS Trans Nonachlor 3.17 NS NS NS 4,4'-DDE 39.2 41.2 3.65 54 Dieldrin 6.35 8.87 3.65 69 2,4'-DDD 12.4 NS NS NS 2,4'-DDT 0.22 U NS NS NS 4,4'-DDD 14.1 16.9 3.65 76 Endosulfan II 0.33 U 2.47 3.65 59 4,4'-DDT 8.41 8.13 3.65 NR ^(e)			11			
Endosulfan I0.33 UNSNSNSa-Chlordane4.79NSNSNSTrans Nonachlor3.17NSNSNS4,4'-DDE39.241.23.6554Dieldrin6.358.873.65692,4'-DDD12.4NSNSNS2,4'-DDT0.22 UNSNSNS4,4'-DDD14.116.93.6576Endosulfan II0.33 U2.473.65594,4'-DDT8.418.133.65NR						
a-Chlordane4.79NSNSNSTrans Nonachlor3.17NSNSNS4,4'-DDE39.241.23.6554Dieldrin6.358.873.65692,4'-DDD12.4NSNSNS2,4'-DDT0.22UNSNS4,4'-DDD14.116.93.6576Endosulfan II0.33U2.473.65594,4'-DDT8.418.133.65NR	•		-			
Trans Nonachlor3.17NSNSNS4,4'-DDE39.241.23.6554Dieldrin6.358.873.65692,4'-DDD12.4NSNSNS2,4'-DDT0.22UNSNS4,4'-DDD14.116.93.6576Endosulfan II0.33U2.473.65594,4'-DDT8.418.133.65NR			Ŭ			
4,4'-DDE39.241.23.6554Dieldrin6.358.873.65692,4'-DDD12.4NSNSNS2,4'-DDT0.22UNSNS4,4'-DDD14.116.93.6576Endosulfan II0.33U2.473.65594,4'-DDT8.418.133.65NR						
Dieldrin6.358.873.65692,4'-DDD12.4NSNSNS2,4'-DDT0.22UNSNSNS4,4'-DDD14.116.93.6576Endosulfan II0.33U2.473.65594,4'-DDT8.418.133.65NR						
2,4'-DDD12.4NSNSNS2,4'-DDT0.22 UNSNSNS4,4'-DDD14.116.93.6576Endosulfan II0.33 U2.473.65594,4'-DDT8.418.133.65NR (e)	•					
2,4'-DDT0.22 UNSNS4,4'-DDD14.116.93.6576Endosulfan II0.33 U2.473.65594,4'-DDT8.418.133.65NR (e)						
4,4'-DDD14.116.93.6576Endosulfan II0.33 U2.473.65594,4'-DDT8.418.133.65NR (e)			U	NS	NS	NS
4,4'-DDT 8.41 8.13 3.65 NR ^(e)	•	14.1		16.9	3.65	76
	Endosulfan II	0.33	U	2.47	3.65	59
Endosulfan Sulfate 0.33 U NS NS NS	4,4'-DDT	8.41		8.13	3.65	NR ^(e)
	Endosulfan Sulfate	0.33	U	NS	NS	NS
PCB 8 27.9 NS NS NS	PCB 8	27.9		NS	NS	NS
PCB 18 82.2 NS NS NS	PCB 18	82.2		NS	NS	
PCB 28 131 140 3.18 283 ^(d)	PCB 28	131		140	3.18	283 ^(d)
PCB 44 60.1 NS NS NS	PCB 44			NS	NS	NS
PCB 49 67.0 NS NS NS	°CB 49	67.0		NS	NS	
	°CB 52	77.1				152 ^(d)
PCB 66 95.9 NS NS NS		95.9		NS	NS	
PCB 87 13.5 NS NS NS						
						139 ^(d)
PCB 105 17.8 NS NS NS						
PCB 118 44.1 NS NS NS						
PCB 128 5.89 NS NS NS						
						184 ^(d)
PCB 170 10.1 NS NS NS						
PCB 180 19.3 NS NS NS						
PCB 183 5.66 NS NS NS						
PCB 184 0.26 U NS NS NS			0			
PCB 187 12.3 NS NS NS PCB 195 2.38 NS NS NS						
PCB 195 2.38 NS NS NS PCB 206 5.58 NS NS NS						
PCB 200 5.58 NS NS NS NS						
		5.56		110		NO
Surrogate Recoveries (%)						
PCB 103 (SIS) NA ⁽¹⁾ 50 NA NA			(t)			
PCB 198 (SIS) NA S5 NA NA	PCB 198 (SIS)	NA		55	NA	NA

<u>Table A.5.</u> Quality Control Data (Recovery of Matrix Spike) for Pesticides and PCB Congeners, Dry Weight, in Liberty Island Anchorage Sediment

(a) Mean of replicated sample.
(b) U Undetected at or above the given concentration.
(c) NS Not spiked.
(d) Outside quality control criteria (50% - 120%) for matrix spike recoveries.

	Standard	ial (ng/g)	
	SRM	Certified	SRM %
Analyte	1941A	value	Recovery
Heptachlor	0.05 U ^(a)	NA ^(b)	NA
Aldrin	0.18 U	NA	NA
Heptachlor Epoxide	0.26 U	NA	NA
2,4'-DDE	0.56 U	0.73	NA
Endosulfan I	1.31	NA	NA
a-Chlordane	1.98	2.33	85
Trans Nonachlor	0.19 U	NA	NA
4,4'-DDE	7.63	6.59	116
Dieldrin	0.17 U	NA	NA
2,4'-DDD	0.17 U	NA	NA
2,4'-DDT	0.20 U	NA	NA
4,4'-DDD	4.73	5.06	93
Endosulfan II	0.30 U	NA	NA
4,4'-DDT	2.44	NA	NA
Endosulfan Sulfate	0.30 U	NA	NA
PCB 8	6.47	NA	NA
PCB 18	5.41	NA	NA
PCB 28	0.10 U	NA	NA
PCB 44	5.35	4.80	111
PCB 49	7.02	9.50	74
PCB 52	9.05	6.89	131 ^(c)
PCB 66	8.51	6.80	125
PCB 87	4.61	6.70	69 ^(c)
PCB 101	12.0	11.0	109
PCB 105	3.61	3.65	99
PCB 118	9.55	10.0	96
PCB 128	1.34	1.87	72
PCB 138	9.02	13.4	67 ^(c)
PCB 153	12.1	17.6	69 ^(c)
PCB 170	16.9	3.00	563 ^(c)
PCB 180	10.1	5.38	188 ^(c)
PCB 183	2.11	NA	NA
PCB 184	0.24 U	NA	NA
PCB 187	5.55	NA	NA
PCB 195	1.39	NA	NA
PCB 206	4.49	3.67	122
PCB 209	10.6	8.34	127
Surrogate Recoveries (%)			
PCB 103 (SIS)	40	NA	NA
PCB 198 (SIS)	25	NA	NA

Table A.6. Quality Control Data (Standard Reference Material) for Pesticides and PCB Congeners, Dry Weight, in Liberty Island Anchorage Sediment

(a) U Undetected at or above the given concentration.(b) NA Not applicable.(c) Outside quality control criteria (±30%) for SRMs.

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<u>TABLE A.7</u>. Quality Control Data (Analytical Replicates) for Polynuclear Aromatic Hydrocarbon (PAH), Dry Weight, in Liberty Island Anchorage Sediment

Analytical Replicates (ng/g dry wt.)					
Method	COMP LI	COMP LI	COMP LI	Percent	
Blank	REP 1	REP 2	REP 3	RSD ^(a)	
		· · · · · · · · · · · · · · · · · · ·		······································	
6.38 ^(b)	397	372	370	4	
				3	
				3	
				3	
				1	
				2	
2.13 U	1550	1550	1620	3	
	1520	1540	1600	3	
0.77 U	698	715	749	4	
0.85 U	787	795	809	1	
1.62 U	994	949	983	2	
2.74 U	359	331	339	4	
2.14 U	735	719	744	2	
0.98 U	457	449	447	1	
1.24 U	115	112	114	1	
0.90 U	393	389	390	1	
2.06 U ^(c)	208	215	190	6	
16 ^(a)	19 ^(a)	32	25 ^(a)	NA	
18 ^(d)		41	34	NA	
16 ^(a)	36	51	43	NA	
22 ^(a)	37	53	43	NA	
20 ^(a)	40	58	48	NA	
	6.38 ^(b) 2.19 U ^(c) 1.96 U 2.91 U 9.23 ^(b) 5.62 U 2.13 U 9.45 ^(b) 0.77 U 0.85 U 1.62 U 2.74 U 2.14 U 0.98 U 1.24 U 0.90 U 2.06 U ^(c)	MethodCOMP LI BlankBlankREP 1 $6.38^{(v)}$ 397 $2.19 U^{(v)}$ 69.7 $1.96 U$ 238 $2.91 U$ 284 $9.23^{(v)}$ 1040 $5.62 U$ 437 $2.13 U$ 1550 $9.45^{(v)}$ 1520 $0.77 U$ 698 $0.85 U$ 787 $1.62 U$ 994 $2.74 U$ 359 $2.14 U$ 735 $0.98 U$ 457 $1.24 U$ 115 $0.90 U$ 393 $2.06 U^{(v)}$ 20816 (a)19 (a)16 (a)3622 (a)37	MethodCOMP LI REP 1COMP LI REP 2BlankREP 1REP 2 $6.38^{(v)}$ 397372 $2.19 U^{(v)}$ 69.768.5 $1.96 U$ 238227 $2.91 U$ 284271 $9.23^{(v)}$ 10401020 $5.62 U$ 437423 $2.13 U$ 15501550 $9.45^{(v)}$ 15201540 $0.77 U$ 698715 $0.85 U$ 787795 $1.62 U$ 994949 $2.74 U$ 359331 $2.14 U$ 735719 $0.98 U$ 457449 $1.24 U$ 115112 $0.90 U$ 393389 $2.06 U^{(v)}$ 208215 $16^{(a)}$ 19^{(a)}32 $18^{(a)}$ 27^{(a)}41 $16^{(a)}$ 3651 $22^{(a)}$ 3753	Method BlankCOMP LI REP 1COMP LI REP 2COMP LI REP 3 $6.38^{(v)}$ 397 372 370 $2.19 U^{(c)}$ 69.7 68.5 65.3 $1.96 U$ 238 227 238 $2.91 U$ 284 271 271 $9.23^{(v)}$ 1040 1020 1040 $5.62 U$ 437 423 429 $2.13 U$ 1550 1550 1620 $9.45^{(v)}$ 1520 1540 1600 $0.77 U$ 698 715 749 $0.85 U$ 787 $.795$ 809 $1.62 U$ 994 949 983 $2.74 U$ 359 331 339 $2.14 U$ 735 719 744 $0.98 U$ 457 449 447 $1.24 U$ 115 112 114 $0.90 U$ 393 389 390 $2.06 U^{(c)}$ 208 215 190	

(a) Relative standard deviation.

(b) Ion ratio out or confirmation ion not detected.

(c) Undetected at or above the given concentration.

(d) Outside SIS recovery range (30-150%).

TABLE A.8 Quality Control Data (Recovery of Matrix Spike) for Polynuclear Aromatic Hydrocarbons (PAH), Dry Weight, in Liberty Island Anchorage Sediment

	Matrix Spike (ng/g dry wt.)				
A			Amount	MS %	
Analyte	COMP LI ^(a)	COMP LI, MS	Spiked	Recovery	
Naphthalene	380	437	36.6	157 ^(a)	
Acenaphthylene	68	104	36.6	99	
Acenaphthene	234	279	36.6	122 ^(a)	
Fluorene	275	339	36.6	174	
Phenanthrene	1033	1070	36.6	100	
Anthracene	430	528	36.6	269 ^(a)	
Fluoranthene	1573	1830	36.6	701 ^(a)	
Pyrene	1553	1770	36.6	592 ^(a)	
Benz(a)anthracene	721	926	36.6	561 ^(a)	
Chrysene	797	1080	· 36.6	773 ^(a)	
Benzo(b)fluoranthene	975	1170	36.6	532 ^(a)	
Benzo(k)fluoranthene	343	466	36.6	336 ^(a)	
Benzo(a)pyrene	733	869	36.6	372 ^(a)	
Indeno(123-cd)pyrene	451	520	36.6	189 ^(a)	
Dibenz(a,h)anthracene	114	157	36.6	118	
Benzo(g,h,i)perylene	391	445	36.6	148 ^(a)	
1,4-Dichlorobenzene	204	256	36.6	141 ^(a)	
Surrogate Internal Standards (%)					
d4 1,4-Dichlorobenzene	NA ^(D)	25 ^(c)	NA	NA	
d8 Naphthalene	NA	34	NA	NA	
d10 Acenaphthene	NA	41	NA	NA	
d12 Chrysene	NA	39	NA	NA	
d14 Dibenz(a,h,i)Anthracene	NA	44	NA	NA	

(a) Mean of replicate sample.(b) Outside quality control criteria (50%-120%) for matrix spike recoveries.

(c) NA Not applicable.(d) Outside SIS recovery range (30-150%).

	Standard Reference Material (ng/g dry wt.					
	SRM	Certified	Percent			
Analyte	1941A	value	Difference			
Naphthalene	910	1010	10%			
Acenaphthylene	46.2	NC ^(a)	NA ^(D)			
Acenaphthene	44.1	NC	NA			
Fluorene	89.5	97.3	8%			
Phenanthrene	569	489	16%			
Anthracene	212	184	15%			
Fluoranthene	895	981	9%			
Pyrene .	683	811	16%			
Benz(a)anthracene	376	427	12%			
Chrysene	560	380	47% ^(c)			
Benzo(b)fluoranthene	1220	740	65% ^(c)			
Benzo(k)fluoranthene	465	361	29% ^(c)			
Benzo(a)pyrene	540	628	14%			
Indeno(123-cd)pyrene	459	501	8%			
Dibenz(a,h)anthracene	102	74	38% ^(c)			
Benzo(g,h,i)perylene	352	525	33% ^(c)			
1,4-Dichlorobenzene	107	NC ^(a)	NA			
Surrogate Internal Standards (%)						
d4 1,4-Dichlorobenzene	17 ^(a)	NA	NA			
d8 Naphthalene	19 ^(a)	NA	NA			
d10 Acenaphthene	22 ^(a)	NA	NA			
d12 Chrysene	28 ^(a)	NA	NA			
d14 Dibenz(a,h,i)Anthracene	26 ^(a)	NA	NA			

TABLE A.9. Quality Control Data (Standard Reference Material) for Polynuclear Aromatic Hydrocarbons (PAH), Dry Weight, in Liberty Island Anchorage Sediment

(a) Not certified.

