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Evaluation of Older Bay Mud Sediment from Richmond Harbor, California

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FROM RICHMOND HARBOR, CALIFORNIA**

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SUMMARY

The older bay mud (OBM) unit predates modern man and could act as a barrier to the downward transport of contaminants from the younger bay mud (YBM) because of its hard-packed consistency. However, its chemical and biological nature have not been well characterized. Battelle/Marine Sciences Laboratory (MSL) conducted three independent studies of OBM sediment in January 1993, January 1994, and October 1994. These studies evaluated potential chemical contamination and biological effects of OBM that could occur as a result of dredging and disposal activities. These evaluations were performed by conducting chemical analysis, solid-phase toxicity tests, suspended-particulate-phase (SPP) toxicity tests, and bioaccumulation tests on the OBM sediment. If the sediment chemistry and toxicity results showed no or minimal contamination and toxicological responses, then either the OBM could be left exposed in Richmond Harbor after dredging the YBM without leaving a source of contamination, or if the project depths necessitate, the OBM would be acceptable for disposal at an appropriate disposal site.

In January 1993, OBM sediment was collected from six locations in Richmond Harbor. The six samples were combined in a single composite, OBM COMP. Chemical analyses of the OBM COMP and the three native controls (West Beach control [C-WB], Sequim Bay control [C-SB], and San Pablo Bay control [C-SPB]) consisted of U.S. Environmental Protection Agency (EPA) priority pollutant polynuclear aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), chlorinated pesticides, metals, butyltins (tributyltin [TBT] and dibutyltin [DBT]), and conventional sediment parameters. In conjunction with the chemical inventory, a biological evaluation was performed using a solid-phase acute toxicity test with *Rhepoxynius abronius*, two SPP toxicity tests with *Mytilus galloprovincialis* larvae and *Holmesimysis costata*, and one bioaccumulation potential test with *Macoma nasuta*.

In January 1994, OBM sediment was collected from 23 selected sites in Richmond Harbor. The sediment from the 23 sites was combined into a single composite, OBM COMP. The intent of this study was to confirm the bioaccumulation of tributyltin in the tissues of *M. nasuta* that was observed in the 1993 study and to perform a more in-depth toxicological analysis of OBM sediment. The OBM COMP and the three reference sediments (the Deep Off-Shelf Reference Area [R-OS], the Bay Farm Borrow Area [R-BF], and the Alcatraz Environs Reference Area [R-AM]) were chemically analyzed for PCBs, chlorinated pesticides, and butyltins. The control sediment was archived for future potential analysis. The OBM COMP was tested in three acute toxicity solid-phase tests using *R. abronius*, *M. nasuta*, and *Nephtys caecoides*. The SPP prepared from the OBM COMP was exposed to *Citharichthys stigmaeus*, *M. galloprovincialis* larvae, and *H. costata*. *M. nasuta* were exposed to

the OBM COMP to measure the bioaccumulation potential in the tissues after a 28-day exposure period. The tissues were then analyzed for PCBs, chlorinated pesticides, and butyltins.

In October 1994, OBM sediment was collected from four sites in Richmond Harbor. As in the previous studies, sediment was combined in a single composite. The focus of this study was to examine the low survival of *N. caecoides* exposed to the OBM sediment collected in January 1994. It is suspected that the hard-packed nature of the OBM makes it difficult for *N. caecoides* to burrow, and it was further suspected that the low concentration of total organic carbon (TOC) represented an inadequate food supply. The OBM COMP and the three reference sediments (R-OS, R-BF, and R-AM) were chemically analyzed for the same parameters as those in the January 1993 study. The control sediment was archived for future potential analysis. The OBM COMP was tested in an acute toxicity solid-phase test using *N. caecoides* in which the OBM COMP sediment was enriched with different food sources and softened with seawater prior to the addition of test organisms.

The results of the *R. abronius* test showed that the OBM COMP from the 1993 study was not acutely toxic relative to any reference treatment. The OBM COMP tested in 1994 was acutely toxic to *R. abronius* relative to both of the in-bay reference treatments. The OBM COMP was not acutely toxic to *M. nasuta* relative to any reference treatment. The results of the *N. caecoides* test showed that the OBM COMP from the 1994 study was acutely toxic when compared with all three reference treatments. The feeding studies conducted in October 1994 showed that this toxicity was caused by a combination of the hard-packed nature of the OBM sediment and its relatively low TOC content. When the OBM was softened with seawater, survival increased to 65%; however, the sediment was still acutely toxic to *N. caecoides* relative to all three reference treatments. However, by first softening the OBM sediment and then amending with food, survival increased and the sediment was not considered acutely toxic to *N. caecoides* relative to any reference treatment.

Estimates of water column toxicity were evaluated by exposing *M. galloprovincialis*, *C. stigmaeus*, and *H. costata* to three concentrations of SPP and a dilution water control (Sequim Bay seawater). Acute toxicity was determined by statistical comparison of the 0% and 100% SPP using a t-test and calculations of LC₅₀ and EC₅₀ values using the Trimmed Spearman-Kärber estimator. To calculate LC₅₀ or EC₅₀ values, there must be greater than 50% mortality or some other sublethal effect occurring in the sediment treatments. There was not a 50% decrease in survival for any test species or a 50% decrease in the percentage of normal larvae for *M. galloprovincialis* test.

The potential for bioaccumulation of contaminants was evaluated through a 28-day solid-phase exposure of *M. nasuta* to OBM sediments followed by chemical analysis of the tissues. *M. nasuta* tissue analyses for the 1993 study consisted of PAHs, pesticides, PCBs, metals, and

butyltins. The analyses for the 1994 study consisted of pesticides, PCBs, and butyltins. PAHs and PCBs were not detected in the tissues of *M. nasuta* exposed to OBM COMP from either study relative to any of the reference treatment tissues; therefore, a statistical evaluation was not conducted. For the 1993 study, the concentrations of four pesticides (4,4'-DDD, 4,4'-DDE, 4,4'-DDT, and dieldrin) were statistically significantly elevated in *M. nasuta* tissues exposed to the OBM COMP. This is contrary to the results of the 1994 study, which showed no detectable concentrations of pesticides in *M. nasuta* tissues. All 10 metals were detected in the tissues of *M. nasuta* exposed to the OBM COMP. Four metals were statistically significantly elevated in *M. nasuta* tissues exposed to the OBM COMP relative to at least one reference treatment. The results of the 1993 study showed that both TBT and DBT were statistically significantly elevated in *M. nasuta* tissue exposed to the OBM COMP. The confirmatory tests conducted in 1994 did not reflect the results of the 1993 study. In the 1994 studies, concentrations of TBT and DBT were not statistically significantly elevated in *M. nasuta* tissues exposed to the OBM COMP relative to any reference treatment.

In summary, the two objectives of this study were to 1) determine whether OBM sediments could be left exposed after YBM sediments were dredged, and 2) determine whether OBM is acceptable for disposal at various disposal sites. OBM is primarily composed of silt and hard-packed clay, has a low percentage of TOC, and contains no detectable concentrations of oil and grease, total petroleum hydrocarbons, pesticides, PCBs, and butyltins, and low concentrations of PAHs, and metals, indicating that OBM does not mix with the overlying YBM sediments. The OBM sediments in Richmond Harbor appear to be an effective barrier to the downward transport of contaminants that are associated with the YBM sediments. The OBM sediments did pass all toxicity testing relative to the ocean site. OBM sediments were acutely toxic to *R. abronius* relative to the in-bay sites. This toxicity is probably caused by the physical nature of the sediment. The decisions regarding leaving OBM sediment exposed during dredging operations and determining suitable disposal sites for this sediment will lie with the regulatory agencies.

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1.0 INTRODUCTION

Richmond Harbor lies on the eastern shoreline of central San Francisco Bay in California and has been studied extensively through chemical and biological evaluations, such as *Environmental Evaluations for Deepening of Richmond Harbor and Santa Fe Channels, Task 4: Chemistry Program* (Brown et al. 1990), *Ecological Evaluation of Proposed Dredged Material from Richmond Harbor* (Pinza et al. 1992), *Ecological Evaluation of Richmond Harbor Maintenance Sediments* (Pinza and Word 1993, letter report), and *Ecological Evaluation of Proposed Dredged Material from Richmond Harbor Deepening Project* (Pinza et al. 1995). These studies have evaluated the potential chemical contamination and biological effects of younger bay mud (YBM), which is one of the two main geologic units in the area. The YBM is underlain by the more compacted older bay mud (OBM). These two geologic units can be distinguished on the basis of consistency and color. The firm to hard, very dry OBM sediments include both estuarine and terrestrial deposits and are various shades of red, brown, yellow, or gray. The YBM sediments are recent marine deposits that are soft and dark gray to black in color.

The OBM unit predates modern man and could act as a barrier to the downward transport of contaminants from the YBM because of its hard-packed consistency. However, its chemical and biological nature have not been well characterized. Battelle/Marine Sciences Laboratory (MSL) conducted three independent studies of OBM sediment in January 1993, January 1994, and October 1994. These studies evaluated potential chemical contamination and biological effects of OBM that could occur as a result of dredging and disposal activities. These evaluations were performed by conducting chemical analysis, solid-phase toxicity tests, suspended-particulate-phase (SPP) toxicity tests, and bioaccumulation tests on the OBM sediment. If the sediment chemistry and toxicity results showed no or minimal contamination and toxicological responses, then either the OBM could be left exposed in Richmond Harbor after dredging the YBM without leaving a source of contamination, or if the project depths necessitate, the OBM would be acceptable for disposal at an appropriate disposal site.

Test organism selections followed the guidelines in the *Evaluation of Dredged Material Proposed for Ocean Disposal (Testing Manual)* (EPA/USACE 1991), also referred to as the *1991 Implementation Manual*. The species chosen for acute toxicity solid-phase tests were the infaunal burrowing amphipod, *Rhepoxynius abronius*, the polychaete, *Nephtys caecoides*, and the bentnose clam, *Macoma nasuta*. Larvae of the bivalve, *Mytilus galloprovincialis*, the mysid, *Holmesimysis costata*, and the juvenile speckled sanddab, *Citharichthys stigmaeus*, were used for the SPP testing. The bentnose clam, *M. nasuta*, was chosen as the test species to measure potential OBM contaminant availability. These species have been successfully used in other programs conducted by the MSL.

The purpose of the solid-phase tests was to examine the potential acute toxicity of the OBM to benthic organisms. The SPP toxicity tests were designed to measure the effects of any suspended and dissolved contaminants on water column organisms after allowing for initial mixing of the proposed dredged material. The bioaccumulation of contaminants in the tissues of *M. nasuta* measured the potential availability of any polynuclear aromatic hydrocarbons (PAHs), chlorinated pesticides, polychlorinated biphenyls (PCBs), metals, and butyltins. Validation of test results is accomplished by observing the response of the test organisms exposed to their native control sediment.

In January 1993, OBM sediment was collected from six locations in Richmond Harbor (Figure 1.1). The six samples were combined in a single composite, OBM COMP. Chemical analyses of the OBM COMP and the three native controls (West Beach control [C-WB], Sequim Bay control [C-SB], and San Pablo Bay control [C-SPB]) consisted of U.S. Environmental Protection Agency (EPA) priority pollutant PAHs, PCBs, chlorinated pesticides, metals, butyltins, and conventional sediment parameters. In conjunction with the chemical inventory, a biological evaluation was performed using a solid-phase acute toxicity test with *R. abronius*, two SPP toxicity tests with *M. galloprovincialis* larvae and *H. costata*, and one bioaccumulation potential test with *M. nasuta*.

In January 1994, OBM sediment was collected from 23 selected sites in Richmond Harbor as shown in Figure 1.1. The sediment from the 23 sites was combined into a single composite, OBM COMP. The intent of this study was to confirm the bioaccumulation of tributyltin in the tissues of *M. nasuta* that was observed in the 1993 study and to perform a more in-depth toxicological analysis of OBM sediment. The OBM COMP and the three reference sediments (the Deep Off-Shelf Reference Area [R-OS], the Bay Farm Borrow Area [R-BF], and the Alcatraz Environs Reference Area [R-AM]) were chemically analyzed for PCBs, chlorinated pesticides, and butyltins. The control sediment was archived for future potential analysis. The OBM COMP was tested in three acute toxicity solid-phase tests using *R. abronius*, *M. nasuta*, and *N. caecoides*. The SPP prepared from the OBM COMP was exposed to *C. stigmaeus*, *M. galloprovincialis* larvae, and *H. costata*. *M. nasuta* were exposed to the OBM COMP to measure the bioaccumulation potential in the tissues after a 28-day exposure period. The tissues were then analyzed for PCBs, chlorinated pesticides, and butyltins.

In October 1994, OBM sediment was collected from four sites in Richmond Harbor as shown in Figure 1.1. As in the previous studies, sediment was combined in a single composite. The focus of this study was to examine the low survival of *N. caecoides* exposed to the OBM sediment collected in January 1994. It is suspected that the hard-packed nature of the OBM makes it difficult for *N. caecoides* to burrow, and it was further suspected that the low concentration of total organic carbon (TOC) represented an inadequate food supply. The OBM

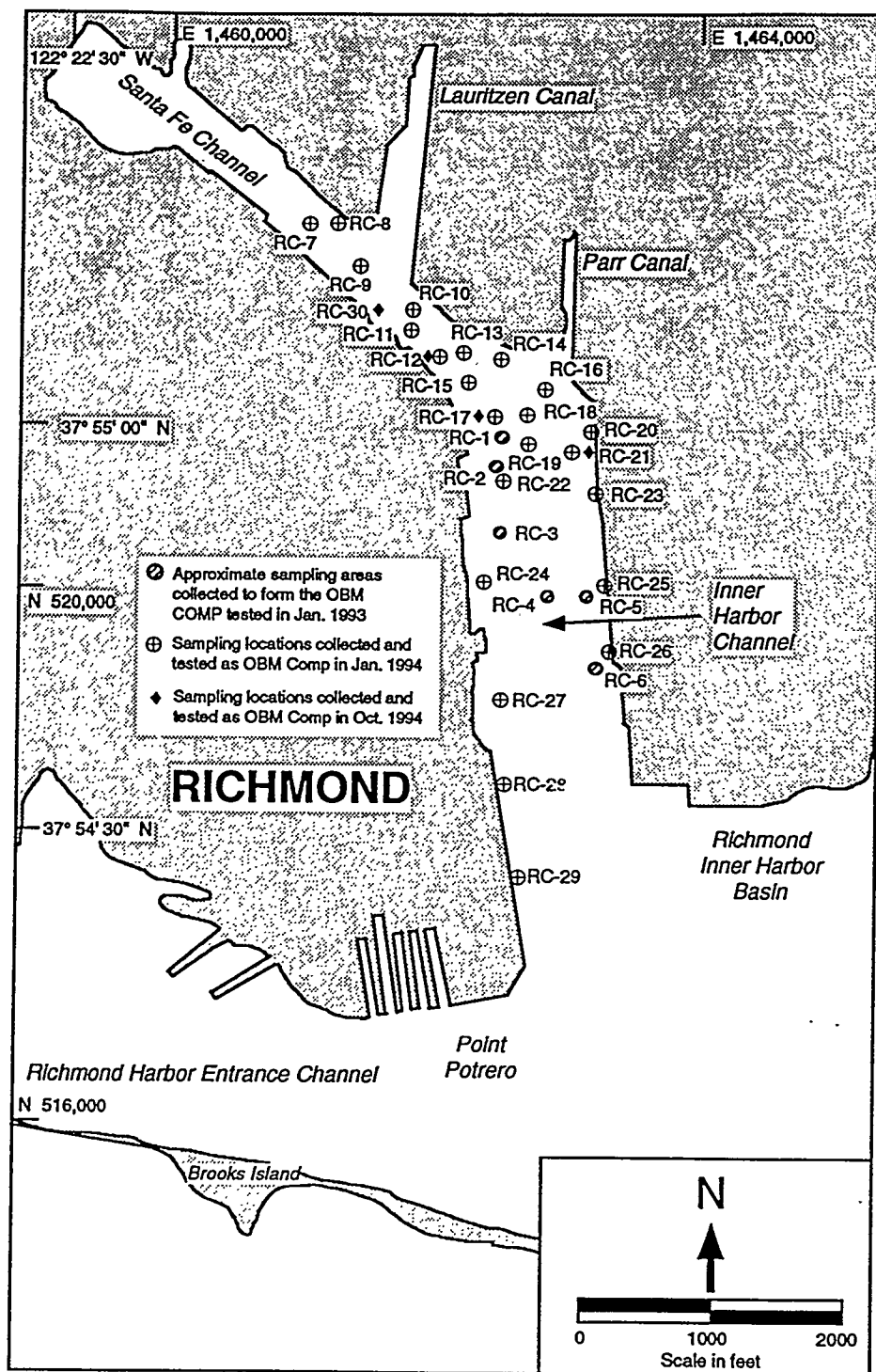


FIGURE 1.1. Site Location Map

COMP and the three reference sediments (R-OS, R-BF, and R-AM) were chemically analyzed for the same parameters as those in the January 1993 study. The control sediment was archived for future potential analysis. The OBM COMP was tested in an acute toxicity solid-phase test using *N. caecoides* in which the OBM COMP sediment was enriched with different food sources and softened with seawater prior to the addition of test organisms.

This report describes the characteristics of the OBM sediments from the three studies and is divided into five sections. Section 1.0 is the introduction, containing a brief overview of the project. Section 2.0 describes the materials and methods used for sample preparation and handling, biological testing, physical and chemical analyses of sediments and tissues, data analysis, and quality assurance requirements. Section 3.0 presents the results of the physical and chemical sediment chemistry analyses and the biological toxicity/bioaccumulation testing for the three OBM composites. Section 4.0 is the discussion of the results and conclusions. Section 5.0 lists the literature cited in support of this document. Appendixes containing detailed data for the following areas are presented as follows:

- Appendix A Sediment Chemistry and Quality Control Data
- Appendix B-H Toxicity Test Data
- Appendix I *M. nasuta* Tissue Chemistry and Quality Control Data.

2.0 MATERIALS AND METHODS

2.1 SEDIMENT AND TEST ORGANISM COLLECTION

Sediment samples were collected from Richmond Harbor during January 1993, January 1994, and October 1994. After each field collection, OBM sediment used for each study was submitted for chemical and biological analysis.

2.1.1 Test Sediment Collection

The January 1993 study included sampling locations for the Inner Channel of Richmond Harbor (RC-1 through RC-6). Samples of sediments were obtained using a 4-in. diameter vibratory hammer core and were stored in the noncontaminating Lexan polycarbonate tubes contained in the core barrel. These core tubes maintain the stratigraphic integrity of the sediments and allowed a geologist to vertically characterize the sediment types present at each field station. Selected sites in Richmond Harbor (RC-7 through RC-29) were sampled for the January 1994 study using a 12-in. diameter vibratory hammer core. The OBM from these sites was taken from the 12-in. core barrel and placed into epoxy coated buckets. The sediments collected for the October 1994 study (RC-12, RC-17, RC-21, and RC-30) were obtained using a 4-in. core barrel. Sediments were maintained at approximately 4°C during shipment in a refrigerated van to Sequim, Washington. Upon arrival at the MSL, sediments were transferred to a climate-controlled cold room and maintained at 4°C until processed for chemical analysis and toxicological testing.

2.1.2 Reference and Control Sediment Collection

Reference sediment samples R-AM, R-BF and R-OS were collected using a pipe dredge deployed from the *FV Cobra*, a charter boat owned and operated by Bob Smith Sportfishing. Sampling locations were determined by LORAN C and variable fix and range radar systems aboard the vessel and by a Raytheon Global Positioning System (GPS). The vessel navigation systems were verified at known fixed locations, such as the Golden Gate channel pilot buoy. Reference sampling records were maintained in a log book, and consisted of station position, date, time, replicate, water depth, sediment type, and comments. All reference samples were kept in labeled coolers on board the sampling vessel until they were off-loaded to the refrigerated van.

The native control sediment sampling locations were Whidbey Island, Washington, (*R. abronius*), Sequim Bay, Washington (*M. nasuta*), and Tomales Bay/Dillon Beach, California (*N. caecoides*). Control sediment and test organisms from Whidbey Island were collected by MSL personnel using a 19-ft Boston Whaler. The sampling location was determined by reference to shoreline features. Whidbey Island sediment was collected with a small, MSL-designed, anchor-dredge sampler that also obtained sufficient quantities of test organisms. The dredge was

deployed in approximately 15 ft of water. Experimental control sediment from Sequim Bay was collected with a modified van Veen grab sampler (0.1 m²) deployed from an MSL research vessel. The Tomales Bay/Dillon Beach sediment was collected using a shovel, bucket, and sieve. In addition to the three toxicity test native controls, a fine-grained control was collected from San Pablo Bay, California, and was tested during the January 1993 study only. This control was analyzed for sediment chemistry to provide a basis of comparison with the OBM.

2.1.3 Test Organism Collection

Six species of organisms were used to evaluate sediments from the OBM segments of the Richmond Harbor study area:

- the amphipod *Rhepoxynius abronius*
- the polychaete *Nephtys caecoides*
- the bivalve *Macoma nasuta*
- the speckled sanddab *Citharichthys stigmaeus*
- larvae of the mussel *Mytilus galloprovincialis*
- the invertebrate *Holmesimysis costata*.

All test organisms were collected either by a commercial supplier or by MSL personnel. The organisms were shipped in native sediment or in a way designed to ensure their viability. After receipt at the MSL, test organisms were gradually acclimated to test conditions. Animals acting abnormally or exhibiting stress were not used in toxicological tests. *R. abronius* were collected from Whidbey Island and were transported in clean coolers containing approximately 10 cm of sediment and 5 gal of clean seawater at a temperature approximating natural conditions. *N. caecoides* were collected from mud flats in Tomales Bay, and placed into clean coolers containing sediment and seawater from the collection site. Prior to overnight shipment to the MSL, the seawater in each cooler was supersaturated with oxygen (22 ppm). *M. nasuta* were collected from the intertidal zones of beaches in Discovery Bay, near Gardiner, Washington, by Gunstone and Johnson, commercial suppliers. The clams were held in large tanks of clean Sequim Bay sediment with flowing 15°C seawater. *C. stigmaeus* were collected from Tomales Bay, in 12 ft to 15 ft of water, using a small trawl with a 0.25-in. mesh net. The trawl was held close to the work boat so a dip net could be used to directly transfer *C. stigmaeus* into double plastic bags containing oxygen-saturated seawater. *H. costata* were collected by Brezina and Associates with a plankton dip net and transferred to a holding container aboard the work boat, where they were sorted by appropriate age and size class and then shipped to the MSL in bags containing oxygen-saturated water. The *M. galloprovincialis* were purchased from Taylor United, Inc. in Quilcene, Washington. These organisms were wrapped in moist paper towels and shipped in styrofoam coolers containing blue ice to maintain an ambient temperature of approximately 15°C.

2.2 SEDIMENT SAMPLE PREPARATION

Sediment sample preparation involved all steps in the laboratory from delivery of the samples to the MSL to the preparation of sediments for chemical and/or biological testing. Sediment used for biological testing was prepared within the 6-week holding period as specified in the *1991 Implementation Manual*. During this holding time, the samples were received at the MSL, inventoried against chain-of-custody forms, processed for testing, subsampled for sediment chemistry parameters, and used in the biological tests. The following sections describe the methods for geologic description of cores, equipment preparation, compositing strategy, preparation of sediments for chemistry, and solid-phase and SPP testing.