(b) NA Not applicable.

(b) Outside quality control criteria (≥30%) for SRMs.
(c) Outside SIS recovery range (30-150%).

State .

	Conc. Found	Conc. Found	lon
Analyte	(pg/g wet)	(pg/g dry)	Ratio
<u>COMP LI, Rep. 1(a)</u>			
2378-TCDD ** ^(b)	13.4	30.6	0.78
12378-PeCDD	0.8 U ^(c)	1.9 U	NA ^(d)
123478-HxCDD	2.50	5.68	1.16
123678-HxCDD	13.6	30.9	1.23
123789-HxCDD	9.83	22.3	1.07
1234678-HpCDD	315	716	1.04
OCDD	2434	5532	0.89
2378-TCDF **	14.1	32.1	0.74
12378-PeCDF	4.09	9.30	1.57
23478-PeCDF	5.57	12.7	1.56
123478-HxCDF	16.1	36.7	1.32
123678-HxCDF	2.99	6.79	1.31
123789-HxCDF	0.9 U	2.1 U	NA
234678-HxCDF	2.5	5.58	1.34
1234678-HpCDF	57.3	130	1.01
1234789-HpCDF '	3.6	8.24	0.91
OCDF	154	349	0.87
COMP LI, Rep. 2			
2378-TCDD **	11.1	25.2	0.89
12378-PeCDD	0.05 U	0.12 U	NA
123478-HxCDD	1.52	3.44	1.3
123678-HxCDD	10.5	23.8	1.26
123789-HxCDD	7.46	17.0	1.19
1234678-HpCDD	177	402	1.04
OCDD	1310	2977	0.88
2378-TCDF **	7.92	18.0	0.72
12378-PeCDF	2.29	5.22	1.55
23478-PeCDF	2.76	6.28	1.71
123478-HxCDF	12.9	29.3	1.26
123678-HxCDF	2.51	5.70	1.18
123789-HxCDF	0.08 U	0.19 U	NA
234678-HxCDF	1.77	4.03	1.26
1234678-HpCDF	42.7	97.2	1.03
1234789-HpCDF	2.47	5.61	0.99
OCDF	80.3	182	0.87

TABLE A.10. Dioxin and Furan Congeners in Liberty Island Anchorage Sediment

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Analyte	Conc. Found (pg/g wet)	Conc. Found (pg/g dry)	Ion Ratio	
COMP LI, Rep. 3-1				
2378-TCDD **	8.43	19.2	0.76	
12378-PeCDD	0.3 U	0.8 U	NA	
123478-HxCDD	1.63	3.71	1.41	
123678-HxCDD	11.1	25.2	1.22	
123789-HxCDD	8.03	18.3	1.07	
1234678-HpCDD	169	385	1.03	
OCDD	1236	2809	0.88	
2378-TCDF **	7.42	16.87	0.71	
12378-PeCDF	1.99	4.52	1.73	
23478-PeCDF	3.20	7.27	1.64	
123478-HxCDF	10.1	23.02	1.18	
123678-HxCDF	2.45	5.58	1.22	
123789-HxCDF	0.2 U	0.5 U	NA	
234678-HxCDF	1.55	3.53	1.29	
1234678-HpCDF	41.6	94.5	1.03	
1234789-HpCDF	2.32	5.28	0.97	
OCDF	83.4	190	0.87	
COMP LI Rep. 3-2				
2378-TCDD **	12.7	28.9	0.7	
12378-PeCDD	0.2 U	0.54 U	NA	
123478-HxCDD	1.97	4.48	1.29	
123678-HxCDD	13.7	31.2	1.22	
123789-HxCDD	9.78	22.2	1.09	
1234678-HpCDD	214	485	1.04	
OCDD	1597	3629	0.89	
2378-TCDF **	8.90	20.2	0.71	
12378-PeCDF	2.54	5.78	1.57	
23478-PeCDF	3.40	7.72	1.53	
123478-HxCDF	14.8	33.6	1.25	
123678-HxCDF	3.23	7.35	1.14	
123789-HxCDF	0.2 U	0.43 U	NA	
234678-HxCDF	2.90	6.60	1.19	
1234678-HpCDF	52.7	120	1.02	
1234789-HpCDF	3.12	7.08	1.11	
OCDF	102	233	0.89	

A.12

Analyte	Conc. Found (pg/g wet)	Conc. Found (pg/g dry)	lon Ratio
COMP LI, Rep. 3-3			
2378-TCDD **	11.6	26.3	0.84
12378-PeCDD	0.2 U	0.54 U	NA
123478-HxCDD	2.11	4.80	1.21
123678-HxCDD	15.2	34.5	1.22
123789-HxCDD	10.0	22.8	1.26
1234678-HpCDD	223	507	1.05
OCDD	1608	3654	0.88
2378-TCDF **	9.66	21.9	0.75
12378-PeCDF	3.02	6.87	1.75
23478-PeCDF	3.88	8.82	1.74
123478-HxCDF	12.6	28.6	1.25
123678-HxCDF	5.12	11.6	1.24
123789-HxCDF	0.2 U	0.4 U	NA
234678-HxCDF	3.00	6.81	1.26
1234678-HpCDF	54.4	124	1.03
1234789-HpCDF	0.4 U	0.85 U	NA
OCDF	106	241	0.885

(a) Sample diluted 4 times with decane.

(b) ** Value from second column confirmation analysis.

(c) U Undetected at or above concentration shown.

(d) NA Not applicable.

A.13

Analyte	Method Blank Conc. Found (pg/g dry wt.)	ion Ratio
2378-TCDD	0.1 U ^(a)	NA ^(b)
12378-PeCDD	0.2 U	NA
123478-HxCDD	0.5 U	NA
123678-HxCDD	0.4 U	NA
123789-HxCDD	0.3 U	NA
1234678-HpCDD	0.53	1.09
OCDD	3.60	0.87
2378-TCDF	0.2 U	NA
12378-PeCDF	0.2 U	NA
23478-PeCDF	0.2 U	NA
123478-HxCDF	0.45	1.37
123678-HxCDF	0.4 U	NA
123789-HxCDF	0.4 U	NA
234678-HxCDF	0.34	1.14
1234678-HpCDF	0.41	1.16
1234789-HpCDF	0.6 U	NA
OCDF	1.27	0.81

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TABLE A.11. Quality Control Data (Method Blank) for Dioxin and Furan Congeners in Liberty Island Anchorage Sediment

(a) U Undetected at or above concentration shown.

(b) NA Not applicable.

	i ulan oongene		isianu Anonora	age Seument	
				Corrected	MS
	Conc. Found	Spike	Conc. Found	Conc.	Percent
	COMP LI	Conc.	COMP LI, MS	Found	Recovery
Analyte	(pg/g dry wt.)	(pg/g dry wt.)	(pg/g dry wt.)	(pg/g dry wt.)	(%)
			-		
2378-TCDD ** ^(a)	25.2	28.7	69.8	44.6	155 ^{• (b)}
12378-PeCDD	0.1 U ^(c)	144	147	147	102
123478-HxCDD	3.44	144	142	139	97
123678-HxCDD	23.8	144	161	137	96
123789-HxCDD	17.0	144	179	162	113
1234678-HpCDD	402	144	637	235	164 *
OCDD	2977	287	3935	958	333 *
2378-TCDF **	18.0	28.7	46.7	28.7	100
12378-PeCDF	5.22	144	149	144	100
23478-PeCDF	6.28	144	130	123	86
123478-HxCDF	29.3	144	172	143	99
123678-HxCDF	5.70	144	151	145	101
123789-HxCDF	0.2 U	144	134	134	94
234678-HxCDF	4.03	144	· 143	139	97
1234678-HpCDF	97.2	144	245	148	103
1234789-HpCDF	5.61	144	150	144	101
OCDF	182	287	479	296	103

TABLE A.12. Quality Control Data (Recovery of Matrix Spike) for Dioxin and Furan Congeners in Liberty Island Anchorage Sediment

(a) ** Values from second column confirmation.
(b) * Outside data quality limit of 50-120% recovery.
(c) U Undetected at or above concentration shown.

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Analyte	Target Value (pg/g dry wt.)	Conc. Found (pg/g dry wt.)	Percent Recovery (%)	Percent Difference (%)
0070 TODD	500		100	
2378-TCDD	500	511	102	2.2
12378-PeCDD	1000	1015	102	1.5
123478-HxCDD	1000	945	94	5.5
123678-HxCDD	1000	947	95	5.3
123789-HxCDD	1000	1110	111	11
1234678-HpCDD	1500	1428	95	4.8
OCDD	3500	2908	83	17
2378-TCDF	500	493	99	1.3
12378-PeCDF	1000	932	93	6.8
23478-PeCDF	1000	876	88	12
123478-HxCDF	1000	938	94	6.2
123678-HxCDF	1000	965	97	3.5
123789-HxCDF	1000	902	90	9.8
234678-HxCDF	1000	922	92	7.8
1234678-HpCDF	1500	1327	88	12
1234789-HpCDF	1500	1435	96	4.3
OCDF	2500	2061	82	18

TABLE A.13. Quality Control Data (Standard Reference Material) for Dioxin and Furan Congeners in Liberty Island Anchorage Sediment

Concentration (pg/g dry wt.)						
Analyte	COMP LI, Rep 3 Rep 1	COMP LI, Rep 3 Rep 2	COMP LI, Rep 3 Rep 3	Mean		Percent RSD ^(a)
*				moun		
2378-TCDD ** ^(b)	19.2	28.9	26.3	24.8	5.0	20.3
12378-PeCDD	0.8 U ^(c)	0.5 U	0.5 U	0.3 U	NA ^(d)	NA
123478-HxCDD	3.71	4.48	4.80	4.33	0.6	13.0
123678-HxCDD	25.2	31.2	34.5	30.3	4.8	15.7
123789-HxCDD	18.3	22.2	22.8	21.1	2.5	11.7
1234678-HpCDD	385	485	507	459	65.0	14.2
OCDD	2809	3629	3654	3364	481	14.3
2378-TCDF **	16.9	20.2	21.9	19.7	2.6	13.1
12378-PeCDF	4.52 .	5.78	6.87	5.72	1.2	20.6
23478-PeCDF	7.27	7.72	8.82	7.94	0.8	10.1
123478-HxCDF	23.0	33.6	28.6	28.4	5.3	18.7
123678-HxCDF	5.58	7.35	11.6	8.19	3.1	38.1 * ^(e)
123789-HxCDF	0.5 U	0.4 U	0.4 U	0.2 U	NA	NA
234678-HxCDF	3.53	6.60	6.81	5.65	1.8	32.5 *
1234678-HpCDF	94.5	120	124	113	15.8	14.1
1234789-HpCDF	5.28	7.08	0.9 U	4.27	3.43	80.3 *
OCDF	190	233	241	221	27.6	12.5

TABLE A.14. Quality Control Data (Triplicate Analysis) for Dioxin and Furan Congeners in Liberty Island Anchorage Sediment

(a) RSD Relative standard deviation.

(b) ** Values from second column confirmation.

(c) U Undetected at or above concentration shown.

(d) NA Not applicable.

(e) * Results are outside data quality limit of <25% RSD for this analyte.

	Spike Conc.	Conc. Found	Percent Recovery	lon
Labeled Compounds	(pg/g dry wt.)	(pg/g dry wt.)	(%)	Ratio
Method Blank				
2378-TCDD-13C12	200	200	100	0.80
12378-PeCDD-13C12	200	203	102	1.55
123478-HxCDD-13C12	200	213	107	1.25
123678-HxCDD-13C12	200	170	85	1.26
1234678-HpCDD-13C12	200	185	93	1.05
OCDD-13C12	400	382	96	0.89
2378-TCDF-13C12	200	187	94	0.79
12378-PeCDF-13C12	200	199	100	1.57
23478-PeCDF-13C12	200	· 240	120	1.56
123478-HxCDF-13C12	200	176	88	0.52
123678-HxCDF-13C12	200	167	84	0.52
123789-HxCDF-13C12	200	187	94	0.51
234678-HxCDF-13C12	200	176	88	0.52
1234678-HpCDF-13C12	200	188	94	0.44
1234789-HpCDF-13C12	200	187	94	0.45
CLEANUP STANDARD				
2378-TCDD-37Cl4	80.0	85.0	106	NA ^(a)
2010 1022 01014	00.0	00.0	100	INA.
COMP LI, Rep. 1				
2378-TCDD-13C12	280	270	96	0.80
12378-PeCDD-13C12	280	348	124	1.57
123478-HxCDD-13C12	280	211	75	1.26
123678-HxCDD-13C12	280	171	61	1.27
1234678-HpCDD-13C12	280	233	83	1.05
OCDD-13C12	559	502	90	0.89
2378-TCDF-13C12	280	344	123	0.76
12378-PeCDF-13C12	280	308	110	1.53
23478-PeCDF-13C12	280	283	101	1.61
123478-HxCDF-13C12	280	167	60	0.54
123678-HxCDF-13C12	280	153	55	0.51
123789-HxCDF-13C12	280	239	86	0.52
234678-HxCDF-13C12	280	181	65	0.51
1234678-HpCDF-13C12	280	200	72	0.44
1234789-HpCDF-13C12	280	232	83	0.43
CLEANUP STANDARD				
2378-TCDD-37Cl4	112	132	118	NA

TABLE A.15. Quality Control Data (Recovery of Labeled Compounds) for Dioxin and Furan Congeners in Liberty Island Anchorage Sediment