2.2.1 Geologic Description of Individual Core Samples

A geologic description of the sediment contained in the 4-in. cores from each sampling location was conducted by an MSL geologist during the January 1993 and October 1994 studies. The OBM collected in January 1994 was transferred from the 12-in. core barrel to epoxy-coated buckets in the field; therefore, the cores were not described. All 4-in. core sections from one station were removed from storage and the Lexan tube was cut longitudinally with a circular saw. A linoleum knife was used to split the core open, exposing the sediment profile. The geologist measured and described the entire core from mudline to bottom, recording information on a core data log. The geologic characterization protocol was consistent with American Society of Testing Materials (ASTM) Method D2488-84 (ASTM 1984). The OBM segments of each core were removed from the core tube and composited into one sediment treatment for testing.

2.2.2 Laboratory Glassware and Equipment Preparation

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, including test containers, aquaria, and sediment transfer dishes, was washed with warm, soapy water, rinsed five times with deionized water, then soaked in a 5% reagent grade nitric acid bath for a minimum of 4 h. After soaking, glassware was rinsed with deionized water five times and allowed to dry. Polyvinyl chloride (PVC), Nalgene, and Teflon tools were also washed and soaked in acid baths in the same manner as for glassware.

Stainless-steel bowls, spoons, spatulas, and other utensils were washed with warm, soapy water, rinsed five times with deionized water, and allowed to air dry. They were then rinsed with methylene chloride under a fume hood, and the methylene chloride was allowed to evaporate under the hood.

Neoprene stoppers and polyethylene sheets or other porous materials were washed with warm, soapy water and rinsed five times 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 mixer that is used to mix sediment, were washed with a mild soap solution and thoroughly rinsed with tap water followed by deionized water.

Equipment used to determine water quality, such as pH, dissolved oxygen (DO), temperature, salinity, and ammonia were calibrated according to the manufacturer's specifications and internal MSL procedures.

2.2.3 Preparation of Solid-Phase Treatments for Chemical and Biological Testing

Solid phase refers to the sediment itself, as opposed to sediment suspended or dissolved in water. In the biological tests, the solid phase of sediments represents 1) dredged material once it has settled at an aquatic disposal site, or 2) the native environment of a benthic test organism (control sediment). The OBM sediments from the Richmond Harbor sampling sites for each study were homogenized into one sediment composite for each study and evaluated for toxicity relative to the appropriate control treatments. To thoroughly homogenize the OBM COMP for each study, the OBM sediment was grated using a stainless steel grater and then mixed in an epoxy-coated cement mixer until the composite was homogeneous. All solid-phase samples were thoroughly homogenized before use in biological tests or chemical analysis.

Control sediments were press sieved through a 1.0-mm screen to remove predators. Test sediments were processed by combining the sediment from all contributing stations into an epoxy-coated mixer and mixing until a homogenous color and texture were evident. Between mixing of the test and control sediments, all equipment was thoroughly rinsed with 0.45- μ m-filtered seawater to avoid cross-contamination. After compositing, sample aliquots for chemical analyses were placed in cleaned and labeled containers appropriate for the parameters to be measured and a subsample of each sediment treatment was archived. All sediment treatments (test and control) were then placed in precleaned containers that were sealed, labeled and maintained at 4°C $\pm$ 2°C until needed for biological testing.

2.2.4 Preparation of Suspended-Particulate-Phase Samples for Biological Testing

The SPP of sediment samples was used to evaluate potential water-column effects that could occur during dredging or after disposal at open-water dredged material disposal sites. The SPP is the liquid supernatant that remains after mixing sediment with seawater and allowing heavier particles to settle to the bottom. Because the SPP preparation does not involve filtration, this phase contains suspended particles as well as dissolved constituents. The SPP tests evaluate effects caused by both the physical presence of the suspended particles and the chemical toxicity of contaminants associated with the particles or dissolved constituents. The process is intended to approximate exposure conditions created as a result of the disturbance

materials during the dredging process or after discharge through the water column during dredge material disposal operations.

The SPP is prepared by creating a 4:1 (volume:volume) water to sediment slurry in 1-L glass jars with Teflon-lined lids. The jars were marked at 200 mL, 400 mL, and 1 L. Filtered seawater (0.45- μ m) was added to the 200-mL mark, then homogenized sediment was added until the water was displaced to the 400-mL mark. The jar was then filled to 1 L with filtered seawater. Twelve jars of sediment and water were placed on a shaker table and agitated for 30 min at a shaking rate of 120 to 150 cycles/min. After shaking, the slurry was poured into 500-mL Teflon containers with tightly fitting lids. These containers were placed in a centrifuge and spun for 10 to 15 min at a g-force of 777 (equivalent to a 24-h settling period). The centrifugation was necessary to ensure that larval test organisms would be visible at the first microscopic observation after exposure to SPP test treatments. After centrifugation, the supernatant was poured into clean, labeled, 1-gal jars and used in the SPP tests as soon as possible (within 24 h). The Teflon jars were rinsed out after each use and the above process was continued until an adequate amount of SPP was produced for one sediment treatment. Between sediment treatments, the Teflon jars were cleaned as described in Section 2.2.2.

2.3 TOXICOLOGICAL AND BIOACCUMULATION TESTING PROCEDURES

Toxicological tests involving six test species were conducted in support of the three OBM studies. The following solid-phase tests were conducted: a 10-day solid-phase static acute toxicity test using the amphipod *R. abronius*, a 10-day solid-phase flow-through acute toxicity test using *M. nasuta* and *N. caecoides*, and a 10-day solid-phase flow-through acute toxicity test with *N. caecoides* subjected to different concentrations of TOC and food sources. A 28-day solid-phase flow-through test was conducted to evaluate the bioaccumulation potential of OBM contaminants in tissues of the clam *M. nasuta*. Each test had five replicates of OBM sediment plus five replicates of the appropriate native control. Three SPP tests were conducted: 1) a 48-h exposure using the larvae of the mussel *M. galloprovincialis*, 2) a 96-h exposure using the mysid *H. costata*, and 3) a 96-h exposure using the sanddab *C. stigmaeus*. The SPP treatments were prepared as described in Section 2.2.4. There were three replicates of each SPP concentration: 10%, 50%, and 100% (SPP) and three replicates of the 0% SPP (dilution water). All test containers were placed in random positions in the water tables.

The MSL facilities provided the required conditions for flow-through solid-phase tests, static solid-phase tests, and static SPP tests. Laboratory equipment included a controlled-temperature environment, flow-through seawater supply, lighting control, and air supply.

2.3.1 10-Day Solid-Phase Static Test with *R. abronius*

The *R. abronius* were held in a large holding tank containing their native sediment with flowing 15°C seawater. Organisms were not fed during the holding period, which was less than two weeks before test initiation. The *R. abronius* test was conducted in 1-qt static Mason jars, which were placed in random positions on a water table maintained at 15°C. Prior to test initiation, sediment was added to the jars to a depth of 2 cm, then each jar was slowly filled with 0.45- μ m-filtered seawater to a total volume of 750 mL. The jars were placed on the water table overnight to stabilize to test conditions. After settling, initial water quality parameters were measured in each container and recorded on water quality forms. Gentle aeration was applied to each jar, and the test was conducted under 24-h illumination.

The *R. abronius* test was initiated by adding 20 organisms to each test container. The amphipods were gently sieved from the holding tank into clean seawater and counted into small transfer containers. The number of organisms was then confirmed by a second observer before organisms were transferred into the test containers. The *R. abronius* were observed daily during testing, and the number of organisms floating on the surface, swimming in the jar, or on the sediment surface were recorded on observation forms. Amphipods floating on the surface were gently pushed below the water surface with a pipet tip, and observations were made as to whether they remained on the sediment surface, buried below the sediment, or returned to the surface of the water.

For the January 1994 study, two additional tests were set up for each sediment treatment. These additional replicates, referred to as *dummy jars*, were prepared in the same manner as the test containers, with the exception that no organisms were added. The dummy jars were sacrificed to obtain a porewater ammonia measurement; therefore, the porewater ammonia measurements of the sediment treatments were conducted prior to start of the test and on Day 10 (termination).

Water temperature, salinity, pH, and DO were measured daily in one replicate of each sediment treatment, and in all containers at initiation and termination of the test. For the January 1994 study, ammonia was measured in the overlying water on Days 0, 1, 3, 7, and 10 and in the porewater on Days 0 and 10. Acceptable ranges for water quality parameters for the static *R. abronius* test were as follows:

DO	≥ 6.0 mg/L
pH	7.8 ± 0.5 units
Salinity	30 ± 2.0 ‰
Temperature	15 ± 2.0 °C
Ammonia (porewater)	≤ 30 mg/L

At the end of the test (Day 10), the contents of each jar were sieved through a 0.5-mm Nytex screen to collect the *R. abronius*. The organisms were placed in clean seawater in a glass

dish labeled with the sediment treatment and replicate number. The number of live and dead organisms was counted and examined under a dissecting microscope. The presence or absence of body parts recovered at the end of the test was also noted. If an individual organisms did not respond to gentle probing it was considered dead. At least 10% of the mortality counts were confirmed by a second observer.

The cadmium (Cd) reference toxicant test was conducted at the same time as the 10-day test and was used to assess the health and relative sensitivity of the test organisms. The reference toxicant concentration series for each study is as follows: the January 1993 study was 0, 0.5, 1.0, 2.0, and 4.0 mg Cd/L and the January 1994 study was 0, 0.38, 0.75, 1.5, and 3.0 mg Cd/L. The reference toxicant test followed the same testing procedure as the 10-day test.

A 96-h ammonia reference toxicant test was also conducted to establish the expected response of the test organisms to ammonia. The ammonia reference toxicant test was conducted in the same manner as the Cd reference toxicant test. *R. abronius* were exposed to a seawater control plus six nominal concentrations of ammonia (10, 20, 40, 80, 120, and 160 mg/L as ammonium chloride). The actual concentrations of ammonia were measured in this experiment and are reported in Section 3.0 of this report. There were three replicates of each concentration.

2.3.2 10-Day Solid-Phase Flow-Through Test with *M. nasuta* and *N. caecoides*

This test was performed only during the January 1994 study. Prior to testing, *N. caecoides* were held in their native sediment in shallow trays covered with well-aerated 15°C seawater from a gravity-fed flow-through system. *M. nasuta* were held in large water tables or holding tanks containing clean sediment with flow-through 15°C seawater. Temperature, pH, DO, and salinity of water in each holding tank were monitored daily. The organisms were not fed during the holding period.

The flow-through test with *M. nasuta* and *N. caecoides* was conducted in 10-gal aquaria placed in random positions on water tables. Figure 2.1 shows the system used for flow-through tests. Each aquarium was filled with approximately 8 L of sand-filtered seawater via the flow-through system. Then, sediment was added to a depth of 3 cm by measuring the required amount (3870 mL) into a glass container and pouring it into the aquarium while washing it with seawater to distribute the sediment evenly over the bottom. The flow-through system was initiated, and aquaria were allowed to fill to a total volume of approximately 36 L. For 4 h, the sediment in the aquaria was allowed to settle in the absence of flowing seawater. Then the flow-through system was adjusted and calibrated to deliver 125 ± 10 mL/min of seawater flow to each aquarium. The system was allowed to operate overnight before the addition of test organisms.

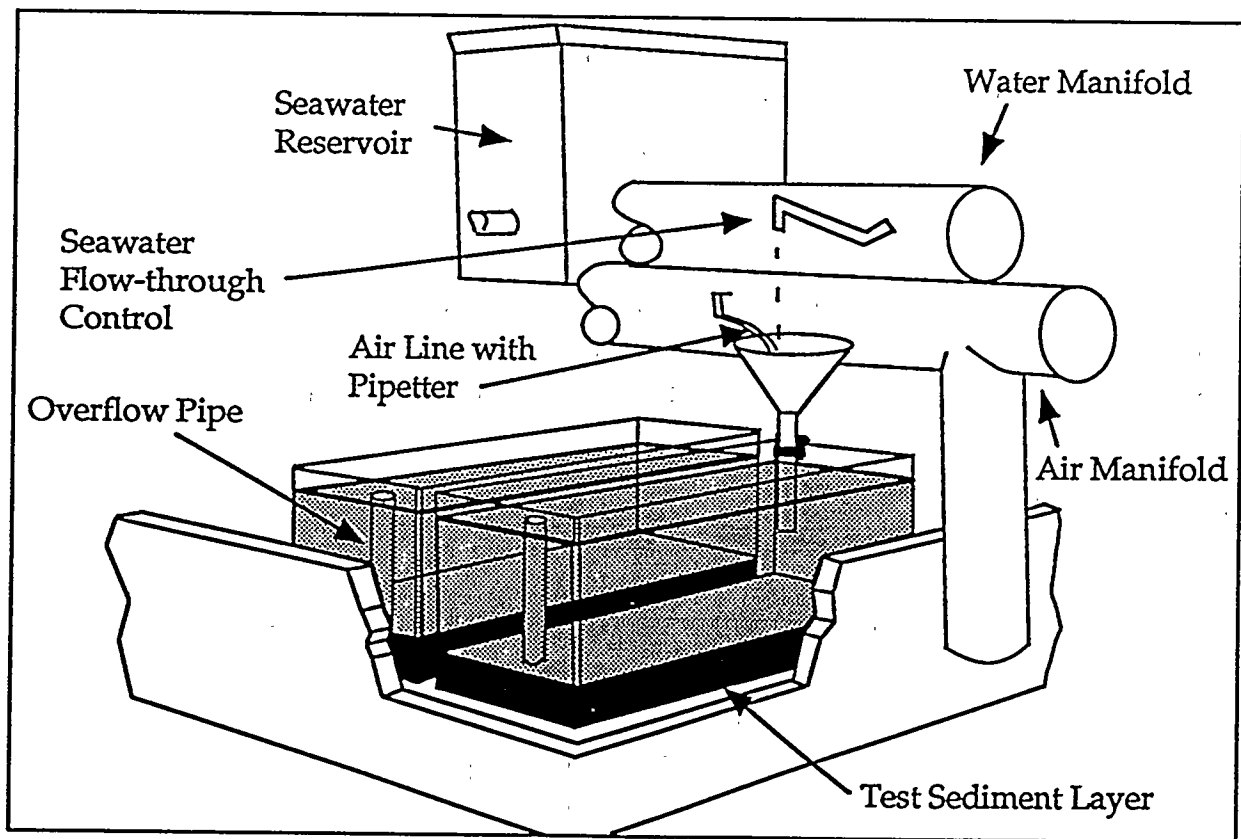


FIGURE 2.1. Flow-Through Aquaria for the 10-Day and the 28-Day *M. nasuta* and *N. caecoides* Toxicity Tests

To initiate the test, 25 *M. nasuta* and 20 *N. caecoides* were collected from the holding tanks and placed in each aquarium. Each test aquarium was labeled with initiation time/date and the initials of the examiner who placed the organisms in each chamber.

Daily observations of test-organism behavior were made and recorded on data forms. The number of *M. nasuta* on the sediment surface and the number of those with their siphons exposed was noted, as well as the number of *N. caecoides* on the sediment surface and the number of those with only their heads exposed. In addition, the number of dead organisms in each aquarium was recorded on daily observation forms. Dead organisms were removed from the aquaria and incinerated; no replacement of dead organisms occurred during testing. If any dead *N. caecoides* was removed, it was identified as a whole animal, a head, or a tail portion.

Water quality parameters (below) were measured daily in at least one replicate of each sediment treatment and recorded on water quality data sheets. The water quality parameters and ranges established for the tests were as follows:

DO	≥6.0 mg/L
pH	7.30-8.30
Salinity	30‰ ±2.0‰
Temperature	15.0°C ±2.0°C
Flow Rates	125 ±10 mL/min
Ammonia	measured but presently not established.

A replicate of each sediment treatment dummy jar was used to measure the interstitial porewater ammonia of the sediment treatments prior to initiation of the organisms and on Day 10 (termination).

At the end of the 10-day test, water quality measurements were performed in all replicates, and the contents of each aquarium were gently passed through a 1.0-mm Nytex screen to recover the *N. caecoides* and *M. nasuta*. The organisms were placed in glass dishes and labeled with the sediment treatment number. At this time, the number of dead and live organisms of each species was counted. Death was determined by observing whether the *N. caecoides* reacted to gentle probing; if there was no movement, the organism was considered dead. Death of *M. nasuta* was determined by the presence of gaping shells. The mortality data were recorded on the termination forms. At least 10% of the mortality counts were confirmed by a second observer.

2.3.3 28-Day Solid-Phase Flow-Through Test with *M. nasuta*

The procedure for conducting the 28-day solid-phase flow-through test with *M. nasuta* was identical to that of the 10-day test, with three exceptions: 1) only *M. nasuta* were exposed in the bioaccumulation test; 2) the exposure period was increased from 10 to 28 days; 3) the surviving test organisms were depurated, and the tissues were sampled for chemical analysis. The ranges for water quality parameters, as well as the test conditions, such as temperature and

flow rate, were the same for the 28-day test as for the 10-day test. Water quality parameters were measured, and mortality of the test organisms monitored at the same frequency for both tests.

When the 28-day test was terminated, the living *M. nasuta* were collected for chemical evaluation of bioaccumulation. To ensure that tissue chemistry results would not be biased by contaminants associated with sediment grains in the digestive tract, the test organisms were allowed to depurate, or void the digestive tract, for 48 h following the 28-day exposure. The surviving *M. nasuta* were placed in a glass dish (without sediment), and placed in an aquarium with flowing seawater. After 48 h of depuration, the *M. nasuta* shells were cleaned with a scrub brush prior to the removal of tissues using titanium knives. The tissues were placed into clean jars and submitted for chemical analysis.

2.3.4 48-Hour Suspended-Particulate-Phase Static Test with *M. galloprovincialis*

Mussels 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 SPP (0%, 10%, 50%, and 100%) were made with Sequim Bay seawater and prepared individually for each test replicate. After all chambers reached testing temperatures ($16^{\circ}\text{C} \pm 2^{\circ}\text{C}$), initial water quality parameters were measured in all replicates. Test chambers were placed in random positions on a water table and provided with gentle aeration.

Spawning was induced by placing adult *M. galloprovincialis* into 15°C , filtered Sequim Bay seawater and then rapidly raising the holding water temperature to 20°C . Spawning generally occurred within 1 h of temperature elevation. Spawning males and females were isolated in individual jars with filtered Sequim Bay seawater and allowed to shed gametes for approximately 45 min. Eggs from each female were then filtered into separate jars through a $75\text{ }\mu\text{m}$ Nytex screen 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 the individual egg stocks. Egg-sperm solutions were mixed every 10 min with a perforated plunger. Fertilization proceeded for 1 h, then the fertilization rate (percentage fertilized) was determined by removing a subsample and observing the number of multi-cell stage embryos. Fertilization was considered successful if greater than 90% of the embryos were in the multi-cell stage. To prevent polyspermy, egg stocks with greater than 90% fertilization were rinsed on a $20\text{-}\mu\text{m}$ Nytex screen to remove excess sperm. Embryo stock-solution density was estimated by removing a 0.1 mL subsample and counting all multi-cell 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 10% formaldehyde to determine actual stocking density. Water quality parameters were measured in one replicate per treatment daily throughout the test. Acceptable ranges for water quality parameters were as follows:

DO	≥ 4.0 mg/L
Temperature	$16^{\circ}\text{C} \pm 2^{\circ}\text{C}$
pH	7.30 to 8.30
Salinity	$30 \pm 2.0\%$
Ammonia	measured but presently not established.

The bivalve test was terminated after 48 h, when greater than 90% of the larvae in the controls had reached the D-cell stage (prodissococonch I). Final water quality parameters were recorded for all replicates. In addition, ammonia concentrations were measured in one replicate of all 0% SPP and 100% SPP treatments.

Each chamber was then homogenized with a perforated plunger, and a 10-mL subsample was removed and placed into 20-mL scintillation vial. The subsample was then fixed with 1 mL of 50% formaldehyde in seawater. Samples were scored for the appearance of normal D-shaped larvae (prodissococonch I), abnormal prodissococonch I larvae, developmentally delayed/abnormal larvae, and total number of larvae. At least 10% of the counts were confirmed by a second observer.

A 48-h copper (Cu) reference toxicant test was conducted with each batch of test larvae 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 SPP tests. *M. galloprovincialis* larvae were exposed to a filtered Sequim Bay seawater control plus copper sulfate concentrations of 1 $\mu\text{g/L}$, 4 $\mu\text{g/L}$, 16 $\mu\text{g/L}$, and 64 $\mu\text{g/L}$ as Cu, with three replicates per concentration. Because not all treatments could be tested concurrently, a separate reference test was performed with each round of tests to compare gamete sensitivity to a known toxicant.

A 48-h ammonia reference toxicant test was conducted in January 1994 to establish the sensitivity of test organisms to ammonia. *M. galloprovincialis* were exposed to a seawater control plus four nominal concentrations of ammonia (1, 10, 20, 80, and 100 mg/L as ammonium chloride) and was conducted in the same manner as the SPP tests. Actual ammonia concentrations were measured for this experiment and are reported in Section 3.0 of this report. There were three replicates of each concentration.

2.3.5 96-Hour Suspended-Particulate-Phase Static Test with *C. stigmaeus*

The test chambers for the January 1994 SPP test with *C. stigmaeus* were 10-gal static aquaria randomly positioned on the water tables, with 20 to 24 aquaria per table. Test temperature was maintained by a circulating water bath on the water table. Aeration was

provided through a glass pipet connected by silastic tubing to an overhead air manifold. Aquaria were labeled with a sediment treatment code, concentration, and replicate number. The volume of test material in each aquarium was 10 L. To obtain the 100% SPP treatment, 10 L of 100% SPP was added directly to the aquarium; the 0% SPP treatment consisted of 10 L of 0.45- μ m-filtered Sequim Bay seawater. To prepare the 10% and 50% SPP concentrations, appropriate volumes of 100% SPP and 0.45- μ m-filtered Sequim Bay dilution water were mixed directly in the test aquaria and filled to a volume of 10 L.

After SPP preparation and placement of containers on the water table, aeration was started and initial water quality parameters were measured in all replicates. Ten *C. stigmaeus* were then removed from the holding tanks, using a net, and added to each test container. The test population for each concentration was 50 individuals (200 individuals per SPP treatment). Initiation time and date were documented on test containers and data forms.

Observations of *C. stigmaeus* activity and behavior in each test container were made at test initiation and at 4, 24, 48, and 72 h. An organism was considered dead if it did not respond to gentle probing.

C. stigmaeus were not fed during the 96-h exposure. After initiation, DO, pH, salinity, and temperature were measured daily in at least one replicate. Ammonia was measured in at least one replicate of the 100% SPP prior to initiation of test organisms and at the end of the 96-h test. Acceptable ranges for the water quality parameters during the experiment were as follows:

DO	≥ 6.0 mg/L
pH	7.30-8.30
Salinity	$30\text{‰} \pm 2.0\text{‰}$
Temperature	$15.0^{\circ}\text{C} \pm 2.0^{\circ}\text{C}$
Ammonia	measured but presently not established.