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	Spike Conc.	Conc. Found	Percent Recovery	lon
Labeled Compounds	(pg/g dry wt.)	(pg/g dry wt.)	(%)	Ratio
COMPLE Den 0				
<u>COMP LI, Rep. 2</u> 2378-TCDD-13C12	287	309	100	0 70
12378-PeCDD-13C12	287	309 344	108	0.79
123478-HxCDD-13C12	287	344 254	120	1.55
123678-HxCDD-13C12	287 287	254 222	88	1.24
			77	1.26
1234678-HpCDD-13C12	287	237	82	1.03
OCDD-13C12	575	423	74	0.89
2378-TCDF-13C12	287	283	99	0.79
12378-PeCDF-13C12	287	319	111	1.58
23478-PeCDF-13C12	287	312	108	1.57
123478-HxCDF-13C12	287	244	85	0.52
123678-HxCDF-13C12	287	193	67	0.52
123789-HxCDF-13C12	287	277	96	0.52
234678-HxCDF-13C12	287	232	81	0.52
1234678-HpCDF-13C12	287	215	75	0.45
1234789-HpCDF-13C12	287	226	79	0.44
• • • •				
CLEANUP STANDARD				
2378-TCDD-37Cl4	115 [.]	96	83	NA
COMP LI, Rep. 2, Matrix Sp	ike			
2378-TCDD-13C12	287	311	108	0.79
12378-PeCDD-13C12	287	348	121	1.55
123478-HxCDD-13C12	287	277	96	1.25
123678-HxCDD-13C12	287	193	67	1.26
1234678-HpCDD-13C12	287	246	86	1.05
OCDD-13C12	575	428	74	0.90
2378-TCDF-13C12	287	252	88	0.73
12378-PeCDF-13C12	287	318	111	1.57
23478-PeCDF-13C12	287	306	106	1.55
123478-HxCDF-13C12	287	282	98	0.54
123678-HxCDF-13C12	287	168	58	0.49
123789-HxCDF-13C12	287	281	98	0.52
234678-HxCDF-13C12	287 -	204	71	0.52
1234678-HpCDF-13C12	287	215	75	0.45
1234789-HpCDF-13C12	287	235	82	0.44
CLEANUP STANDARD				
2378-TCDD-37Cl4	115	120	105	NA
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	Spike Conc.	Conc. Found	Percent Recovery	lon
Labeled Compounds	(pg/g dry wt.)	(pg/g dry wt.)	(%)	Ratio
COMP LI, Rep. 3-1				
2378-TCDD-13C12	277	263	95	0.80
12378-PeCDD-13C12	277	267	96	1.54
123478-HxCDD-13C12	277	211	76	1.27
123678-HxCDD-13C12	277	180	65	1.26
1234678-HpCDD-13C12	277	234	85	1.05
OCDD-13C12	554	469	85	0.89
2378-TCDF-13C12	277	258	93	0.80
12378-PeCDF-13C12	277	275	99	1.57
23478-PeCDF-13C12	277	256	92	1.56
123478-HxCDF-13C12	277	200	72	0.54
123678-HxCDF-13C12	277	174	63	0.51
123789-HxCDF-13C12	277	253	91	0.52
234678-HxCDF-13C12	277	202	73	0.52
1234678-HpCDF-13C12	277	214	77	0.45
1234789-HpCDF-13C12	277	243	88	0.44
CLEANUP STANDARD				
2378-TCDD-37Cl4	111	112	101	NA
COMP LI Rep. 3-2	076	200	100	0.00
2378-TCDD-13C12 12378-PeCDD-13C12	276 276	300 331	109 120	0.80
				1.56
123478-HxCDD-13C12	276	243	88	1.26
123678-HxCDD-13C12	276	210	76	1.26
1234678-HpCDD-13C12	276	249	90	1.05
OCDD-13C12	552	469	85	0.89
2378-TCDF-13C12	276	286	104	0.74
12378-PeCDF-13C12	276	325	118	1.57
23478-PeCDF-13C12	276	304	110	1.57
123478-HxCDF-13C12	276	232	84	0.52
123678-HxCDF-13C12	276	186	67	0.52
123789-HxCDF-13C12	276	276	100	0.51
234678-HxCDF-13C12	276	224	81	0.52
1234678-HpCDF-13C12	276	224	81	0.45
1234789-HpCDF-13C12	276	247	90	0.44
CLEANUP STANDARD				
2378-TCDD-37Cl4	110	96.9	88	NA

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Labeled Compounds	Spike Conc. (pg/g dry wt.)	Conc. Found (pg/g dry wt.)	Percent Recovery (%)	Ion Ratio
				,
COMP LI, Rep. 3-3				
2378-TCDD-13C12	276	249	90	0.80
12378-PeCDD-13C12	276	267	97	1.55
123478-HxCDD-13C12	276	204	74	1.26
123678-HxCDD-13C12	276	173	63	1.25
1234678-HpCDD-13C12	276	201	73	1.05
OCDD-13C12	552	372	67	0.91
2378-TCDF-13C12	276	265	96	0.78
12378-PeCDF-13C12	276	268	97	1.57
23478-PeCDF-13C12	276	252	91	1.56
123478-HxCDF-13C12	276	208	75	0.52
123678-HxCDF-13C12	276	146	53	0.52
123789-HxCDF-13C12	276	230	83	0.52
234678-HxCDF-13C12	276	187	68	0.52
1234678-HpCDF-13C12	276	183	66	0.45
1234789-HpCDF-13C12	276	202	73	0.44
CLEANUP STANDARD				
2378-TCDD-37Cl4	110	108	98	NA
		100	00	i Wi Y
Standard Reference Materia	al			
2378-TCDD-13C12	200	227	113	0.79
12378-PeCDD-13C12	200	246	123	1.59
123478-HxCDD-13C12	200	188	94	1.26
123678-HxCDD-13C12	200	151	75	1.26
1234678-HpCDD-13C12	200	215	107	1.05
OCDD-13C12	400	433	108	0.89
2378-TCDF-13C12	000	192	96	0.70
12378-PeCDF-13C12	200 200	222	90 111	0.79 1.55
23478-PeCDF-13C12	200	273	136 ^(b)	1.55
123478-HxCDF-13C12	200	161	80 71	0.52
123678-HxCDF-13C12	200	142	71	0.53
123789-HxCDF-13C12	200	215	108	0.52
234678-HxCDF-13C12	200	169	84 07	0.52
1234678-HpCDF-13C12	200	194	97	0.45
1234789-HpCDF-13C12	200	222	111	0.45
CLEANUP STANDARD				
2378-TCDD-37Cl4	80	89	111	NA
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	Spike Conc.	Conc. Found	Percent	1
Labeled Compounds	(pg/g dry wt.)	(pg/g dry wt.)	Recovery (%)	Ion Ratio
MDL-1	-			-
2378-TCDD-13C12	277	326	118	0.79
12378-PeCDD-13C12	277	301	109	1.55
123478-HxCDD-13C12	277	261	94	1.24
123678-HxCDD-13C12	277	222	80	1.25
1234678-HpCDD-13C12	277	255	92	1.03
OCDD-13C12	555	523	94	0.89
2378-TCDF-13C12	277	286	103	0.79
12378-PeCDF-13C12	277	304	110	1.55
23478-PeCDF-13C12	277	287	103	1.59
123478-HxCDF-13C12	277	228	82	0.52
123678-HxCDF-13C12	277	188	68	0.52
123789-HxCDF-13C12	277	276	100	0.51
234678-HxCDF-13C12	277	228	82	0.52
1234678-HpCDF-13C12	277	232	84	0.45
1234789-HpCDF-13C12	277	252	91	0.45
CLEANUP STANDARD				
2378-TCDD-37Cl4	111	121	109	NA
<u>MDL-2</u>				
2378-TCDD-13C12	278	263	95	0.79
12378-PeCDD-13C12	278	280	101	1.57
123478-HxCDD-13C12	278	220	79	1.24
123678-HxCDD-13C12	278	179	64	1.26
1234678-HpCDD-13C12	278	209	75	1.05
OCDD-13C12	556	407	73	0.90
2378-TCDF-13C12	278	246	88	0.74
12378-PeCDF-13C12	278	263	94	1.58
23478-PeCDF-13C12	278	246	88	1.56
123478-HxCDF-13C12	278	190	68	0.51
123678-HxCDF-13C12	278	154	55	0.52
123789-HxCDF-13C12	278	226	81	0.52
234678-HxCDF-13C12	278	186	67	0.52
1234678-HpCDF-13C12	278	182	65	0.45
1234789-HpCDF-13C12	278	198	71	0.44
CLEANUP STANDARD				
2378-TCDD-37Cl4	111	129	116	NA

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TABLE A.15.	(Contd)
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	Spike	Conc.	Percent	
	Conc.	Found	Recovery	lon
Labeled Compounds	(pg/g dry wt.)	(pg/g dry wt.)	(%)	Ratio
<u>MDL-3</u>				
2378-TCDD-13C12	279	306	110	0.79
12378-PeCDD-13C12	279	314	113	1.54
123478-HxCDD-13C12	279	254	91	1.25
123678-HxCDD-13C12	279	210	75	1.27
1234678-HpCDD-13C12	279	242	87	1.04
OCDD-13C12	558	471	84	0.90
			•	0.00
2378-TCDF-13C12	279	283	101	0.80
12378-PeCDF-13C12	279	302	108	1.56
23478-PeCDF-13C12	279	282 /	101	1.56
123478-HxCDF-13C12	279	223	80	0.52
123678-HxCDF-13C12	279	176	63	0.52
123789-HxCDF-13C12	279	267	96	0.50
234678-HxCDF-13C12	279	215	77	0.50
1234678-HpCDF-13C12	279	217	78	0.45
1234789-HpCDF-13C12	279	236	85	0.44
CLEANUP STANDARD				
2378-TCDD-37Cl4	112	130	116	NA
<u>MDL-4</u>				
2378-TCDD-13C12	278	295	106	0.79
12378-PeCDD-13C12	278	322	116	1.55
123478-HxCDD-13C12	278	254	91	1.26
123678-HxCDD-13C12	278	188	68	1.27
1234678-HpCDD-13C12	278	235	84	1.05
OCDD-13C12	556	441	79	0.91
2378-TCDF-13C12	278	272	98	0.75
12378-PeCDF-13C12	278	300	108	1.58
23478-PeCDF-13C12	278	285	102	1.57
123478-HxCDF-13C12	278	237	85	0.51
123678-HxCDF-13C12	278	168	60	0.52
123789-HxCDF-13C12	278	257	93	0.52
234678-HxCDF-13C12	278	212	76	0.52
1234678-HpCDF-13C12	278	201	72	0.44
1234789-HpCDF-13C12	278	229	82	0.45
CLEANUP STANDARD				
2378-TCDD-37Cl4	111	129	116	NA
(a) NA Not applicable.				

(a) NA NOT applicable.(b) Outside data quality limit of 40-135% recovery.

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		Co	oncentratio	on (pg/g d	ry wt.)					Average	
Analyte	Target Spike Level	Background	Spike 1	Spike 2	Spike 3	Spike 4	Spike Average	Standard Deviation	Percent RSD ^(a)	Percent Recovery ^(b)	MDL ^(c)
2378-TCDD	7.0	30.6	34.8	41.8	39.7	40.2	39.1	3.0	7.7	104.4	13.7
12378-PeCDD	34.8	1.9 ^(d)	42.9	41.4	42.7	42.1	42.3	0.7	1.6	121.6	3.1
123478-HxCDD	34.8	5.7	39.7	39.9	39.6	36.5	38.9	1.6	4.2	96.2	7.5
123678-HxCDD	34.8	30.9	57.6	63.0	62.1	68.9	62.9	4.7	7.4	95.8	21.2
123789-HxCDD	34.8	22.3	53.7	58.8	57.5	61.4	57.8	3.2	5.5	101.3	14.5
1234678-HpCDD	34.8	715.8	408.6	490.5	459.4	483.1	460.4	37.0	8.0	61.3	167.9
OCDD	69.5	5531.9	2724.4	3546.6	3111.9	3386.9	3192.4	360.0	11	57.0	1634.3
2378-TCDF	7.0	96.4	91.0	109.4	108.3	107.7	104.1	8.7	8.4	100.7	39.7
12378-PeCDF	34.8	9.3	40.6	39.4	39.2	38.9	39.5	0.8	2.0	89.7	3.5
23478-PeCDF	34.8	12.7	38.4	42.2	41.3	36.8	39.7	2.5	6.3	83.7	11.4
123478-HxCDF	34.8	36.7	66.1	69.4	66.9	66.6	67.2	1.5	2.2	94.1	6.7
123678-HxCDF	34.8	6.8	41.5	40.9	41.6	51.3	43.8	5.0	11	105.5	22.8
123789-HxCDF	34.8	2.1 U	36.4	36.0	35.7	36.5	36.2	0.4	1.0	104.1	1.7
234678-HxCDF	34.8	5.6	40.5	41.5	41.6	39.9	40.9	0.8	1.9	101.3	3.6
1234678-HpCDF	34.8	130.2	125.5	143.0	133.4	141.7	135.9	8.1	6.0	82.4	36.9
1234789-HpCDF	34.8	8.2	42.7	44.9	44.1	44.4	44.0	0.9	2.1	102.4	4.3
OCDF	69.5	349.4	233.9	276.8	262.4	285.1	264.6	22.5	8.5	63.2	102.1

<u>TABLE A.16</u>. Quality Control Data (Method Detection Limit Verification) for Dioxin and Furan Congeners in Liberty Island Anchorage Sediment

(a) RSD Relative standard deviation.

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(b) Average % recovery is determined by [spike average/(target level + background level)] x 100.

(c) Method detection limit is calculated as 4.54 x standard deviation.

(d) U Undetected at or above concentration shown.

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Appendix B.

Sec. 1 Sec. 2

Quality Assurance/Quality Control Data for Water and Elutriate Analyses, Liberty Island Anchorage ~

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QA/QC SUMMARY

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PROGRAM: New York/New Jersey Federal Projects 3

PARAMETER: Metals

LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington

MATRIX: Water

QA/QC DATA QUALITY OBJECTIVES

	Reference	Range of	SRM	Relative	Detection
	Method	Recovery	<u>Accuracy</u>	Precision	Limit (µg/L)
Cadmium	ICP/MS	75-125%	≤20%	≤20%	0.025
Chromium	GFAA	75-125%	≤20%	≤20%	
Copper	ICP/MS	75-125%	≤20%	≤20%	1.0 0.35
Lead	ICP/MS	75-125%	≤20%	≤20%	0.35
Mercury	CVAA	75-125%	≤20%	≤20%	0.002
Nickel	ICP/MS	75-125%	≤20%	≤20%	0.30
Silver	ICP/MS	75-125%	≤20%	≤20%	0.25
Zinc	GFAA	75-125%	≤20%	≤20%	0.15

METHOD

A total of eight metals was analyzed in water and elutriate samples: silver (Ag), cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), nickel (Ni), lead (Pb) and zinc (Zn). Hg was analyzed using coldvapor atomic absorption spectroscopy (CVAA) according to the method of Bloom and Crecelius (1983). Cr and Zn were analyzed by Graphite Furnace Atomic Absorption (GFAA) spectrometry following the EPA Method 200.9 (EPA 1991). The remaining metals were analyzed by inductively coupled plasma mass spectrometry (ICP/MS) following a procedure based on EPA method 200.8 (EPA 1991).

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All water and elutriate samples were acidified to pH <2 upon receipt in the laboratory. Five metals, Cd, Cu, Pb, Ni and Ag, were extracted from the water according to a procedure based on EPA method 218.3 (EPA 1979). This preconcentration involves addition of a chelating agent which results in precipitation of the metals from solution, followed by filtration, and digestion of the filter in concentrated acid in order to achieve low detection limits. The digestates were then analyzed by ICP/MS as described above.

HOLDING TIMES One water sample was received on 6/16/94 and two samples on 7/6/94 in good condition. Samples were logged into Battelle's log-in system, acidified to pH<2 and held at ambient temperature until analysis. All samples have a 28 day holding time. Liberty Island site water was analyzed 45 days after collection. These data are flagged. Samples were all analyzed for the remaining metals within 180 days of collection.

QA/QC SUMMARY/METALS (Contd)

<u>Taşk</u>	<u>Date</u>
APDC Extraction	7/19/94
ICP-MS	8/16/94
CVAA-Hg	7/22/94
GFAA-Cr	8/10/94
GFAA-Zn	8/9/94

- **DETECTION LIMITS** Target detection limits were met for all metals except Zn. Detection limits for Zn exceeded the target limit; however, all sample values were well above the detection limits achieved. MDLs for Ag, Cd, Cu, Hg, Ni and Pb were determined by spiking 8 replicates of laboratory deionized water and multiplying the standard deviation of the resulting analysis by the student t value for n=8. MDLs reported for Cr and Zn were determined by taking the standard deviation of 3 replicate analyses of the method blank and multiplying the standard deviation by 3. MDL verification consisted of analyzing 4 replicates of Sequim Bay seawater and multiplying the standard deviation of the resulting analysis by 3.5. Detection limits for Cd, Cu, Hg and Zn. Cu and Zn concentrations in all samples were above the MDL verification level. Cd concentrations in two of three replicates for one sample were just below the MDL verification level. Hg concentrations for all three replicates of all samples were below the MDL level.
- METHOD BLANKS Two APDC procedural blanks were analyzed and no APDC metals (Ag, Cd, Cu, Ni and Pb) were detected in the blank. The blanks reported for Hg, Cr and Zn (the metals analyzed on waters directly) consist of a dilute nitric acid solution which is used to dilute all samples for analysis. Cr, Cu, Ni and Zn were detected in the blanks. Cr levels detected were less than 3 times the MDL. Zn and Cu were detected at levels greater than 3 times the MDLs. All data is corrected for the blank concentrations and therefore no data is flagged.
- MATRIX SPIKES One sample was spiked in duplicate with all metals (ICP-MS spike was performed on a different sample). The APDC metals were spiked prior to sample processing and the other metals were spiked just prior to analysis. All recoveries were within the QC limits of 75-125% with the exception of Pb in one spike duplicate. The recovery of Pb was 127%, just above the upper QC limit.
- **REPLICATES** Each sample was analyzed in triplicate. Precision for triplicate analyses is reported by calculating the relative standard deviation (RSD) between the replicate results. The RSDs for Ag (21%) and Cd (21%) exceeded the QC limits of ±20% in the Liberty Island site water sample. The RSD for Hg in the control seawater exceeded the QC limit but the concentrations were near the detection limit.

QA/QC SUMMARY/METALS (contd)

SRMs

SRM, CASS-2, a certified salt water sample from the National Institute of Standards and Technology, (NIST), was analyzed for all metals with the exception of Ag and Hg, which are not certified in this SRM. Cd and Pb were recovered above the upper control limit. The remaining metals were recovered within ±20 % of mean certified value.

A second SRM, 1641b, a freshwater sample from NIST, was analyzed twice for Hg. Results were within ± 20 % of mean certified value. No salt water SRMS are available which are certified for Ag.

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REFERENCES

Bloom, N. S., and E.A. Crecelius. 1983. "Determination of Mercury in Seawater at Sub-Nanogram per Liter Levels." <u>Mar. Chem.</u> 14:49-59.

EPA (U.S. Environmental Protection Agency). 1979. Revised (1983). Methods for the Chemical Analysis of Water and Wastes. EPA-600/4-79-020. Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

EPA (U.S. Environmental Protection Agency). 1991 Methods for the Determination of Metals in Environmental Samples. EPA-600/4-91-010. Environmental Services Division, Monitoring Management Branch, Washington D.C.

QA/QC SUMMARY

PROGRAM: New York/New Jersey Federal Projects 3

- PARAMETER: Chlorinated Pesticides
- LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington
- MATRIX: Water

QA/QC DATA QUALITY OBJECTIVES

Reference	Surrogate	MS	Relative	Detection
Method	<u>Recovery</u>	<u>Recovery</u>	Precision	Limit
GC/ECD	30-150%	50-120%	≤30%	2-20 ng/L

SAMPLE CUSTODY One water sample was received on 6/16/94 and two samples on 7/6/94 in good condition. Additional water from Sequim Bay was received on 6/16/94 to use for spiking. Samples were logged into Battelle's log-in system and stored at approximately 4°C until extraction.

- METHOD Water samples were extracted with methylene chloride using a separatory funnel under ambient conditions following a procedure that is based on methods used by the National Oceanic and Atmospheric Administration for its Status and Trends Program (Krahn et al. 1988). Samples were then cleaned using silica/alumina (5% deactivated) chromatography followed by HPLC cleanup (Krahn et al. 1988). Extracts were analyzed for 15 chlorinated pesticides using gas chromatography/electron Capture Detection (GC/ECD) following a procedure based on EPA method 8080 (EPA 1986). The column used was a J&W DB-17 and the confirmatory column was a DB-1701, both capillary columns (30-m x 0.25-mm I.D.).
- **HOLDING TIMES** Samples were extracted within 7 days of receipt. The first batch of samples received on 6/16/94 were extracted on 6/17/94 and the second batch of samples received on 7/6 were extracted on 7/7/94. All extracts were analyzed by GC/ECD from 7/9 through 7/11/94, within the established holding time of 40 days.
- **DETECTION LIMITS** Target detection limits were met for all pesticides. Actual method detection limits ranged from 0.28 to 2.22 ng/L depending on the pesticide compound. MDLs were determined from multiplying the standard deviation of 7 spiked replicates of Sequim Bay seawater by the student t value.
- METHOD BLANKS One method blank was extracted with each extraction batch for a total of 2 method blanks. No pesticides were detected above the MDL in any of the method blanks.

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QA/QC SUMMARY/CHLORINATED PESTICIDES (contd)

SURROGATES Two compounds, PCB congeners 103 and 198, were added to all samples prior to extraction to assess the efficiency of the analysis. Sample surrogate recoveries were all within the QC guidelines of 30-150%. Following the surrogate internal standard method, all results are corrected for surrogate recoveries. MATRIX SPIKES One water sample was spiked with 10 pesticides. All spike recoveries were within the control limit range of 50-120%. **BLANK SPIKES** Sequim Bay seawater was spiked with the same 10 pesticide compounds prior to extraction as a blank spike. Recoveries ranged from 84 to 112%. REPLICATES Each samples was extracted and analyzed in triplicate. Precision was measured by calculating the relative standard deviation between the replicate results. Few pesticide compounds were detected above the MDL in samples, however, RSDs for all detectable values were below the target precision goal of $\leq 30\%$

SRMs Not applicable.

MISCELLANEOUS

All pesticide detections were confirmed on a second, dissimilar column. Quantitations were considered confirmed if the RPD between the two columns is 75%.

REFERENCES

Krahn, M.M., C.A. Wigren, R.W. Pearce, L.K. Moore, R.G. Bogar, W.D. MacLeod, Jr., S.L. Chan, and D.W. Brown. 1988 "New HPLC Cleanup and Revised Extraction Procedures for Organic Contaminants," NOAA Technical Memorandum NMFS F/NWC-153. NOAA/NMFS/NWFSC, Seattle, Washington.

EPA (U.S. Environmental Protection Agency). 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SW-846. U.S. Document No. 955-001-00000, U.S. Environmental Protection Agency, Washington D. C.

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QA/QC SUMMARY

PROGRAM: New York/New Jersey Federal Projects 3

PARAMETER: PCB Congeners

LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington

MATRIX: Water

QA/QC DATA QUALITY OBJECTIVES

Reference	Surrogate	MS	Relative	Detection
Method	<u>Recovery</u>	<u>Recovery</u>	Precision	Limit
GC/ECD	30-150%	50-120%	≤30%	0.4 ng/g

SAMPLE CUSTODY One water sample was received on 6/16/94 and two samples on 7/6/94 in good condition. Additional water from Sequim Bay was received on 6/16/94 to use for spiking. Samples were logged into Battelle's log-in system and stored at approximately 4°C until extraction.

- METHOD Water samples were extracted with methylene chloride using a separatory funnel under ambient conditions following a procedure that is based on methods used by the National Oceanic and Atmospheric Administration for its Status and Trends Program (Krahn et al. 1988). Samples were then cleaned using Silica/Alumina (5% deactivated) chromatography followed by HPLC cleanup (Krahn et al. 1988). Extracts were analyzed for 15 chlorinated pesticides using Gas Chromatography/Electron Capture Detection (GC/ECD) following a procedure based on EPA method 8080 (EPA 1986). The column used was a J&W DB-17 and the confirmatory column was a DB-1701, both capillary columns (30-m x 0.25-mm 1.D.).
- **HOLDING TIMES** Samples were extracted within 7 days of receipt. The first batch of samples received on 6/16/94 were extracted on 6/17/94 and the second batch of samples received on 7/6 were extracted on 7/7/94. All extracts were analyzed by GC/ECD from 7/9 through 7/11/94, within the established holding time of 40 days.
- **DETECTION LIMITS** Target detection limits were met for all PCB congeners. Actual method detection limits ranged from 0.17 to 1.2 ng/L depending on the PCB congener. MDLs were determined from multiplying the standard deviation of 7 spiked replicates of Sequim Bay seawater by the student t value.

METHOD BLANKS One method blank was extracted with each extraction batch for a total of 2 method blanks. No PCB congeners were detected in method blanks

QA/QC SUMMARY/PCB CONGENERS (contd)

- SURROGATES Two compounds, PCB congeners 103 and 198, were added to all samples prior to extraction to assess the efficiency of the analysis. Sample surrogate recoveries were all within the QC guidelines of 30-150%. Following the surrogate internal standard method, all results are corrected for surrogate recoveries.
- MATRIX SPIKES Five (5) out of the 19 congeners analyzed were spiked. Matrix spike recoveries for the sample spiked ranged from 70 to 97%, within the control limit range of 50-120%.
- **BLANK SPIKES** Sequim Bay seawater was spiked with the same 5 congeners prior to extraction as a blank spike. Recoveries ranged from 84 to 112%.
- **REPLICATES** Each sample was extracted and analyzed in triplicate. Precision was measured by calculating the relative standard deviation between the replicate results. The calculated RSDs for Liberty Island elutriate samples ranged from 20% to 59%. Only 3 RSDs were >30% and had analyte concentrations >10 times the method detection limit.
- SRMs Not applicable.

MISCELLANEOUS

All pesticide detections were confirmed on a second, dissimilar column. Quantitations were considered confirmed if the RPD between the two columns is 75%.

REFERENCES

Krahn, M.M., C.A. Wigren, R.W. Pearce, L.K. Moore, R.G. Bogar, W.D. MacLeod, Jr., S.L. Chan, and D.W. Brown. 1988 "New HPLC Cleanup and Revised Extraction Procedures for Organic Contaminants," NOAA Technical Memorandum NMFS F/NWC-153. NOAA/NMFS/NWFSC, Seattle, Washington.

EPA (U.S. Environmental Protection Agency). 1986. Test Methods for Evaluating Solid Waste: *Physical/Chemical Methods*. SW-846. U.S. Document No. 955-001-00000, U.S. EPA, Washington D. C.

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	_				Concentrat	tions in µg/L			
		Ag	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Sample Type	Replicate	ICP/MS	ICP/MS	GFAA	ICP/MS	CVAF	ICP/MS	ICP/MS	GFAA
target detection limi	t	0.25	0.025	1.0	0.35	0.002	0.30	0.35	0.15
method detection limi		0.018	0.003	0.039	0.021	0.0001	0.028	0.010	0.203
MDL verificatior	ו	NA ^(a)	0.030	0.039	0.065	0.0004	0.091	0.013	0.690
Liberty Island Site Water	1	0.060	0.098	1.13	2.74	0.0146 ^(b)	1.10	1.87	16.1
Liberty Island Site Water	2	0.053	0.096	0.953	2.73	0.0126 ^(b)	1.08	1.82	14.9
Liberty Island Site Water	З	0.039	0.066	1.11	2.16	0.0120 ^(b)	0.836	1.45	15.6
Liberty Island Elutriate	1	0.018 U ^(c)	0.029	1.09	0.808	0.0066	2.03	0.881	2.22
Liberty Island Elutriate	2	0.015 J ^(d)	0.030	1.11	0.795	0.0060	1.97	0.863	2.68
Liberty Island Elutriate	3	0.016 J	0.029	1.20	0.795	0.0066	2.05	0.889	2.22
Control Seawater (Sequim Bay)	1	0.018 U	0.069	0.222	0.437	0.0001	0.359	0.046	0.70
Control Seawater (Sequim Bay)	2	0.018 U	0.063	0.266	0.462	0.0002	0.331	0.050	0.93
Control Seawater (Sequim Bay)	3	0.018 U	0.053	0.266	0.384	0.0002	0.299	0.034	0.70

TABLE B.1. Metals in Liberty Island Anchorage Site Water and Elutriate

(a) NA Not applicable.