Before termination of the test at 96 h, water quality parameters were measured in all replicates. At 96 h, the number of live and dead organisms was counted in each test container. As a quality control check, a second observer confirmed at least 10% of the mortality counts.

A 96-h Cu reference toxicant test was also conducted to establish the health and expected response of the test organisms. The reference toxicant test was conducted in the same manner as the SPP tests. *C. stigmaeus* were exposed to a seawater control plus four concentrations of copper sulfate (0.5, 1.0, 1.5, and 2.0 mg/L as Cu). There were three replicates of each concentration.

A 96-h ammonia reference toxicant test was also conducted to establish the expected response of the test organisms to ammonia. The ammonia reference toxicant test was conducted in the same manner as the SPP tests. *C. stigmaeus* were exposed to a seawater control plus four nominal concentrations of ammonia (15, 20, 25, and 30 mg/L as ammonium chloride). The

actual concentrations of ammonia were measured in this experiment and are reported in Section 3.0 of this report. There were three replicates of each concentration.

2.3.6 96-Hour Suspended-Particulate-Phase Static Test with *H. costata*

Prior to testing, *Holmesimysis costata* were held for at least 48-h in static, aerated aquaria maintained at test temperature ($15^{\circ}\text{C} \pm 2^{\circ}\text{C}$). In the holding tanks, *H. costata* were fed a dense solution of brine shrimp nauplii (*Artemia salina*) twice a day, and water quality parameters were monitored daily.

Test chambers for the *H. costata* were 500-mL glass jars placed in random positions on water tables. Test temperature was maintained by immersing these containers in a circulating water bath. Aeration was provided through a pipet connected by silastic tubing to an overhead air manifold. Appropriate volumes of 100% SPP and 0.45- μm -filtered Sequim Bay seawater were added to 1-gal jars to make 0%, 10%, 50%, and 100% SPP concentrations for the *H. costata* test. A total of 1500 mL was prepared for each dilution to provide the test volume of 300 mL in each of five replicates. All test chambers were labeled with position number, treatment code, concentration, and replicate number.

After the SPP concentrations were prepared and placed on the water table, gentle aeration was started and water quality parameters were measured in all test chambers. *H. costata* were then randomly removed from the holding tank using a wide-bore pipet. To initiate the test, 10 individuals were added to each container to produce a test population of 50 individuals per concentration. In the January 1993 study, three replicates were tested to produce a test population of 30 individuals per concentration.

Observations of test organisms were made at test initiation and at 4, 24, 48, and 72 h, using a light table to enhance visibility of the *H. costata*. During the 96-h exposure period, *H. costata* were fed brine shrimp daily. Excess food was removed with a small pipet before daily observations, using extra caution not to disturb test animals. Molted exoskeletons and any particulates that had settled from the SPP solution were also removed.

After test initiation, water quality parameters were measured daily in at least one replicate per SPP concentration. Ammonia was measured in at least one replicate of the 100% SPP prior to initiation of the organisms and at the end of the 96-h test. Acceptable ranges for the water quality parameters during the experiment were as follows:

DO	≥ 4.0 mg/L
pH	7.8 ± 0.5 units
Salinity	30 ± 2.0 ‰
Temperature	$15.0 \pm 2.0^{\circ}\text{C}$
Ammonia	measured but presently not established.

Prior to test termination, water quality parameters were measured in all replicates. At 96 h, the number of live and dead animals was counted in each test chamber. An organism was considered dead if it did not respond to gentle probing. As a quality control check, a second observer recounted surviving test organisms in at least 10% of the test containers.

A 96-h Cu reference toxicant test was conducted to establish the health and expected response of the test organisms. The reference toxicant test was conducted in the same manner as the SPP test. In the January 1993 study, *H. costata* were exposed to a seawater control along with four concentrations of copper (50, 100, 200, and 400 µg/L as Cu). In the January 1994 study, *H. costata* were exposed to a seawater control and four concentrations of copper (50, 100, 150, and 200 µg/L as Cu). There were three replicates of each treatment.

A 96-h ammonia reference toxicant test was conducted in January 1994 to establish the expected response of the test organisms to concentrations of ammonia. The ammonia reference toxicant test was conducted in the same manner as the SPP tests. *H. costata* were exposed to a seawater control plus 11 concentrations of ammonia (0.25, 0.5, 1, 2, 5, 10, 20, 40, 60, 80, and 100 mg/L as ammonium chloride). Actual ammonia measurements were determined for this experiment and are reported in Section 3.0 of this report. There were three replicates of each concentration.

2.3.7 10-Day Solid-Phase Flow-Through Preliminary and Definitive Feeding Studies with *N. caecoides*

These tests were performed only during the October 1994 study and follow the same methods of testing described in Section 2.3.2, with the exception that 2.5-gal aquaria were used instead of 10-gal and no *M. nasuta* were included in the test containers. The following paragraphs describe the method of food additions for both the preliminary and definitive studies.

2.3.7.1 Preliminary Test Method

The preliminary study was conducted to determine the following: 1) the most appropriate food source for *N. caecoides*; 2) the amount of food to be used; and 3) the best method for food distribution.

The three food sources, alfalfa, the algae *Enteromorpha* spp., and Tetramin, were chosen based on previous work conducted with the test species *Neanthes arenaceodentata* (Johns 1989). The alfalfa and Tetramin were obtained from a commercial supplier, and the *Enteromorpha* spp. was collected at low tide from the beach located at the MSL and then dried overnight. All three food sources were ground in a Waring blender and then sieved through a 0.5-mm sieve to achieve a fine powder.

The OBM sediment was prepared for testing by grating it into smaller particle sizes and mixing it in a large-capacity epoxy-coated mixer with approximately 10-L of filtered seawater.

The addition of the filtered seawater was necessary to achieve a "smooth" consistency that allowed an even distribution of sediment and food. A total of 12 gal of OBM sediment was prepared in this manner for the feeding study. An additional five gal of OBM sediment were grated and mixed in the epoxy-coated mixer without the addition of seawater. This was done to assess whether the softening of OBM sediments increases *N. caecoides* survival.

The quantities of food were selected based upon the regression depicted in Figure 2.2 that shows a high degree of correlation between TOC (ranging between 0.17% to 1.23%) and *N. caecoides* survival. The data points used in this regression were taken from studies previously conducted at the MSL. The sediment treatments for the feeding study were the following: OBM sediment, OBM sediment softened with seawater, and OBM sediment softened with seawater and amended with food to approximate a TOC range of 0.4%, 0.8%, and 1.2%.

To achieve the desired TOC percentages, the following three variables were determined: the percentage of dry weight of OBM sediment, the percentage of TOC of OBM sediment, and the wet weight of the volume of OBM sediment required for one test aquarium. For these experiments, it was assumed that the weight of each food source was entirely organic carbon. The following equations were used to determine how much food to dispense to each test aquarium:

- 950 mL of OBM sediment was required for each 2.5-gal aquarium
- 950 mL of wet OBM sediment weighs 1666 g
- $\text{weight of wet sediment} \times \text{percent dry weight} = \text{dry weight of sediment}$
 $1666 \text{ g wet sediment} \times 67\% \text{ dry weight} = 1116.2 \text{ g of dry sediment}$
- the amount of organic carbon already present in 1116.2 g of dry sediment =
 $1116.2 \text{ g} \times 0.18\% \text{ organic carbon} = 2.01 \text{ g of organic carbon}$
- the 0.4% organic carbon level desired for testing was 1116.2 g of OBM sediment * 0.4% organic carbon = 4.46 g of food. Since OBM already contains 2.01 g of organic carbon, an additional 2.45 g of food was added to reach 0.4% organic carbon.

The other percentages of TOC used in the preliminary and definitive studies were calculated using the same equations.

Two methods were used to deliver the food to each test aquarium to determine which was more suitable for the test species *N. caecoides*. The first feeding method required mixing the food sources directly into the sediment in a clean 0.5-gal container using a stainless steel spoon. A preweighed amount of OBM and food were added to the container and then placed on a roller table for 24 h. Once a homogenous consistency of OBM and food was achieved by using the rolling technique, the mixture was layered in a 2.5-gal aquarium. Each container on the roller table represented the amount of sediment and food necessary to supply one test chamber.

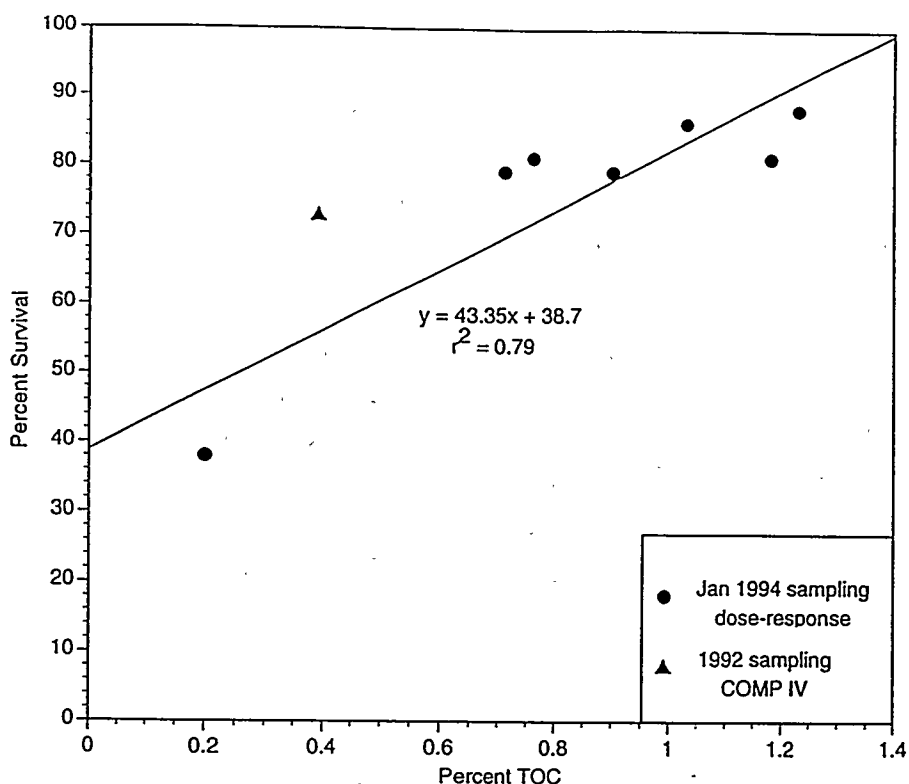


Figure 2.2 Linear Regression of Percentage TOC vs. Percentage Survival of *N. caecoides*

The second feeding method required the food to be sprinkled into each test container. Before the food was sprinkled into the 2.5-gal aquarium, the flow-through system was turned off, the food was added to the water and allowed to settle on top of the sediment. After the food had settled, the flow-through system was turned on. The amount of food (0.8% TOC) to be sprinkled in each aquarium was separated into three portions. One portion was sprinkled in the aquarium at the beginning of the test, and if the food disappeared from the sediment, the second aliquot was added. This was done to minimize the impact of uneaten food decaying on the sediment surface. Only one aliquot of food was added to each test aquarium, because it appeared as though the food were virtually uneaten by the test organisms. By the end of the test, the food was decaying, and a visible scum was present on the uneaten food.

2.3.7.2 Definitive Study Methods

The results of the preliminary study were evaluated and used to determine the test design for the definitive study. The *Enteromorpha* spp. was chosen for the definitive study using the roller method. This food source was chosen because it is natural and it represents a potential food source most likely encountered by the test species. The procedures for preparing *Enteromorpha* spp., the OBM sediment, and the mixing method are the same as previously described for the preliminary study.

The experimental design included OBM sediment, OBM sediment softened with filtered seawater, and softened OBM mixed with food, to represent TOC concentrations (including TOC already present in the OBM sediment) of 0.4%, 0.6%, 1.0%, and 1.4%. These were estimated TOC levels based on the amount of *Enteromorpha* spp. added to the sediment. Aliquots of the OBM sediment mixed with *Enteromorpha* spp. were analyzed for TOC by Analytical Resources, Inc. (ARI). The actual TOC levels present in the OBM sediment were approximately half of the estimated percentages.

2.4 SEDIMENT AND TISSUE CHEMISTRY

The OBM COMP sediment from Richmond Harbor and all control sediments collected in January 1993 were analyzed for conventional and other chemical parameters. Conventional parameters included grain size, total solids, TOC, total volatile solids (TVS), and oil and grease. Sediments were analyzed for PAHs, PCB/pesticides, metals, and butyltins. Samples representing *M. nasuta* tissue exposed to the OBM COMP sediment treatment were also analyzed for chlorinated pesticides, PCBs, PAHs, metals, and butyltins.

In January 1994, the OBM COMP, the reference sediments, and the *M. nasuta* tissues exposed to the OBM COMP and reference sediments, were analyzed for PCBs, pesticides, and butyltins. The OBM COMP collected and tested in October 1994 was analyzed for the same conventional parameters and organic and inorganic analytes as the January 1993 study, with the addition of total petroleum hydrocarbon (TPH) analysis. Table 2.1 presents the analytical parameters, the method used to perform each analysis, and the target analytical detection limits.

The following sections briefly describe the methods used for analysis of sediments and tissues, which followed established EPA procedures, where applicable. Quality control samples included method blanks, matrix spikes (MS), standard reference materials (SRM), and analytical replicates. The MS and SRMs were used as a measurement of analytical accuracy. The analytical replicates were used to evaluate analytical precision.

2.4.1 Conventional Sediment Measurements

Grain size analysis and total solids for all three studies were conducted by Soil Technology, Bainbridge Island, Washington. In January 1993, four grain size fractions (expressed in microns) were quantified as: gravel ($>2000\ \mu\text{m}$), sand ($62.5 - 2000\ \mu\text{m}$), silt ($3.9 - 62.5\ \mu\text{m}$), and clay ($<3.9\ \mu\text{m}$). In the 1994 studies, 16 grain size fractions were determined by a combination of sieve and pipet techniques from the Puget Sound Estuary Program (PSEP) *Protocols for Measuring Selected Environmental Variables in Puget Sound* (PSEP 1986). These methods are consistent with ASTM Method D2217 (ASTM 1985) D422 (ASTM 1972) (with the substitution of a No. 100 sieve for a No. 140 sieve). These data are reported as "apparent" grain

size, since organic material is included in the analysis. An additional measurement for salt content was performed, and each grain size fraction was corrected for this salt measurement.

Approximately 25 g of each sediment sample was analyzed for total solids, and another 10-g to 100-g aliquot was weighed for grain size analysis. To separate the coarser sand and gravel fraction from the silt/clay fraction, sediment was washed with distilled water through a 63.5- μm (4.0 phi) sieve into a 1-L graduated cylinder. The coarse fraction was dried, weighed, and shaken through a nest of sieves to yield the required seven coarse subfractions. Any material still passing through the final 63.5- μm sieve was added to the previous fines in the 1-L graduated cylinder. The silt/clay fraction was then subdivided by a pipet technique based on Stoke's Law of differential settling velocities for different sized particles. The silt/clay fraction was disassociated by adding a dispersant (sodium hexametaphosphate) to distilled water contained in a 1-L graduated cylinder. At specified time intervals and specified depths below the surface, 20-mL aliquots were withdrawn from the graduated cylinder, delivered to a preweighed container, and dried at $90^{\circ}\text{C} \pm 2^{\circ}\text{C}$ to a constant weight. Replicate analyses of a sample from each batch of samples were performed as a quality control measure. Other quality control measures, such as spikes, SRMs, or minimum detection limits, do not apply to grain size analysis.

Analysis of TOC consisted of measuring the amount of nonvolatile, partially volatile, volatile, and particulate organic carbon compounds in a sample. The analyses of TOC for the January 1993 and January 1994 studies were performed by Global Geochemistry in Canoga Park, California, following modified Method SW846 EPA-9060 (EPA 1986). Each sediment treatment was dried and ball-milled to a fine powder. Before combustion, inorganic carbon in the sample was removed by acidification. The TOC in sediment was then determined by measuring the quantity of carbon dioxide released during combustion of the sample and reporting the release as percentage of dry weight. Quality control measures included method blanks and replicate analyses for each batch of samples. Analysis of TOC for the October 1994 study was performed by ARI in Seattle, Washington. Briefly, ARI used the method described in Plumb (1981), which involves direct combustion at 850°C in a resistance furnace. Combustion products are carried in the oxygen stream through a catalytic converter (to assure complete oxidation to CO_2) and into the infrared spectrophotometer (IR). Prior to analysis, samples are purged of inorganic carbon by acidification, dried at 70°C , and then ground to pass through a 120- μm mesh sieve. The methods used by ARI and Global Geochemistry are comparable in performing TOC analysis.

Analyses of TVS measures the fraction of total solids that are lost on ignition at a higher temperature than that used to determine total solids. Analysis of TVS was performed by the MSL using EPA 160.4 (EPA 1979) in the January 1993 study and performed at ARI using PSEP (1986) in the January 1994 and October 1994 studies. The method used by the two

laboratories to perform TVS analysis are similar. The sample is first oven-dried to constant weight, removed and combusted at 550°C, cooled in a desiccator, and then reweighed. The amount of sample lost during ignition was then defined as the *volatile solids fraction*. The TVS measurements are used as an estimate for the amount of organic matter in the total solids. Operationally, TVS is defined by the combustion temperature and does not always represent the organic content of a sample, because some of the more volatile organic material can be lost during drying, and some inorganic material can be lost during combustion. Quality control measures include method blanks and replicate analyses for each batch of samples.

Total oil and grease includes vegetable oils, animal fats, soaps, waxes, and any other carbon-hydrogen material extractable by the solvent Freon. The TPH comprises the nonpolar mineral fraction of total oil and grease that is not removed by silica gel absorption. The oil and grease and TPH analyses were performed by ARI for all three studies. The IR was used to determine concentrations of oil and grease (Method 413.2, EPA 1979) and petroleum hydrocarbons (Method 418.1, EPA 1979). A 20-g aliquot of sample was dried with anhydrous sodium sulfate, then extracted with Freon. For total oil and grease, sample extracts were scanned from 4000 to 600 cm⁻¹ on an IR, and the peak height measured at 2930 cm⁻¹. Since this wavelength represents the -CH₂ configurations of hydrocarbons, it was the standard used to determine oil and grease. For TPH, silica gel was added to the extract to remove the more polar animal- and vegetable-based oils. The extract was then shaken and allowed to settle. An aliquot was then removed and scanned the same way as was the oil and grease sample. The relationship of peak height to oil concentration was determined by regressing the peak height versus a known concentration of fuel oil and vegetable oil for TPH and oil and grease, respectively. Quality control measures include method blanks, matrix spikes, and replicate analyses for each batch of samples.

2.4.2 Polynuclear Aromatic Hydrocarbons

Analyses for 16 PAHs (Table 2.1) in the OBM COMP sediment were performed by ARI in January 1993 and by the MSL in January 1994 and October 1994 studies. The analyses of tissue samples were performed by the MSL for the January 1993 study and 1994 studies. The semivolatile organic compounds analyzed in sediments and tissues were the 16 PAHs listed in EPA Method 610. These compounds were extracted with methylene chloride in accordance with the National Oceanic and Atmospheric Administration (NOAA) Status and Trends method (Krahn et al. 1988) or SW-846 organic extraction method (EPA 1986). The extracts were dried over sodium sulfate, passed through a cleanup column, and concentrated in preparation for further cleanup by liquid chromatography. Surrogate compounds were added to all samples prior to extraction to assess extraction efficiency. The PAHs in sample extracts were analyzed via high resolution capillary gas chromatography/mass spectroscopy (GC/MS) in the selective ion mode

TABLE 2.1 List of Analytes, Methods, and Target Detection Limits(a)

<u>Analyte</u>	<u>Reference Method</u>	<u>Detection Limit</u>
Grain Size	ASTM D2217 & D422	1.0%
Total Solids	160.3 (EPA 1979)	1.0%
Total Organic Carbon	9060 (EPA 1986)	0.1%
Oil and Grease	413.2 (EPA 1979)	20 mg/kg
Total Volatile Solids	160.4 (EPA 1979)	0.1%
Metals (Sediment)		
Arsenic	PNL SOP for XRF Method	2.5 mg/kg
Cadmium	200.9 (EPA 1991)	0.1 mg/kg
Chromium	PNL SOP for XRF Method	33 mg/kg
Copper	PNL SOP for XRF Method	5.5 mg/kg
Lead	PNL SOP for XRF Method	6.2 mg/kg
Mercury	Bloom and Crecelius 1983	0.01 mg/kg
Nickel	PNL SOP for XRF Method	7.5 mg/kg
Selenium	200.9 (EPA 1991)	0.2 mg/kg
Silver	200.9 (EPA 1991)	0.02 mg/kg
Zinc	PNL SOP for XRF Method	7.8 mg/kg
Metals (Tissues)		
Arsenic	PNL SOP for XRF Method	2.0 mg/kg
Cadmium	200.8 (EPA 1991)	0.1 mg/kg
Chromium	200.8 (EPA 1991)	1.0 mg/kg
Copper	PNL SOP for XRF Method	4.3 mg/kg
Lead	200.8 (EPA 1991)	1.0 mg/kg
Mercury	Bloom and Crecelius 1983	0.01 mg/kg
Nickel	200.8 (EPA 1991)	1.0 mg/kg
Selenium	PNL SOP for XRF Method	1.0 mg/kg
Silver	200.8 (EPA 1991)	0.1 mg/kg
Zinc	PNL SOP for XRF Method	35 mg/kg
Pesticides		
Aldrin	8080 (EPA 1986)	2.0 µg/kg
α-BHC	8080 (EPA 1986)	2.0 µg/kg
β-BHC	8080 (EPA 1986)	2.0 µg/kg
γ-BHC (Lindane)	8080 (EPA 1986)	2.0 µg/kg
δ-BHC	8080 (EPA 1986)	2.0 µg/kg
Chlordane	8080 (EPA 1986)	30 µg/kg
4,4'-DDD	8080 (EPA 1986)	2.0 µg/kg
4,4'-DDE	8080 (EPA 1986)	2.0 µg/kg
4,4'-DDT	8080 (EPA 1986)	2.0 µg/kg
Dieldrin	8080 (EPA 1986)	2.0 µg/kg
Endosulfan I	8080 (EPA 1986)	2.0 µg/kg
Endosulfan II	8080 (EPA 1986)	2.0 µg/kg
Endosulfan sulfate	8080 (EPA 1986)	2.0 µg/kg
Endrin	8080 (EPA 1986)	2.0 µg/kg
Endrin aldehyde	8080 (EPA 1986)	2.0 µg/kg
Heptachlor	8080 (EPA 1986)	2.0 µg/kg
Heptachlor epoxide	8080 (EPA 1986)	2.0 µg/kg
Toxaphene	8080 (EPA 1986)	30 µg/kg

TABLE 2.1 (Contd)

Analyte	Reference Method	Detection Limit
PCBs		
Aroclor, 1242	8080 (EPA 1986)	20 µg/kg
Aroclor, 1248	8080 (EPA 1986)	20 µg/kg
Aroclor, 1254	8080 (EPA 1986)	20 µg/kg
Aroclor, 1260	8080 (EPA 1986)	20 µg/kg
PAHs		
Acenaphthene	8270 (EPA 1986)	20 µg/kg
Acenaphthylene	8270 (EPA 1986)	20 µg/kg
Anthracene	8270 (EPA 1986)	20 µg/kg
Benzo(a)anthracene	8270 (EPA 1986)	20 µg/kg
Benzo(a)pyrene	8270 (EPA 1986)	20 µg/kg
Benzo(b)fluoranthene	8270 (EPA 1986)	20 µg/kg
Benzo(g,h,i)perylene	8270 (EPA 1986)	20 µg/kg
Benzo(k)fluoranthene	8270 (EPA 1986)	20 µg/kg
Chrysene	8270 (EPA 1986)	20 µg/kg
Dibenzo(a,h)anthracene	8270 (EPA 1986)	20 µg/kg
Fluoranthene	8270 (EPA 1986)	20 µg/kg
Fluorene	8270 (EPA 1986)	20 µg/kg
Indeno(1,2,3-cd)pyrene	8270 (EPA 1986)	20 µg/kg
Naphthalene	8270 (EPA 1986)	20 µg/kg
Phenanthrene	8270 (EPA 1986)	20 µg/kg
Pyrene	8270 (EPA 1986)	20 µg/kg
Butyltins		
Monobutyltin	Unger et al. 1986	10 µg/kg
Dibutyltin	Unger et al. 1986	10 µg/kg
Tributyltin	Unger et al. 1986	10 µg/kg

(a) Detection limits are in dry weight for all sediment parameters and for metals in tissues. Detection limits are in wet weight for all organic tissue parameters.