(b) Samples analyzed outside of 28-day holding time.(c) U Undetected at or above concentration shown.

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(d) J Concentration estimated; analyte detected below detection limit.

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			Concentrations in µg/L							
		Ag	Cd	Cr	Cu	Hg	Ni	Pb	Zn	
Sample Type	Replicate	ICP/MS	ICP/MS	GFAA	ICP/MS	CVAF	ICP/MS	ICP/MS	GFAA	
Blank-1	1	0.018 U ^(b)	0.003 U	0.089	0.041	0.0001 U	0.050	0.01 U	0.93	
Blank-2	2	0.018 U	0.003 U	NA	0.071	NA	0.076	0.013	NA	
Mean Blank ^(c)		NA	NA	NA	0.056	NA	0.063	NA	NA	

TABLE B.2. Quality Control Data (Method Blank) for Metals in Liberty Island Anchorage Site Water and Elutriate

(a) NA Not applicable.

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(b) U Undetected at or above concentration shown.

(c) Mean blank used to blank-correct ICP/MS data when value >MDL shown.

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	Concentrations in µg/L								
Sample Type	Ag ICP/MS	Cd ICP/MS	Cr GFAA	Cu ICP/MS	Hg CVAF	Ni ICP/MS	Pb ICP/MS	Zn GFAA	
Amount Spiked	1.00	1.00	NS ^(a)	1.00	NS	1.00	1.00	NS	
Liberty Island Site Water ^(b)	0.051	0.087	NS	2.54	NS	1.00	1.00	NS	
Liberty Island Site Water + Spike 1	0.860	0.880	NS	3.27	NS	1.78	2.98	NS	
Amount Recovered	0.809	0.793	NS	0.729	NS	0.772	1.27	NS	
Percent Recovery	81%	79%	NS	73%	NS	77%	127% ^(c)	NS	
Amount Spiked	5.00	5.00	NS	5.00	NS	5.00	5.00	NS	
Liberty Island Site Water ^(b)	0.051	0.087	NS	2.54	NS	1.01	1.71	NS	
Liberty Island Site Water + Spike 2	4.76	4.37	NS	6.49	NS	4.68	7.98	NS	
Amount Recovered	4.71	4.28	NS	3.95	NS	3.67	6.27	NS	
Percent Recovery	94%	86%	NS	79%	NS	73%	125%	NS	
Amount Spiked	NS	NS	0.97	NS	14.3	NS	NS	4.67	
Liberty Island Site Water (b)	NS	NS	0.251	NS	0.0002	NS	NS	0.78	
Control Seawater + Spike	NS	NS	1.11	NS	14.3	NS	NS	6.06	
Amount Recovered	NS	NS	0.859	NS	14.3	NS	NS	5.28	
Percent Recovery	NS	NS	89%	NS	100%	NS	NS	113%	

TABLE B.3. Quality Control Data (Recovery of Matrix Spike) for Metals in Liberty Island Anchorage Site Water and Elutriate

(a) NS Not spiked.

(b) Mean of triplicate sample analysis.

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(c) Outside data quality criteria of 75-125%.

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	_				Concentra	tions in µg/L			
		Ag	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Sample Type	Replicate	ICP/MS	ICP/MS	GFAA	ICP/MS	CVAF	ICP/MS	ICP/MS	GFAA
Liberty Island Site Water	1	0.060	0.098	1.13	2.74	0.0146	1.10	1.87	16.1
Liberty Island Site Water	2	0.053	0.096	0.953	2.73	0.0126	1.08	1.82	14.9
Liberty Island Site Water	3	0.039	0.066	1.11	2.16	0.0120	0.836	1.45	15.6
% RSD ^(a))	21% ^(b)	21% ^(b)	9%	13%	10%	14%	13%	4%
Control Seawater (Sequim Bay)	1	0.018 U ^(c)	0.069	0.222	0.437	0.0001	0.359	0.046	0.70
Control Seawater (Sequim Bay)	2	0.018 U	0.063	0.266	0.462	0.0002	0.331	0.050	0.93
Control Seawater (Sequim Bay)	3	0.018 U	0.053	0.266	0.384	0.0002	0.299	0.034	0.70
% RSD	•	NA	13%	10%	9%	35% ^(b)	9%	19%	17%
Liberty Island Elutriate	1	0.018 U	0.029	1.09	0.808	0.0066	2.03	0.881	2.22
Liberty Island Elutriate	2	0.015 J ^(d)	0.030	1.11	0.795	0.0060	1.97	0.863	2.68
Liberty Island Elutriate	3	0.016 J	0.029	1.20	0.795	0.0066	2.05	0.889	2.22
% RSD		NA ^(e)	2%	5%	1%	5%	2%	2%	11%

<u>TABLE B.4</u>. Quality Control Data (Replicate Analyses) for Metals in Liberty Island Anchorage Site Water and Elutriate

(a) %RSD Percent relative standard deviation.

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(b) Outside data quality range of ±20% RSD.

(c) U Undetected at or above concentration shown.

(d) J Concentration estimated; analyte detected below detection limit.

(e) NA Not applicable.

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					Concentra	ations in µg/	L		
		Ag	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Sample Type	Replicate	ICP/MS	ICP/MS	GFAA	ICP/MS	CVAF	ICP/MS	ICP/MS	GFAA
CASS-2		0.006 U ^(a)	0.032 ^(b)	0.128	0.553	NA ^(c)	0.227	0.039 ^(c)	2.12
percent difference		NA	68%	6%	18%	NA	24%	105%	8%
certified value		NC (d)	0.019	0.121	0.675	NC	0.298	0.019	1.97
range		NC	±0.004	±0.016	. ±0.039	NC	±0.036	±0.006	±0.12
1641b	1	NA	NA	NA	NA	1590	NA	NA	NA
percent difference	•	NA	NA	NA	NA	5%	NA	NA	NA
1641b	2	NA	NA	NA	NA	1560	NA	NA	NA
percent difference		NA	NA	NA	NA	3%	NA	NA	NA
certified value		NC	NC	NC	NC	1520	NC	NC	NC
range		NC	NC	NC	NC	±40	NC	NC	NC

TABLE B.5. Quality Control Data (Standard Reference Material) for Metals in Liberty Island Anchorage Site Water and Elutriate

(a) U Undetected at or above concentration shown.

(b) Outside data quality range of $\pm 20\%$ of certified value.

(c) NA Not applicable.

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(d) NC SRM not certified for this analyte.

	_	Concentrations in µg/L							
		Ag	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Sample Type	Replicate	ICP/MS	ICP/MS	GFAA	ICP/MS	CVAF	ICP/MS	ICP/MS	GFAA
Control Seawater (Sequim Bay)	1	0.018 U ^(a)	0.043	0.288	0.232	0.0004	0.481	0.021	0.47
Control Seawater (Sequim Bay)	2	0.018 U	0.047	0.288	0.260	0.0005	0.457	0.018	0.70
Control Seawater (Sequim Bay)	3	0.018 U	0.063	0.288	0.259	0.0004	0.509	0.013	0.58
Control Seawater (Sequim Bay)	4	0.018 U	0.050	0.310	0.224	0.0002	0.453	0.014	0.93
% RSD ⁽¹)	NA ^(c)	17%	4%	8%	34%	5%	22%	29%
SD ⁽	i)	NA	0.0087	0.011	0.019	0.0001	0.026	0.0037	0.197
MDL ())	NA	0.0395	0.050	0.084	0.0005	0.118	0.0168	0.894

<u>TABLE B.6</u>. Quality Control Data (MDL Verification) for Metals in Liberty Island Anchorage Site Water and Elutriate

(a) U Undetected at or above concentration shown.

(b) % RSD Percent relative standard deviation.

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(c) NA Not applicable/available.

(d) SD Standard deviation.

(e) MDL Method detection limit, calculated as SD x 4.54.

	Liberty Island	Liberty Island	Liberty Island	Liberty Island	Liberty Island	
Sample	Site Water	Site Water	Site Water	Site Water	Site Water	
	Rep. 1	Rep. 2	Rep. 3	Mean Conc. ^(a)	Mean Conc. ^(b)	
Sample Size (L)	1.04	1.03	1.04			
Units	ng/L	ng/L	ng/L	ng/L	ng/L	%RSD ^(c)
Heptachlor	1.06 U ^(d)	1.05 U	1.05 U	1.05 U	0.527 U	NA ^(e)
Aldrin	0.79 U	0.78 U	0.78 U	0.783 U	0.392 U	NA
Heptachlor Epoxide	2.22 U	2.20 U	2.20 U	2.21 U	1.103 U	NA
2,4'-DDE	0.77 U	0.76 U	0.76 U	0.763 U	0.382 U	NA
Endosulfan I	1.37 U	1.35 U	1.35 U	1.36 U	0.678 U	NA
alpha-Chlordane	2.00 U	1.98 U	1.98 U	1.99 U	0.993 U	NA
frans-Nonachlor	0.59 U	0.58 U	0.58 U	0.583 U	0.292 U	NA
4,4'-DDE	0.88 U	0.87 U	0.87 U	0.873 U	0.437 U	NA
Dieldrin	0.38 U	0.38 U	0.38 U	0.380 U	0.190 U	NA
2,4'-DDD	1.02 U	1.01 U	1.01 U	1.01 U	0.507 U	NA
2,4'-DDT	0.92 U	0.91 U	0.91 U	0.913 U	0.457 U	NA
4,4'-DDD	0.29 U	0.28 U	0.28 U	0.283 U	0.142 U	NA
Endosulfan II	1.37 U	1.35 U	1.35 U	1.36 U	0.678 U	NA
4,4'-DDT	0.52 U	0.52 U	0.52 U	0.520 U	0.260 U	NA
Endosulfan Sulfate	1.37 U	1.35 U	1.35 U	1.36 U	0.678 U	NA
PCB 8	1.20 U	1.19 U	1.19 U	1.19 U	0.597 U	NA
PCB 18	0.78 U	0.77 U	0.77 U	0.773 U	0.387 U	NA
PCB 28	0.63	0.58	0.62	0.610	0.610	4
PCB 52	0.41 U	0.41 U	0.41 U	0.410 U	0.205 U	NA
PCB 49	0.51	0.41 U	0.50	0.473	0.405	NA
PCB 44	4.43	0.56 U	6.06	3.68	3.59	NA
PCB 66	0.44 U	0.43 U	0.43 U	0.433 U	0.217 U	NA
PCB 101	0.20 U	0.20 U	0.20 U	0.200 U	0.100 U	NA
PCB 87	0.37 U	0.36 U	0.36 U	0.363 U	0.182 U	NA
PCB 118	0.24 U	0.24 U	0.24 U	0.240 U	0.120 U	NA
PCB 184	0.42 U	0.99	0.94	0.783	0.713	NA
PCB 153	0.17 U	0.17 U	0.17 U	0.170 U	0.085 U	NA
PCB 105	0.17 U	0.17 U	0.17 U	0.170 U	0.085 U	NA
PCB 138	0.19 U	0.19 U	0.19 U	0.190 U	0.095 U	NA
PCB 187	0.38 U	0.37 U	0.37 U	0.373 U	0.187 U	NA
PCB 183	0.42 U	0.41 U	0.41 U	0.413 U	0.207 U	NA
PCB 128	0.30 U	0.29 U	0.29 U	0.293 U	0.147 U	NA
PCB 180	0.34 U	0.33 U	0.33 U	0.333 U	0.167 U	NA
PCB 170	0.37 U	0.37 U	0.37 U	0.370 U	0.185 U	NA
PCB 195	1.17 U	1.16 U	1.16 U	1.16 U	0.582 U	NA
PCB 206	0.23 U	0.23 U	0.23 U	0.230 U	0.115 U	NA
PCB 209	0.25 U	0.24 U	0.24 U	0.243 U	0.122 U	NA
TOTAL PCB ()					18.2	
	(9/)					
Surrogate Recoveries		74.48	77 47	NA	NA	NA
PCB 103 (SIS) ^(g)	77.11		77.47			
PCB 198 (SIS)	88.82	80.37	84.07	NA	NA	NA

<u>TABLE B.7</u>. Pesticides and Polychlorinated Biphenyls in Liberty Island Anchorage Site Water and Elutriate With Quality Control Data (Replicate Analyses)

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<u>TABLE B.7</u> .	(contd)
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Sample	Liberty Island Elutriate Rep. 1	Liberty Island Elutriate Rep. 2	Liberty Island Elutriate Rep. 3	Liberty Island Elutriate Mean Conc. ^(a)	Liberty Island Elutriate Mean Conc. ^(b)	
Sample Size (L) Units	1.05 ng/L	1.05 ng/L	1.05 ng/L	ng/L	ng/L	%RSD
Heptachlor	1.07 U	1.07 U	1.07 U	1.07 U	0.535 U	NA
Aldrin	0.79 U	0.79 U	0.79 U	0.79 U	0.395 U	NA
Heptachlor Epoxide	2.24 U	2.24 U	2.24 U	2.24 U	1.12 U	NA
2.4'-DDE	0.78 U	0.78 U	0.78 U	0.78 U	0.39 U	NA
Endosulfan I	1.38 U	1.38 Ū	1.38 U	1.38 U	0.69 U	NA
alpha-Chlordane	2.02 U	2.02 U	2.02 U	2.02 U	1.01 U	NA
trans-Nonachlor	0.59 U	0.59 U	0.59 U	0.59 U	0.295 U	NA
4.4'-DDE	8.39	7.58	6.72	7.56	7.56	11
Dieldrin	5.51	5.34	4.66	5.17	5.17	9
2,4'-DDD	1.26	1.35	1.19	1.27	1.27	6
2,4'-DDT	0.93 U	0.93 U	0.93 U	0.93 U	0.465 U	NĂ
4,4'-DDD	0.29 U	0.29 U	0.29 U	0.29 U	0.145 U	NA
Endosulfan II	1.38 U	1.38 U	1.38 U	1.38 U	0.690 U	NA
4,4'-DDT	0.53 U	0.53 U	0.53 U	0.53 U	0.265 U	NA
Endosulfan Sulfate	1.38 U	1.38 U	1.38 U	1.38 U	0.690 U	NA
PCB 8	1.21 U	1.21 U	1.21 U	1.21 U	0.605 U	NA
PCB 18	21.9	18.7	14.6	18.4	18.4	20
PCB 28	10.8	8.7	6.38	8.63	8.63	26
PCB 52	10.3	8.02	5.48	7.93	7.93	30
PCB 49	7.59	5.73	3.97	5.76	5.76	31 ^(h)
PCB 44	13.6	12.6	9.14	11.78	11.8	20
PCB 66	11.1	8.56	6.32	8.66	8.66	28
PCB 101	4.81	3.59	2.35	3.58	3.58	34 ^(h)
PCB 87	0.37 U	0.37 U	0.37 U	0.370 U	0.185 U	NA
PCB 118	3.46	2.44	1.89	2.60	2.60	31
PCB 184	1.41	0.42 U	0.42 U	0.750 U	0.610	NA
PCB 153	3.23	2.31	1.81	2.45	2.45	29
PCB 105	0.17 U	0.17 U	0.17 U	0.170 U	0.085 U	NA
PCB 138	5.00	2.82	2.06	3.29	3.29	46 ^(h)
PCB 187	0.38 U	0.38 U	0.38 U	0.380 U	0.190 U	NA
PCB 183	0.42 U	0.42 U	0.42 U	0.420 U	0.210 U	NA
PCB 128	1.22	0.67	0.35	0.747	0.747	59 ^(h)
PCB 180	2.63	2.1	1.11	1.95	1.95	40 ^(h)
PCB 170	0.37 U	0.85	0.71	0.643	0.582	NA
PCB 195	1.18 U	1.18 U	1.18 U	1.18 U	0.590 U	NA
PCB 206	0.86	0.59	0.54	0.663	0.663	26
PCB 209	0.25 U	0.25 U	0.25 U	0.250 U	0.125 U	NA
TOTAL PCB ^(I)					159	
Surrogate Recoverie						
PCB 103 (SIS) ⁽⁹⁾	, 75.41	85.27	87.04	NA	NA	NA ¯
PCB 198 (SIS)	86.98	93.67	94.58	NA	NA	NA

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TABLE B.7. (contd)

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Sample	(Sequim Bay) Rep. 1	Control Water (Sequim Bay) Rep. 2	Control Water (Sequim Bay) Rep. 3	Control Water (Sequim Bay) Mean Conc. ^(a)	Control Water (Sequim Bay) Mean Conc. ^(b)	
Sample Size (L)	1.00	1.00	1.00			
Units	ng/L	ng/L	ng/L	ng/L		%RSD
Heptachlor	1.02 U	1.02 U	1.02 U	1.02 U	0.51 U	NA
Aldrin	0.76 U	0.76 U	0.76 U	0.76 U	0.38 U	NA
Heptachlor Epoxide	2.14 U	2.14 U	2.14 U	2.14 U	1.07 U	NA
2,4'-DDE	0.74 U	0.74 U	0.74 U	0.74 U	0.37 U	NA
Endosulfan I	1.31 U	1.31 U	1.31 U	1.31 U	0.655 U	NA
alpha-Chlordane	1.93 U	1.93 U	1.93 U	1.93 U	0.965 U	NA
trans-Nonachlor	0.57 U	0.57 U	0.57 U	0.57 U	0.285 U	NA
4,4'-DDE	0.84 U	0.84 U	0.84 U	0.84 U	0.42 U	NA
Dieldrin	0.36 U	0.36 U	0.36 U	0.36 U	0.18 U	NA
2,4'-DDD	0.98 U	0.98 U	0.98 U	0.98 U	0.49 U	NA
2,4'-DDT	0.88 U	0.88 U	0.88 U	0.88 U	0.44 U	NA
4,4'-DDD	0.28 U	0.28 U	0.28 U	0.28 U	0.14 U	NA
Endosulfan II	1.31 U	1.31 U	1.31 U	1.31 U	0.655 U	NA
4,4'-DDT	0.50 U	0.50 U	0.50 U	0.50 U	0.25 U	NA
Endosulfan Sulfate	1.31 U	1.31 U	1.31 U	1.31 U	0.655 U	NA
PCB 8	1.15 U	1.15 U	1.15 U	1.15 U	0.575 U	NA
PCB 18	0.75 U	0.75 U	0.75 U	0.75 U	0.375 U	NA
PCB 28	0.32 U	0.32 U	0.32 U	0.32 U	0.16 U	NA
PCB 52	0.40 U	0.40 U	0.40 U	0.40 U	0.20 U	NA
PCB 49	0.40 U	0.40 U	0.40 U	0.40 U	0.20 U	NA
PCB 44	0.55 U	0.55 U	0.55 U	0.55 U	0.275 U	NA
PCB 66	0.42 U	0.42 U	0.42 U	0.42 U	0.21 U	NA
PCB 101	0.19 U	0.19 U	0.19 U	0.19 U	0.095 U	NA
PCB 87	0.35 U	0.35 U	0.35 U	0.35 U	0.175 U	NA
PCB 118	0.23 U	0.23 U	0.23 U	0.23 U	0.115 U	NA
PCB 184	0.40 U	0.40 U	0.40 U	0.40 U	0.20 U	NA
PCB 153	0.17 U	0.17 U	0.17 U	0.17 U	0.085 U	NA
PCB 105	0.17 U	0.17 U	0.17 U	0.17 U	0.085 U	NA
PCB 138	0.18 U	· 0.18 U	0.18 U	0.18 U	0.09 U	NA
PCB 187	0.36 U	0.36 U	0.36 U	0.36 U	0.18 U	NA
PCB 183	0.40 U	0.40 U	0.40 U	0.40 U	0.20 U	NA
PCB 128	0.29 U	0.29 U	0.29 U	0.29 U	0.145 U	NA
PCB 180	0.32 U	0.32 U	0.32 U	0.32 U	0.16 U	NA
PCB 170	0.35 U	0.35 U	0.35 U	0.35 U	0.175 U	NA
PCB 195	1.12 U	1.12 U	1.12 U	1.12 U	0.56 U	NA
PCB 206	0.23 U	0.23 U	0.23 U	0.23 U	0.115 U	NA
PCB 209	0.24 U	0.24 U	0.24 U	0.24 U	0.12 U	NA
TOTAL PCB ^(I)					9.0	
Surrogate Recoveries	<u>s (%)</u>					
PCB 103 (SIS) (g)	78.12	88.05	83.14	NA	NA	NA
PCB 198 (SIS)	90.15	96.68	91.43	NA	NA	NA

(a) Mean concentration of triplicate samples calculated using full undetected value when analyte was undetected.

(b) Mean concentration of triplicate samples calculated using one-half undetected value when analyte was undetected.

(c) %RSD Percent relative standard deviation.

(d) U Not detected at or above concentration shown.

(e) NA Not applicable.

(f) Total PCB calculated as 2x, where x=sum of all PCB congeners; ½ detection limit used when analyte was undetected.

(g) SIS Surrogate internal standard.(h) Outside quality control range (µ30%).

	Control W	/ater	Control Water	Matrix Spike
Sample	(Sequim Bay)	Conc.	+ Matrix Spike	Percent
	Mean Conc. ^(a)	Spiked	Conc. Recovered	Recovery
Sample Size (L)	1.00		1.00	
Units	ng/L	ng/L	ng/L	%
Heptachlor	1.02 U ^(b)	25	16.6	66
Aldrin	0.76 U	25	27.1	108
Heptachlor Epoxide	2.14 U	25	25.8	103
2,4'-DDE	0.74 U	NS ^(c)	NS	NS
Endosulfan I	1.31 U	25	19.0	76
alpha-Chlordane	1.93 U	NS	NS	NS
trans-Nonachlor	0.57 U	NS	NS	NS
4,4'-DDE	0.84 U	25	18.5	74
Dieldrin	0.36 U	25	20.0	80
2,4'-DDD	0.98 U	NS	NS	NS
2,4'-DDT	• 0.88 U	NS	NS	NS
4,4'-DDD	0.28 U	25	20.8	83
Endosulfan II	1.31 U	25	22.3	89
4,4'-DDT	0.50 U	25	21.3	85
Endosulfan Sulfate	1.31 U	25	21.5	86
PCB 8	1.15 U	NS	NS	NS
PCB 18	0.75 U	NS	NS	NS
PCB 28	0.32 U	31.9	31.0	97
PCB 52	0.40 U	66.5	58.8	88
PCB 49	0.40 U	NS	NS	NS
PCB 44	0.55 U	NS	NS	NS
PCB 66	0.42 U	NS	NS	NS
PCB 101	0.19 U	45.1	43.7	97
PCB 87	0.35 U	NS	NS	NS
PCB 118	0.23 U	NS	NS	NS
PCB 184	0.40 U	NS	NS	NS
PCB 153	0.17 U	26.4	18.6	70
PCB 105	0.17 U	NS	NS	NS
PCB 138	0.18 U	20.4	15.6	76
PCB 187	0.36 U	NS	NS	NS
PCB 183	0.40 U	NS	NS	NS
PCB 128	0.29 U	NS	NS	NS
PCB 180	0.32 U	NS	NS	NS
PCB 170	0.35 U	NS	NS	NS
PCB 195	1.12 U	NS	NS	NS
PCB 206	0.23 U	NS	NS	NS
PCB 209	0.24 U	NS	NS	NS -
Surrogate Recoveries (%)				
PCB 103 (SIS) (d)	NA ^(e)	NA	61.11	NA
PCB 198 (SIS)	NA	NA	93.19	NA

TABLE B.8. Quality Control Data (Recovery of Matrix Spike) for Pesticides and Polychlorinated Biphenyls (PCB) in Liberty Island Anchorage Site Water and Elutriate

(a) Mean concentration of triplicate samples calculated using full undetected value when analyte was undetected.
(b) U Not detected at or above detection limit shown.
(c) NS Not spiked.
(d) SIS Surrogate internal standard.
(e) NA Not applicable.

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Appendix C.

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Water-Column Toxicity Test Data, Liberty Island Anchorage

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Sediment Treatment	Concentration (Percent SPP)	Replicate	Live ^(a)	Dead	Proportion Surviving	Mean Proportion Surviving	Standard Deviation
	-					· ·	
COMP LI	0	1	10	0	1.00		
COMP LI	0	2	10	0	1.00		
COMP LI	0	3	10	0	1.00		
COMP LI	0	4	10	0	1.00		
COMP LI	0	5	9	1	0.90	0.98	0.04
COMP LI	10	1	10	0	1.00		
COMP LI	10	2	10	0	1.00		
COMP LI	10	3	9	1	0.90		
COMP LI	10	4	9	1	0.90		
COMP LI	10	5	10	0	1.00	0.96	0.05
COMP LI	50	1	10	0	1.00		
COMP LI	50	2	10	0	1.00		
COMP LI	50	3	10	0	1.00		
COMP LI	50	4	10	0	1.00		
COMP LI	50	5	10	0	1.00	1.00	0.00
	•••	-		•			
COMP LI	100	1	8	2	0.80		
COMP LI	100	2	9	1	0.90		
COMP LI	100	3	10	0	1.00		
COMP LI	100	4	9	1	0.90		
COMP LI	100	5	8	2	0.80	0.88	0.08
		U	Ŭ	-	0.00	0.00	0.00

<u>TABLE C.1</u>. Results of *M. beryllina* 96-Hour Water-Column Toxicity Test, Liberty Island Anchorage

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(a) Survival based on the initial exposure of 10 organisms per replicate.

						Diss	olved		
		•	erature			Oxy	rgen	Sali	inity
Sediment	Concentration	(°,	(°C)		<u>H</u>	(m	g/L)	(0/00)	
Treatment	Percent SPP	Min	<u>Max</u>	Min	Max	Min	Max	Min	Max
Acceptable Range		18.0	22.0	7.30	8.30	≥3.0	NA ^(a)	28.0	32.0
COMP LI	0	19.2	19.8	7.72	7.92	7.4	8.3	30.5	31.0
COMP LI	10	19.2	19.7	7.60	8.15	7.4	8.3	30.5	31.0
COMP LI	50	19.1	19.7	7.79	8.18	7.4	8.3	30.0	31.0
COMP LI	100	19.3	19.8	7.70	8.29	6.7	8.3	29.0	31.0

TABLE C.2. Water Quality Summary for *M. beryllina* 96-Hour Water-Column Toxicity Test, Liberty Island Anchorage

(a) NA Not applicable.

Copper Concentration (µg/L)	Replicate	Live ^(a)	Dead or Missing	Proportion Surviving	Mean Proportion Surviving	Standard Deviation
						-
0	1	10	0	1.00		
0	2	10	0	1.00	1.00	0.00
16	1	10	0	1.00		
16	2	10	0	1.00	1.00	0.00
64	1	10	0	1.00		
64	2	9	1	0.90	0.95	0.07
160	1	1	9	0.10		
160	2	0	10	0.00	0.05	0.07
400	1	0	10	0.00		
400	2	0	10	0.00	0.00	0.00

<u>TABLE C.3</u>. Results of *M. beryllina* 96-Hour Copper Reference Toxicant Test, Liberty Island Anchorage

(a) Survival based on initial exposure of 10 organisms per replicate.

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					Dissolved				
Copper	Tempe	erature			Оху	gen	Sali	nity	
Concentration	(°(C)	p	Н	(mg	j/L)	(o/	00)	
(µg/L)	Min Max		Min	Max	Min	Max	Min	Max	
Acceptable									
Range	18.0	22.0	7.30	8.30	<u>≥</u> 3.0	NA ^(a)	28.0	32.0	
0	19.2	19.8	7.62	7.96	6.0	8.5	30.5	31.0	
16	19.2	19.8	7.57	8.03	5.6	8.4	30.5	31.5	
64	19.2	19.9	7.50	7.96	5.4	8.4	30.5	30.5	
160	19.1	19.8	7.76	7.96	4.4	8.4	30.5	31.0	
400	19.2	19.7	7.72	7.95	4.7	. 8.5	30.5	30.5	

<u>TABLE C.4</u>. Water Quality Summary for *M. beryllina* 96-Hour Copper Reference Toxicant Test, Liberty Island Anchorage

(a) NA Not applicable.