(SIM) following modified EPA SW-846 Method 8270 (EPA 1986). In the SIM mode, each PAH compound was monitored simultaneously for the presence of a parent ion and a confirming second ion. Tissue extracts were run through gel permeation chromatography (GPC) prior to analysis to remove any additional interferences. Quality control measures include method blanks, matrix spikes, SRMs, and replicates for each batch of samples. Concentrations for both sediments and tissues are reported in µg/kg.

2.4.3 Chlorinated Pesticides and Polychlorinated Biphenyls

The PCB and pesticide analyses for the OBM COMP sediment was performed by ARI in January 1993 and by the MSL in January 1994 and October 1994. The tissue analyses were performed by the MSL in the January 1993 study and the 1994 studies. Chlorinated pesticides

and PCBs in sediments and tissues were quantified by gas chromatography/electron capture detection (GC/ECD) following modified EPA SW-846 Method 8080 (EPA 1986).

Chlorinated pesticides and PCBs were extracted simultaneously with the PAH compounds. The procedure involved methylene chloride extraction using sonication or roller technique for sediments and the roller technique for tissues. The extract was then concentrated and cleaned up using a column packed with deactivated alumina and silica by the NOAA Status and Trends Method (Krahn et al. 1988). The extracts then were subjected to an additional cleanup treatment using high performance liquid chromatography (HPLC) to remove other interferences. Analytical quantification was performed using GC/ECD analysis. Surrogates were added to each sample before extraction to assess extraction efficiency. Quality control measures include method blanks, matrix spikes, SRMs, and replicates for each batch of samples. Concentrations for both sediments and tissues are reported in $\mu\text{g/kg}$.

2.4.5 Metals

The metals analyses for all three studies were performed by Battelle Pacific Northwest National Laboratory (PNNL) in Richland, Washington, and the MSL. Ten metals were analyzed in sediment and tissue samples: arsenic (As), Cd, chromium (Cr), Cu, lead (Pb), mercury (Hg), nickel (Ni), selenium (Se), silver (Ag) and zinc (Zn). The four techniques used for the analysis of metals were as follows: 1) energy-dispersive x-ray fluorescence (XRF), following a PNL standard operating procedure; 2) Zeeman graphite-furnace atomic absorption spectroscopy (GFAA), following modified EPA 200.9 (EPA 1991); 3) cold-vapor atomic absorption spectroscopy (CVAA), according to the method of Bloom and Crecelius (1983); and 4) inductively coupled plasma mass spectroscopy (ICP/MS), following modified EPA 200.8 (EPA 1991).

To prepare sediment and tissues for analysis, samples were freeze-dried, then blended in a Spex mixer-mill. Approximately 5 g of this mixed sample was then ground in a ceramic ball mill. The XRF analysis was performed on a 0.5-g aliquot of dried, ground material pressed into a pellet with a diameter of 2 cm. For GFAA, CVAA, and ICP/MS analyses, 0.2-g aliquots of dried homogenous sample went through an acid digestion process to separate and isolate the metals from the matrix. Concentrations for both sediments and tissues are reported in mg/kg .

2.4.6 Organotins

Organotins in sediment and tissue samples for all three studies were analyzed at the MSL in Sequim, Washington. Butyltin compounds in sediment and tissue were analyzed using gas chromatography/flame photometric detection (GC/FPD) following the methods of Unger et al. (1986). Wet samples were extracted with methylene chloride and tropolone. Triphenyltin was added before extraction as a surrogate compound to assess extraction efficiency. The mono-, di-, and tributyltin compounds extracted from the sediment, and tissues were derivatized to a less

volatile, more thermally stable form (nonionic n-hexyl/or n-pentyl derivatives). The extracts were passed through a Florisil liquid chromatography column for cleanup, and the butyltins were quantified by GC/FPD. Concentrations for both sediments and tissues are reported in $\mu\text{g/kg}$.

2.5 DATA ANALYSIS AND INTERPRETATION

Several statistical analyses were conducted to determine the magnitude and significance of toxicity or the magnitude of bioaccumulation of contaminants in OBM sediments. Each statistical test was based on a completely randomized design that controlled bias between treatments.

2.5.1 Randomization

All solid-phase and SPP toxicity test organisms were designed as completely random tests, and treatments were randomly positioned on water tables. To determine the randomization, a random number table was generated for each toxicity test using the discrete uniform random number generator in the Excel spreadsheet software. For the SPP tests, *H. costata* and *C. stigmaeus* individuals and *M. galloprovincialis* larvae were randomly allocated to SPP replicates for all concentrations.

2.5.2 Statistical Analysis of Solid-Phase Tests

Solid-phase toxicity of all sediment treatments was evaluated by analysis of variance (ANOVA) tests on the arcsine square root of the proportion of organisms surviving the test. The arcsine square-root transformation stabilizes the within-class variances to meet the assumptions of the ANOVA. All treatments were compared using the conservative Dunn's Test for comparison of all means (Dunn 1961), using an experiment-wise error rate of $\alpha = 0.05$. The Dunn's Test is a multiple-range comparison test that provides information about how each sediment treatment compares with all other treatments. For the purposes of this report, the OBM results obtained for the 1993 study were compared with the OBM and reference results obtained during the 1994 studies. These comparisons assessed the reproducibility of the results among the different studies, the uniformity of the OBM sediments throughout the Richmond Harbor area, and the determination of whether OBM sediments are considered acutely toxic as defined in the *1991 Implementation Manual*. A test treatment was considered acutely toxic if the difference was statistically significant from any of the reference sediments and if the survival of the test organisms exposed to the test treatment was $\geq 10\%$ lower than the survival in the reference sediment ($\geq 20\%$ lower than reference for *R. abronius*).

2.5.3 Statistical Analysis of Suspended-Particulate-Phase Tests

Two statistical tests are presented in the *1991 Implementation Manual* for the interpretation of SPP tests. The first test is a one-sided t-test between survival in control

replicates and survival in the 100% SPP replicates. This test is performed only when survival in the 100% SPP is less than control (0% SPP) survival and when control survival is greater than 90% for nonlarval test and 70% for larval tests (indicating test validity). Prior to conducting the t-test, angular transformation (arcsine of the square root) of the proportion surviving in test replicates is performed to reduce possible heterogeneity of variance between control and 100% SPP mean survivals. The second test required by the *1991 Implementation Manual* is an LC_{50} or EC_{50} calculation, the concentration of SPP that is lethal or produces an effect to 50% of the individuals tested. The LC_{50} or EC_{50} values for these tests were calculated using the Trimmed Spearman-Kärber Method (Finney 1971). The Spearman-Kärber estimator is appropriate only if there is increasing mortality with increasing concentration and if 50% or greater mortality is observed in test solutions when normalized to control survival. If 50% mortality does not occur in the 100% SPP dilutions for any treatments, then LC_{50} values are reported as >100% SPP. The same method was used to calculate EC_{50} values.

2.5.4 Statistical Analysis of Bioaccumulation

Before statistical analysis of *M. nasuta* tissue concentrations, tissue chemistry data were reviewed. If the review showed that a compound was undetected in the replicate samples in test treatments, or that the mean tissue concentration in the reference treatments was greater than that in the test treatments, no further analysis was performed. If a compound was detected in at least one replicate of a test treatment and it exceeded that of any reference treatment replicate, statistical analysis was performed. In all cases, dry weight detection limits were used in numerical calculations when a compound was undetected.

Contaminant levels in tissues of *M. nasuta* exposed to potential dredged material test treatments were compared with those exposed to sediment from each of the three reference areas. Bioaccumulation data were statistically analyzed using Dunn's Test ($\alpha = 0.05$). Any statistically significant bioaccumulation was reported along with the magnitude of the difference between mean contaminant concentrations found in the test treatment tissues and the mean concentration found in the tissues exposed to each of the reference sediments.

2.6 QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES

The quality assurance/quality control (QA/QC) procedures followed for these studies were consistent with the *1991 Implementation Manual* and the EPA protocols (EPA 1986). The procedures were documented by the PNNL Quality Engineering Division in a QA Plan (QAP). Data accumulation notebooks were assigned to each portion of the study and served as records of day-to-day activities during the research. All entries in the notebooks were signed, dated, and reviewed by both the project manager and the quality assurance engineer. The following

discussion summarizes QA/QC procedures followed for the three main portions of these studies: sediment sampling, biological testing, and chemical testing.

2.6.1 Sample Tracking and Storage

All sediment samples were accompanied by chain-of-custody forms from the time of collection to receipt at the MSL. After sample selection and compositing, a new set of custody forms was initiated for the sediment subsamples requiring chemical analyses. These accompanied the samples to the appropriate laboratory where the forms were signed and returned to the MSL project manager. Custody forms were also initiated for all tissue samples upon completion of the biological testing. These forms accompanied the samples to the appropriate laboratory for chemical analyses.

All sediment collected for these studies was stored in glass, cellulose acetate butyrate containers, or steel drums lined with 9-C-4-A-phenolic epoxy, a noncontaminating coating. Sediment core and grab samples were stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ prior to biological testing. Subsamples for chemical analyses were obtained prior to biological testing. These subsamples were stored frozen (except for grain size, which was held at 4°C) until chemical analyses were performed. Samples for organic analyses were stored frozen up to 2 months (PSEP 1986). Samples for metals were freeze-dried upon receipt at the laboratory and held up to 6 months (PSEP 1986; EPA/USACE 1991).

Tissue samples were frozen immediately upon completion of the bioaccumulation tests. Samples for organic analyses and metals were stored in precleaned glass jars with Teflon-lined lids and samples for metals analyses were stored in precleaned plastic jars. Prior to analyses, samples for organic analyses were held for up to 2 months.

2.6.2 Quality Control Procedures for Sediment and Tissue Chemistry

Chemical testing procedures require that specific QA/QC protocols be followed. The QA/QC guidelines specific to this project were provided in a PNNL Quality Assurance Division QAP. These guidelines include the following:

- analysis of a method blank with each batch of samples
- replicate analysis on at least 5% of the samples (triplicate analyses where possible) to assess analytical precision
- analysis of matrix spikes on 5% of the samples (where applicable) with appropriate compounds to assess accuracy
- analysis of SRMs at a frequency of 5%, if available for the analytes of interest and sample matrix
- archiving of all instrument printouts (e.g., raw data and chromatograms from GC analyses) for future review.