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					•	Mean	
Sediment	Concentration			Dead or	Proportion	Proportion	Standard
Treatment	(Percent SPP)	Replicate	Live ^(a)	Missing	Surviving	Surviving	Deviation
COMP LI	0	1	10	0	1.00		
COMP LI	0	2	10	0	1.00		
COMP LI	0	3	9	1	0.90		
COMP LI	0	4	10	0	1.00		
COMP LI	0	5	10	0	1.00	0.98	0.04
	•						
COMP LI	10	1	10	0	1.00		
COMP LI	10	2	10	0	1.00		
COMP LI	10	3	10	0	1.00		
COMP LI	10	4	10	0	1.00		
COMP LI	10	5	10	0	1.00	1.00	0.00
COMP LI	50	1	9	1	0.90		
COMP LI	50	2	10	0	1.00		
COMP LI	50	3	10	0	1.00		
COMP LI	50	4	10	0	1.00		
COMP LI	50	5	9	1	0.90	0.96	0.05
COMP LI	100	1	10	0	1.00		
COMP LI	100	2	9	1	0.90		
COMP LI	100	3	8	2	0.80		
COMP LI	100	4	10	0	1.00		
COMP LI	100	5	10	0	1.00	0.94	0.09

<u>TABLE C.5</u>. Results of *M. bahia* 96-Hour Water Column Toxicity Test, Liberty Island Anchorage

(a) Survival based on initial exposure of 10 organisms per replicate.

						Disso	olved		
		Tempe	rature			Oxy	/gen	Sal	inity
Sediment Concentration		(°(C)	р	H	(m	g/L)	(0/00)	
Treatment	(Percent SPP)	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range		18.0	22.0	7.30	8.30	<u>≥</u> 3.0	NA ^(a)	28.0	32.0
COMP LI	0	19.4	19.9	7.39	8.08	3.7	8.1	30.5	31.5
COMP LI	10	16.6 ^(b)	19.8	7.38	7.91	3.2	8.2	30.5	31.5
COMP LI	50	19.3	19.8	7.51	8.08	3.0	8.1	29.5	31.0
COMP LI	100	19.2	19.8	7.74	8.08	5.5	8.1	29.0	31.0

TABLE C.6. Water Quality Summary for M. bahia 96-Hour Water-Column Toxicity Test Liberty Island Anchorage

(a) NA Not applicable.(b) Data point out of range.

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Copper					Mean	
Concentration			Dead or	Proportion	Proportion	Standard
(µg/L)	Replicate	Live ^(b)	Missing	Surviving	Surviving	Deviation
				¥	¥_	
0	1	10	0	1.00		
0	2	9	1	0.90	0.95	0.07
100	1	10	0	1.00		
100	2	9	[•] 1	0.90	0.95	0.07
150	1	10	0	1.00		
150	2	8	2	0.80	0.90	0.14
200	1	10	0	1.00		
200	2	8	2	0.80	0.90	0.14
300	1	3	7	0.30		
300	2	4	6	0.40	0.35	0.07
(a) This test was a set of the		-1 111	e		• • • •	

<u>TABLE C.7</u>. Results of 96-Hour *M. bahia* Reference Toxicant Test ^(a) Liberty Island Anchorage

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(a) This test was used to assess mysid sensitivity for the water-column toxicity test.(b) Survival based on initial exposure of 10 organisms per replicate.

					Diss	olved		
Copper	Tempe	erature			Оху	rgen	Sali	inity
Concentration	(°	C)	р	<u>н</u>	(mg	g/L)	(o/	00)
(μg/L)	Rep 1	Rep 2	Rep 1	Rep 2	Rep 1	Rep 2	Rep 1	Rep 2
Acceptable						<i>(</i> L)		
Range	18.0	22.0	7.30	8.30	>3.0	NA ^(b)	28.0	32.0
0	19.7	19.8	7.72	8.02	7.4	8.0	31.0	31.0
100	19.7	19.8	7.89	7.80	7.8	8.0	31.0	31.0
150	19.7	19.7	7.42	7.82	4.7	7.2	30.5	30.5
200	19.7	19.8	7.79	7.79	7.7	7.7	31.0	31.0
300	19.7	19.7	8.01	7.92	8.0	7.9	31.0	31.0

<u>TABLE C.8.</u> Water Quality Summary for the *M. bahia* 96-Hour Copper Reference Toxicant Test^(a), Liberty Island Anchorage

(a) This test was used to assess mysid sensitivity for the water-column toxicity test. Data shown are measurements made at test termination (96-h).

(b) NA Not applicable.

			Mean							Mean	Mean	
Sediment	Concentration		Stocking	Number			Proportion	Number	Proportion			Standard
Treatment	(Percent SPP)	Replicate	Density	Normal	Abnormal	Other	Normal	Surviving	Surviving	Normal		Deviation ^(a)
COMP LI	0	1	310	234	0	20	0.76	254	0.82			
COMP LI	0	2	310	294	0	21	0.95	315	1.00			
COMP LI	0	3	310	251	0	14	0.81	265	0.85			
COMP LI	0	4	310	302	0	24	0.97	326	1 00 ^(a)			

Table C.9. Results of Larval M. galloprovincialis 48-Hour Water-Column Toxicity Test, Liberty Island Anchorage

CO co CO COMP LI 0.97 1.00 COMP LI 0.83 0.89 0.86 0.91 0.08 COMP LI 0.82 0.89 COMP LI 0.87 0.91 COMP LI 0.47 0.52 COMP LI 0.85 0.91 COMP LI 0.85 0.90 0.77 0.83 0.17 COMP LI 0.63 0.89 COMP LI 0.16 0.88 COMP LI 0.18 0.84 COMP LI 0.07 0.96 COMP LI 0.15 0.24 0.88 0.82 0.06 COMP LI 0.00 0.89 COMP LI 1.00^(a) 1.00 COMP LI 1.00^(a) 0.00 COMP LI 0.00 0.78 0.98 COMP LI 0.00 0.93 0.20 0.10

(a) Standard deviation is based on proportion surviving.

(b) When number normal or number surviving exceeded the stocking density, a proportion normal and/or proportion surviving of 1.00 was used for mean calculations and statistical analysis.

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		T				Dissolved Oxygen Salinity				Total		
	_	-	erature			Oxy	/gen	Sal	inity	Ammo	onia ^(a)	
Sediment	Concentration	(°	C)	р	Н	(m	g/L)	(o/	00)	(mg		
Treatment	(Percent SPP)	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	-
Acceptable Range		14.0	18.0	7.30	8.30	≥4.9	NA ^(b)		00.0			-
nange		14.0	10.0	7.30	0.30	≥4.9	NA"	28.0	32.0	NE ^(c)	NE	
COMP LI	0	14.5	15.5	7.83	8.05	7.9	8.7	30.5	31.0	<1.00	<1.00	
COMP LI	10	14.6	15.4	7.81	8.06	7.8	8.4	30.0	31.0	1.11	1.11	
COMP LI	50	14.6	15.3	7.84	8.14	7.8	8.3	29.0	30.0	2.93	4.90	
COMP LI	100	14.4	15.3	7.73	8.17	7.8	8.3	28.5	30.0	7.91	11.3	

TABLE C.10. Water Quality Summary for Larval *M. galloprovincialis* 48-Hour Water-Column Toxicity Test, Liberty Island Anchorage

(a) Ammonia measured in overlying water.(b) NA Not applicable.(c) NE Not established.

Copper Concentration (µg/L)	Replicate	Mean Stocking Density		Abnormal	Other	Proportion Normal		Proportion Surviving	Mean Proportion Normal	Mean Proportion Surviving	Standard Deviation ^(a)
										<u> </u>	
~ ~ ~		010		•				(b)			
0.0	1	310	304	2	21	0.98	327	1.00 ^(b)			
0.0	2 3	310	308	0	22	0.99	330	1.00 ^(b)			
0.0		310	258	0	19	0.83	277	0.89			
0.0	4.	310	268	0	17	0.86	285	0.92			
0.0	5	310	241	0	26	0.78	267	0.86	0.89	0.93	0.06
1.0	1	310	232	0	19	0.75	251	0.81			
1.0	2	310	271	0	11	0.87	282	0.91		-	
1.0	3	310	219	Ō	11	0.71	230	0.74	0.78	0.82	0.08
	•		1.0	U	••	0.7 1	200	0.74	0.70	0.02	0.00
4.0	1	310	59	56	180	0.19	295	0.95			
4.0	2	310	106	53	127	0.34	286	0.92			
4.0	3	310	199	28	51	0.64	278	0.90	0.39	0.92	0.03 ,
	Ū	010		20	01	0.04	270	0.30	0.09	0.52	0.03 ,
16.0	1	310	0	0	77	0.00	77	0.25			
16.0	2	310	Ō	0	44	0.00	44	0.14			
16.0	3	310	õ	õ	90	0.00	90	0.14	0.00	0.00	
10.0	0	010	0	0	90	0.00	90	0.29	0.00	0.23	0.08
64.0	1	310	0	0	0	0.00	0	0.00			
							0	0.00			
64.0	2	310	0	0	0	0.00	0	0.00			
64.0	3	310	0	0	0	0.00	0	0.00	0.00	0.00	0.00

Table C.11. Results of Larval M. edulis 48-Hour Copper Reference Toxicant Test, Liberty Island Anchorage

(a) Standard deviation is based on proportion surviving.

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(b) When number normal or number surviving exceeded the stocking density, a proportion normal and/or proportion survival of 1.00 was used for mean calculations and statistical analysis.

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						Total				
Copper	Tempe	erature			Oxy	/gen	Sal	inity	Ammo	onia ^(a)
Concentration	(°	C)	р	Н	(m	g/L)	(o/	00)		g/L)
(µg/L)	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable						(h)				
Range	14.0	18.0	7.30	8.30	<u>≥</u> 4.9	NA ^(b)	27.0	33.0	NE ^(c)	NE
0.0	14.5	14.9	7.78	8.04	7.9	8.7	30.0	31.0	<1.00	<1.00
1.0	14.6	15.1	7.62	8.05	7.8	8.4	30.0	31.0	<1.00	<1.00
4.0	14.5	14.9	7.67	8.05	7.8	8.3	30.0	31.0	<1.00	<1.00
16.0	14.6	15.0	7.66	8.05	7.9	8.3	30.0	31.0	<1.00	<1.00
64.0	14.5	14.8	7.64	8.04	7.8	8.2	30.0	31.0	<1.00	<1.00

TABLE C.12. Water Quality Summary for *M. galloprovincialis* 48-Hour Copper Reference Toxicant Test, Liberty Island Anchorage

(a) Ammonia measured in overlying water.

(b) NA Not applicable.(c) NE Not established.

Appendix D.

Benthic Acute Toxicity Test Data, Liberty Island Anchorage

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					Mean	
Sediment			Dead or	Proportion	Proportion	Standard
Treatment	Replicate	Live ^(a)	Missing	Surviving	Surviving	Deviation
• • • • • • • • • • • • • • • • • • •						,
COMP LI	1	11	9	0.55		
COMP LI	2	8	12	0.40		
COMP LI	3	16	4	0.80		
COMP LI	4	15	5	0.75		
COMP LI	5	2	18	0.10	0.52	0.28
R-MUD	1	20	0	1.00		
R-MUD	2	19	1	0.95		
R-MUD	3	18	2	0.90		
R-MUD	4	17	3	0.85		
R-MUD	5	20	0	1.00	0.94	0.07
C-AMP	1	18	2	0.90		
C-AMP	2	18	2	0.90		
C-AMP	3	18	2	0.90		
C-AMP	4	18	2	0.90		
C-AMP	5	16	4	0.80	0.88	0.04

<u>TABLE D.1</u>. Results of *A. abdita* 10-Day, Static Renewal, Benthic Acute Toxicity Test, Liberty Island Anchorage

(a) Survival based on the initial exposure of 20 organisms per replicate.

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Sediment	•	perature (°C)	pl	4	Dissolved Oxygen Salinity (mg/L) (o/oo)			•	Total Ammonia ^(a) (mg/L)		
Treatment	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	
Acceptable Range	18.0	22.0	7.30	8.30	<u>≥</u> 4.6	NA ^(b)	28.0	32.0	NA	30.0	
COMP LI	19.6	20.0	7.37	8.59 ^(c)	7.1	7.9	30.0	30.5	<1.00	1.21	
R-MUD	19.5	20.2	7.83	8.11	7.3	8.1	30.0	30.5	<1.00	1.58	
A.abdita Control	19.5	20.1	7.82	8.11	7.3	8.4	30.0	30.5	<1.00	1.78	

<u>TABLE D.2</u>. Water Quality Summary for 10-Day, Static-Renewal, Benthic Acute Toxicity Test with *A. abdita*, Liberty Island Anchorage

(a) Ammonia is measured in overlying water.

(b) NA Not applicable.

(c) Data point is outside of acceptable range.

D.2

Cadmium Concentration		(2)	Dead	Proportion	Mean Proportion	Standard
mg/L	Replicate	Live ^(a)	or Missing	Surviving	Surviving	Deviation
0.00	1	20	0	1.00		
0.00	2	18	2	0.90	0.95	0.05
0.00	3	19	1	0.95		
0.19	1	16	4	0.80		
0.19	2	17	3	0.85	0.83	0.03
0.19	3	17	3	0.85		
0.38	1	12	8	0.60		
0.38	2	11	9	0.55	0.63	0.10
0.38	3	, 15	5	0.75		
0.75	1	5	15	0.25		
0.75	2	8	12	0.40	0.35	0.09
0.75	3	8	12	0.40		
1.50	1	0	20	0.00		
1.50	2	0	20	0.00	0.03	0.06
1.50	3	2	18	0.10		
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<u>TABLE D.3</u>. Results of *A. abdita* 96-Hour Cadmium Reference Toxicant Test Liberty Island Anchorage

(a) Survival based on initial exposure of 20 organisms per replicate.

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			Dissolved						
Cadmium Temperature					Sal	inity			
Concentration		<u>`C</u>	-	pH	H (mg/L		0/	00	
mg/L	Min	Max	Min	Max	Min	Max	Min	Max	
Acceptable Range	18.0	22.0	7.30	8.30	<u>≥</u> 4.6	NA ^(a)	28.0	32.0	
nange	10.0	22.0	7.00	0.00	24.0		20.0	02.0	
0.00	19.3	20.5	7.93	8.11	6.6	7.6	30.0	30.5	
0.19	19.5	20.3	7.90	8.17	7.0	7.6	30.0	30.5	
0.38	19.3	20.3	7.85	8.14	6.7	7.5	30.5	31.0	
0.75	19.3	20.4	7.66	8.06	6.9	7.6	30.5	31.0	
1.50	19.5	20.3	7.50	8.07	6.8	7.6	30.0	31.0	

TABLE D.4. Water Quality Summary for A. abdita 96-Hour Cadmium Reference Toxicant Test, Liberty Island Anchorage

(a) NA Not applicable

					Mean	
Sediment			Dead or	Proportion	Proportion	Standard
Treatment	Replicate	Live ^(a)	Missing	Surviving	Surviving	Deviation
						<u></u>
COMP LI	1	19	1	0.95		
COMP LI	2	20	0	1.00		
COMP LI	3	19	1	0.95		
COMP LI	4	18	2	0.90		
COMP LI	5	19	1	0.95	0.95	0.04
C-SB	1	19	1	0.95		
C-SB	2	19	1	0.95		
C-SB	3	18	2	0.90		
C-SB	4	20	0	1.00		
C-SB	5	17	3	0.85	0.93	0.06
R-MUD	1	17	3	0.85		
R-MUD	2	16	4	0.80		
R-MUD	3	20	0	1.00		
R-MUD	4	17	З	0.85		
R-MUD	5	18	2	0.90	0.88	0.08

<u>TABLE D.5</u>. Results of *M. bahia* 10-Day, Static Renewal, Benthic Acute Toxicity Test, Liberty Island Anchorage

(a) Survival based on initial exposure of 20 organisms per replicate.

				Dissolved					Total		
	Tempe	erature			Оху	Oxygen		Salinity		Ammonia ^(a)	
Sediment	i	(°C)	р	H	(m	g/L)	(o/	00)	(mg	;/L)	
Treatment	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	
Acceptable Range	18.0	22.0	7.30	8.30	>3.0	NA ^(b)	28.0	32.0	NA	20.0	
COMP LI	19.3	19.8	7.52	8.09	5.9	7.8	30.0	32.0	<1.00	2.14	
R-MUD	19.3	19.8	7.60	8.01	6.50	8.10	30.0	31.5	<1.00	1.80	
M. bahia Control	19.3	19.8	7.63	8.08	5.80	7.90	30.0	31.0	<1.00	2.30	

TABLE D.6. Water Quality Summary for 10-Day Benthic Acute Toxicity Test with *M. bahia*, Liberty Island Anchorage

(a) Ammonia measured in overlying water.

(b) NA Not applicable.

D.6

Copper Concentration (µg/L)	Replicate	Live ^(b)	Dead or Missing	Proportion Surviving	Mean Proportion Surviving	Standard Deviation
0	1	, 10	0	1.00		
0	2	10	0	1.00	1.00	0.00
100	1	10	0	1.00		
100	2	9	1	0.90	0.95	0.07
150	1	10	0	1.00		
150	2	10	0	1.00	1.00	0.00
200	1	8	2	0.80		
200	2	9	1	0.90	0.85	0.07
300	1	4	6	0.40		
300	2	3	7	0.30	0.35	0.07

TABLE D.7. Results of 96-Hour M. bahia Copper Reference Toxicant Test^(a), Liberty Island Anchorage

(a) This test was used to assess mysid sensitivity for the 10-day Benthic Acute Toxicity Test.(b) Survival based on initial exposure of 10 organisms per replicate.

					Diss	olved		
Copper	Tempe	erature			Oxy	/gen	Sal	inity
Concentration	(°	C)	р	Н	(m	g/L)	(0/00)	
(µg/L)	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable								
Range	18.0	22.0	7.30	8.30	<u>≥</u> 3.0	NA ^(b)	28.0	32.0
0	19.2	19.6	7.53	7.94	5.9	8.1	30.5	31.0
100	19.3	19.7	7.57	7.89	6.1	8.0	30.5	31.5
150	19.2	19.6	7.55	7.92	7.0	7.9	30.5	31.5
200	19.3	19.6	7.73	7.91	7.0	7.9	31.0	31.0
300	19.2	19.6	7.57	7.93	7.1	8.1	30.5	31.0

TABLE D.8. Water Quality Summary for M. Bahia 96-Hour Copper Reference Toxicant Test^(a), Liberty Island Anchorage

(a) This test was used to assess mysid sensitivity for the 10-day Solid-Phase test.(b) NA Not applicable.

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Appendix E.

Bioaccumulation Test Data, Liberty Island Anchorage

Sediment Treatment	Replicate	Live ^(a)	Dead or Missing	Proportion Surviving	Mean Proportion Surviving	Standard Deviation
COMP LI	1	25	0	1.00		
COMP LI	2	25	0	1.00		
COMP LI	3	24	1	0.96		
COMP LI	4	25	0	1.00		
COMP LI	5	24	1	0.96	0.98	0.02
	5	24	•	0.90	0.90	0.02
R-MUD	1	22	3	0.88		
R-MUD	2	24	1	0.96		
R-MUD	3	24	1	0.96		
R-MUD	4	21	4	0.84		
R-MUD	5	24	1	0.96	0.92	0.06
C-SB	1	23	2	0.92		
C-SB	2	22	3	0.88		
C-SB	3	24	1	0.96		
C-SB	4	23	2	0.92		
C-SB	5	25	0	1.00	0.94	0.05

TABLE E.1. Results of 28-Day *M. nasuta* Bioaccumulation Test, Liberty Island Anchorage

(a) Survival based on initial exposure of 25 organisms per replicate

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		•		Diss	olved				
	Tempe	rature			Oxygen Salinity				
Sediment	°C		pН		(mg	/L)	(0/00)	
Treatment	Min	Max	Min	Max	Min	Max	Min	Max	
Acceptable Range	12.0	16.0	7.30	8.30	<u>≻</u> 5	NA ^(a)	28.0	32.0	
COMP LI	14.5	16.0	7.68	8.11	7.1	9.0	30.0	31.5	
R-MUD	14.4	15.6	7.71	8.14	7.0	9.3	30.0	31.5	
C-SB	14.5	15.7	7.66	8.12	7.5	9.4	30.0	31.0	

<u>TABLE E.2.</u> Water Quality Summary for *M. nasuta* 28-Day Bioaccumulation Test,. Liberty Island Anchorage

(a) NA Not applicable.

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Copper Concentration (mg/L)	Live ^(a)	Dead or Missing	Proportion Surviving
0	10	<i>,</i> 0	1.00
0.25	9	, U 1	0.90
		-	
0.50	8	2	0.80
0.75	10	0	1.00
1.00	6	4	0.60
1.50	7	3	0.70
2.50	5	5	0.50

TABLE E.3. Results of *M. nasuta* 96-Hour Copper Reference Toxicant Test, Liberty Island Anchorage

(a) Survival based on initial exposure of 10 organisms per concentration

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Max.
32.0
30.5
30.5
30.5
30.5
30.5
30.5
30.5
32 30 30 30 30 30 30

<u>TABLE E.4</u>. Water Quality Summary for *M. nasuta* 96-Hour Copper Reference Toxicant Test, Liberty Island Anchorage

(a) NA Not applicable.

				•		
o " ·				—	Mean	
Sediment		(-)	Dead or	Proportion	Proportion	Standard
Treatment	Replicate	Live ^(a)	Missing	Surviving	Surviving	Deviation
COMP LI	1	25	0	1.00		
COMP LI	2	25	0	1.00		
COMP LI	3	25	0	1.00		
COMP LI	4	25	0	1.00		
COMP LI	5	25	0	1.00	1.00	0.00
R-MUD	1	25	0	1.00		
R-MUD	2	25	0	1.00		
R-MUD	3	25	0	1.00		
R-MUD	4	25	0	1.00		
R-MUD	5	25	0	1.00	1.00	0.00
C-SB	1	25	0	1.00		
C-SB	2	25	0	1.00		
C-SB	3	25	0	1.00		
C-SB	4	25	0	1.00		
C-SB	5	25	0	1.00	1.00	0.00

<u>TABLE E.5</u>. Results of *T. japonica* 28-Day Bioaccumulation Test, Liberty Island Anchorage

(a) Survival based on initial exposure of 25 organisms per replicate.

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			Dissolved							al
	Tempe	rature			Oxy	gen	Sali	inity	/ Ammonia ^(*)	
Sediment	°C		р	H	(mg	g/L)	(0)	/00)	(mg	/L)
Treatment	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	≥5	NA ^(b)	28.0	32.0	NE ^(c)	NE
COMP LI	14.5	16.0	6.51 ^(d)	8.09	7.4	9.1	30.0	31.5	2.76	2.76
R-MUD	14.6	16.3	7.37	8.11	7.5	8.9	30.5	31.0	<1.0	<1.0
C-SB	14.6	16.0	7.81	8.12	7.3	9.1	30.0	31.0	<1.0	<1.0

TABLE E.6. Water Quality Summary for *T. japonica* 28-Day Bioaccumulation Test, Liberty Island Anchorage

(a) Ammonia measured in overlying water.(b) NA Not applicable.

(c) NE Not established.

(d) Data point out of range.

E.6

Copper				
Concentration		Dead or	Proportion	
(mg/L)	Live(a)	Missing	Surviving	
0	10	0	1.00	
-	10	Ũ	1.00	
0.25	10	0	1.00	
0.50	10	0	1.00	
0.75	10	0	1.00	
1.00	10	0	1.00	
1.50	10	0	1.00	
2.50	10	0	1.00	

<u>TABLE E.7</u>. Results of *T. japonica* 96-Hour Copper Reference Toxicant Test, Liberty Island Anchorage

(a) Survival based on initial exposure of 10 organisms for each concentration.

E.7

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					Diss	olved		
Copper	Tem	perature			Oxy	/gen	Sal	inity
Concentration		(°C)		pН	(m	g/L)	(o/	00)
(mg/L)	<u>Min.</u>	Max.	Min.	Max.	Min.	Max.	Min.	Max.
Acceptable								
Range	13.0	17.0	7.30	8.30	<u>≥</u> 5	NA ^(a)	28.0	32.0
0.00	14.7	15.9	7.66	8.09	7.5	8.7	30.5	30.5
0.25	14.7	15.7	7.91	8.05	7.6	8.9	30.5	30.5
0.50	14.8	15.7	7.85	8.02	7.6	8.8	30.5	30.5
0.75	14.8	15.9	7.78	8.02	7.6	8.7	30.5	30.5
1.00	14.8	15.7	7.81	8.03	5.3	8.8	30.5	30.5
1.50	14.7	15.7	7.51	8.01	7.2	8.5	30.5	30.5
2.50	14.7	15.8	7.56	8.02	7.5	8.5	30.5	30.5

<u>Table E.8</u>. Water Quality Summary for *T. japonica* 96-Hour, Copper Reference Toxicant Test, Liberty Island Anchorage

(a) NA Not applicable.

E.8

	•				Mean	
			Dead or	Proportion	Proportion	Standard
Treatment	Replicate	Live ^(a)	Missing	Surviving	Surviving	Deviation
COMP LI	1	22	3	0.88		
COMP LI	2	25	0	1.00		
COMP LI	3	25	0	1.00	0.97	0.052
COMP LI	4	25	0	1.00		
COMP LI	5	24	1	0.96		
R-MUD	1	25	0	1.00		
R-MUD	.2	25	0	1.00		
R-MUD	3	24	1	0.96	0.99	0.018
R-MUD	4	25	0	1.00		
R-MUD	5	25	0	1.00		
C-NER	1	25	0	1.00		
C-NER	2	25	0	1.00		
C-NER	3	24	1	0.96	0.98	0.022
C-NER	4	25	0	1.00		
C-NER	5	24	1	0.96		

TABLE E.9. Results of *N. virens* 28-Day Bioaccumulation Test, Liberty Island Anchorage

(a) Survival based on initial exposure of 25 organisms per replicate

Sediment	Temp °C	erature	p	н	-	olved /gen g/L)	Salinity (o/oo)	
Treatment	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	≥5	NA ^(a)	28.0	32.0
COMP LI	19.0	20.1	7.48	8.06	6.2	8.2	30.0	31.0
R-MUD	17.6	20.1	7.68	8.10	6.8	8.2	30.0	31.0
C-NER	19.0	20.1	6.78	8.20	6.8	8.3	30.0	31.0

<u>TABLE E.10</u>. Water Quality Summary for 28-Day *N.virens* Bioaccumulation Test, Liberty Island Anchorage

Copper Concentration (mg/L)	Live ^(a)	Dead or Missing	Proportion Surviving
0.00	8	2	0.80
0.050	10	0	1.00
0.075	10	0	1.00
0.150	0	10	0.00
0.200	3	7	0.30
0.250	10	0	1.00
0.350	0	10	0.00

<u>TABLE E.11</u>. Results of *N. virens* 96-Hour, Copper Reference Toxicant Test, Liberty Island Anchorage

(a) Survival based on initial exposure of 10 organisms per replicate.

Copper Concentration	(°	perature C)	р	H	•	olved /gen g/L)		inity oo)
(mg/L)	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
Acceptable								
Range	13.0	17.0	7.30	8.30	≥5	NA ^(a)	28.0	32.0
0.000 0.050 0.075 0.150 0.200 0.250	18.0 18.1 18.1 18.2 18.7 18.5	19.6 19.6 19.6 19.6 19.5 19.4	7.7 7.8 7.7 7.5 7.6 7.8	8.0 8.0 7.9 8.0 8.0 8.0	7.1 7.3 7.4 6.3 7.2 7.3	8.6 8.6 8.7 8.6 8.2 8.4	30.5 30.5 30.5 30.5 30.5 30.5	30.5 30.5 30.5 30.5 30.5 30.5
0.300	18.4	19.3	7.7	7.9	7.4	8.4	30.5	30.5

<u>Table E.12</u>. Water Quality Summary for *N.virens* 96-Hour, Copper Reference Toxicant Test, Liberty Island Anchorage

(a) NA Not applicable.