3.0 RESULTS

3.1 FIELD INFORMATION

The OBM sediment samples were collected for three separate studies in January 1993, January 1994, and October 1994, using a vibratory-hammer coring device deployed from the derrick barge supplied by Manson Construction in Richmond Harbor, California (Figure 1.1). Each of the OBM COMPs for the three studies were collected from selected sites in Richmond Harbor as shown in Table 3.1. Control and reference sediments were collected by MSL personnel or obtained from a commercial supplier for use in the biological toxicity tests. The reference sediment from Bay Farm Borrow Area, Alcatraz Environs Reference Area, and Deep Off-Shelf Reference Area were collected for the two studies in January 1994 and October 1994. Control sediment from Sequim Bay, West Beach, and Tomales Bay were also collected for each of the three studies. All sediment samples were stored in noncontaminating containers, epoxy-lined 5-gal buckets or seasoned coolers at approximately 4°C until ready for processing at the MSL. OBM sediment and reference sediment samples were shipped via refrigerated van to the MSL upon completion of the sampling effort.

3.1.1 Reference Sediment Sampling

The Deep Off-Shelf Reference Area is located approximately 50 nautical miles southwest of the Golden Gate Bridge (Figure 3.1), off the continental shelf, in about 1500 m of water. Sediment samples from this area were collected using a pipe dredge deployed from the *FV Cobra* as described in Section 2.1.2. The R-BF and R-AM sediments were also collected using the *FV Cobra*. The Bay Farm Borrow Area, located in San Francisco Bay (Figure 3.2), was sampled at four locations and the sediments were combined to form one R-BF composite, representative of this area. The Alcatraz Environs Reference Area was sampled at eight locations (Figure 3.3) during the January 1994 field cruise and seven locations during the October 1994 field cruise. Station R-AM-F was omitted from the October cruise after the results from the individual stations surrounding R-AM showed that R-AM-F had much higher levels of low-molecular-weight PAHs (LPAHs) and high-molecular-weight PAHs (HPAHs) relative to the other stations. For both of the field cruises, the individual R-AM stations were combined to form one R-AM composite that was used for all chemical and biological evaluations.

3.1.2 Control Sediment Sampling

Control sediments for use in solid-phase toxicity tests were collected from Sequim Bay (Figure 3.4), West Beach (Figure 3.5), and Tomales Bay (Figure 3.6). Sequim Bay sediment (C-SB) was used in all solid-phase tests as an experimental control as well as for the native control for the clam, *M. nasuta*. West Beach control sediment (C-WB) was used as the native

TABLE 3.1. Sediment Contribution of each Sampling Site for Chemical and Biological Testing, Older Bay Mud Studies

Sampling Sites	Depth of OBM used for OBM COMP (-ft MLLW)
<u>January 1993 Study</u>	
RC-1	42.8 to 45.3
RC-2	43.8 to 46.7
RC-3	35.5 to 38.4
RC-4	43.4 to 46.2
RC-5	44.6 to 46.4
RC-6	33.5 to 37.5
<u>January 1994 Study</u>	
RC-7	40 to 42
RC-8	40 to 42
RC-9	40 to 42
RC-10	40 to 42
RC-11	40 to 42
RC-12	43 to 46
RC-14	40 to 42
RC-15	41.5 to 45
RC-16	40 to 42
RC-17	42.4 to 45.4
RC-18	40 to 42
RC-19	40 to 42
RC-20	40 to 42
RC-21	40 to 42
RC-22	40 to 42
RC-23	41 to 43
RC-24	40 to 42
RC-25	40 to 42
RC-26	39.4 to 42
<u>October 1994 Study</u>	
RC-13	37.1 to 42
RC-18	39 to 42
RC-21	40.6 to 42
RC-26	39.4 to 42

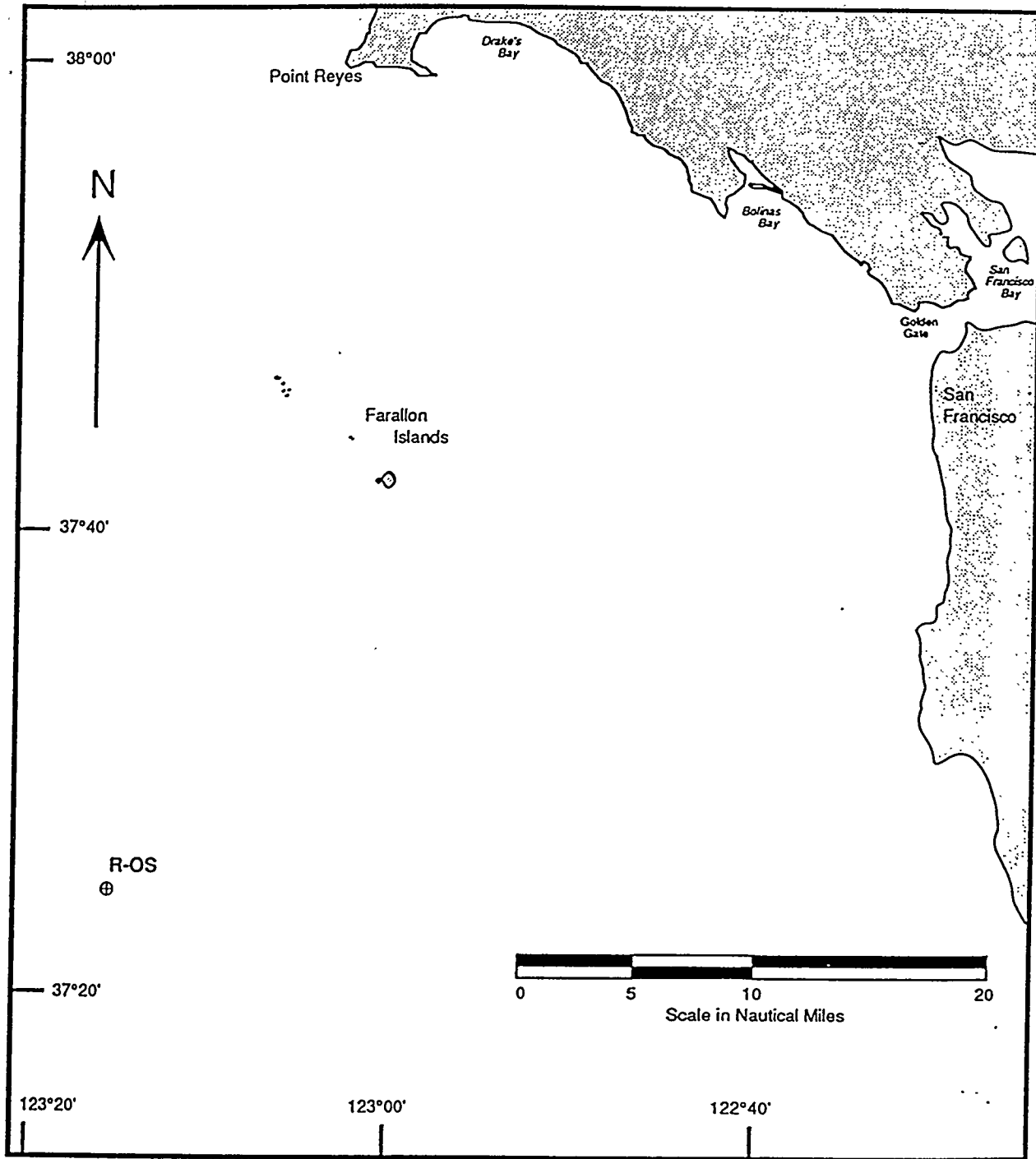


FIGURE 3.1. Location of Deep Off-Shelf Reference Area (R-OS)

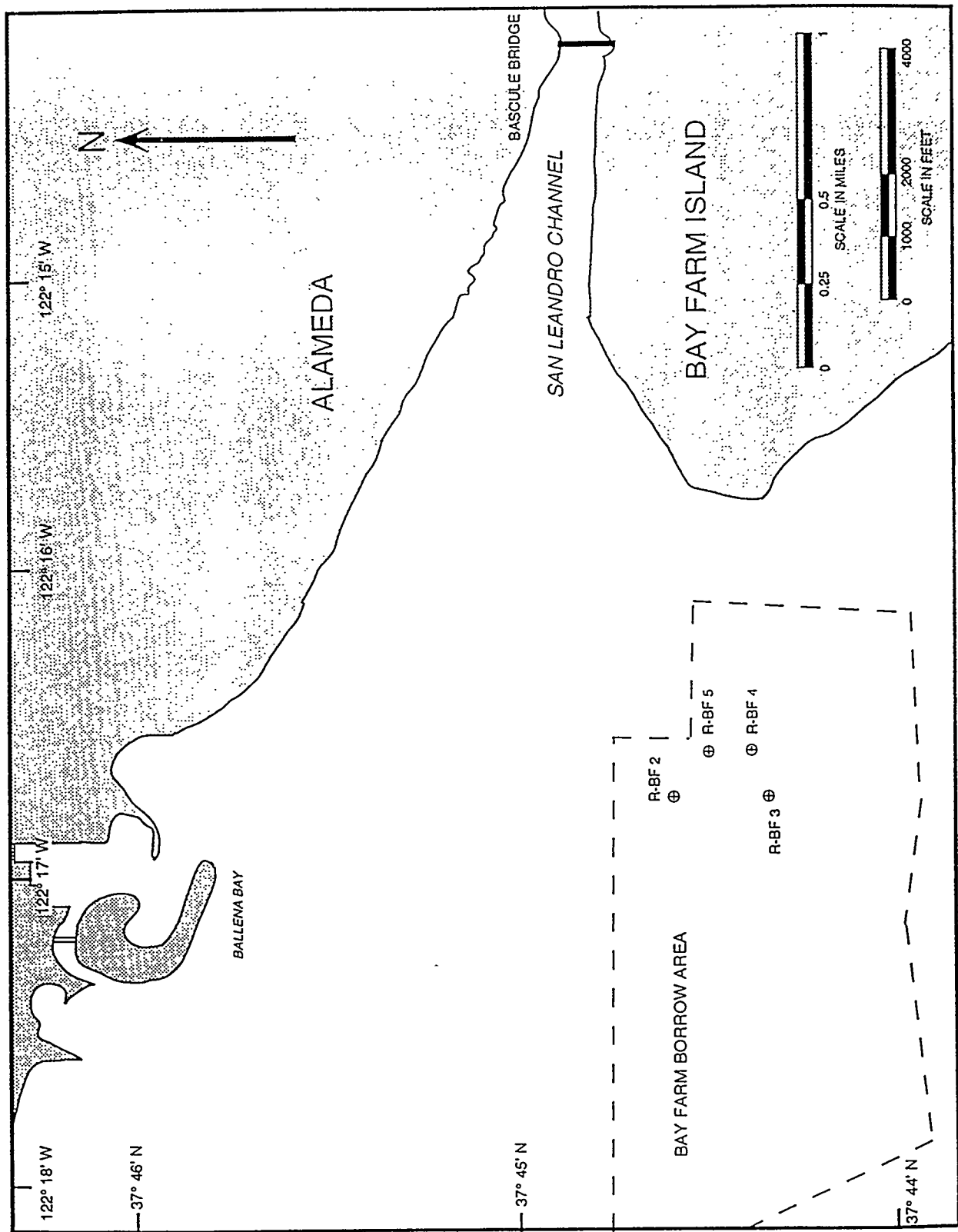


FIGURE 3.2. Location of Bay Farm Borrow Area (R-BF)

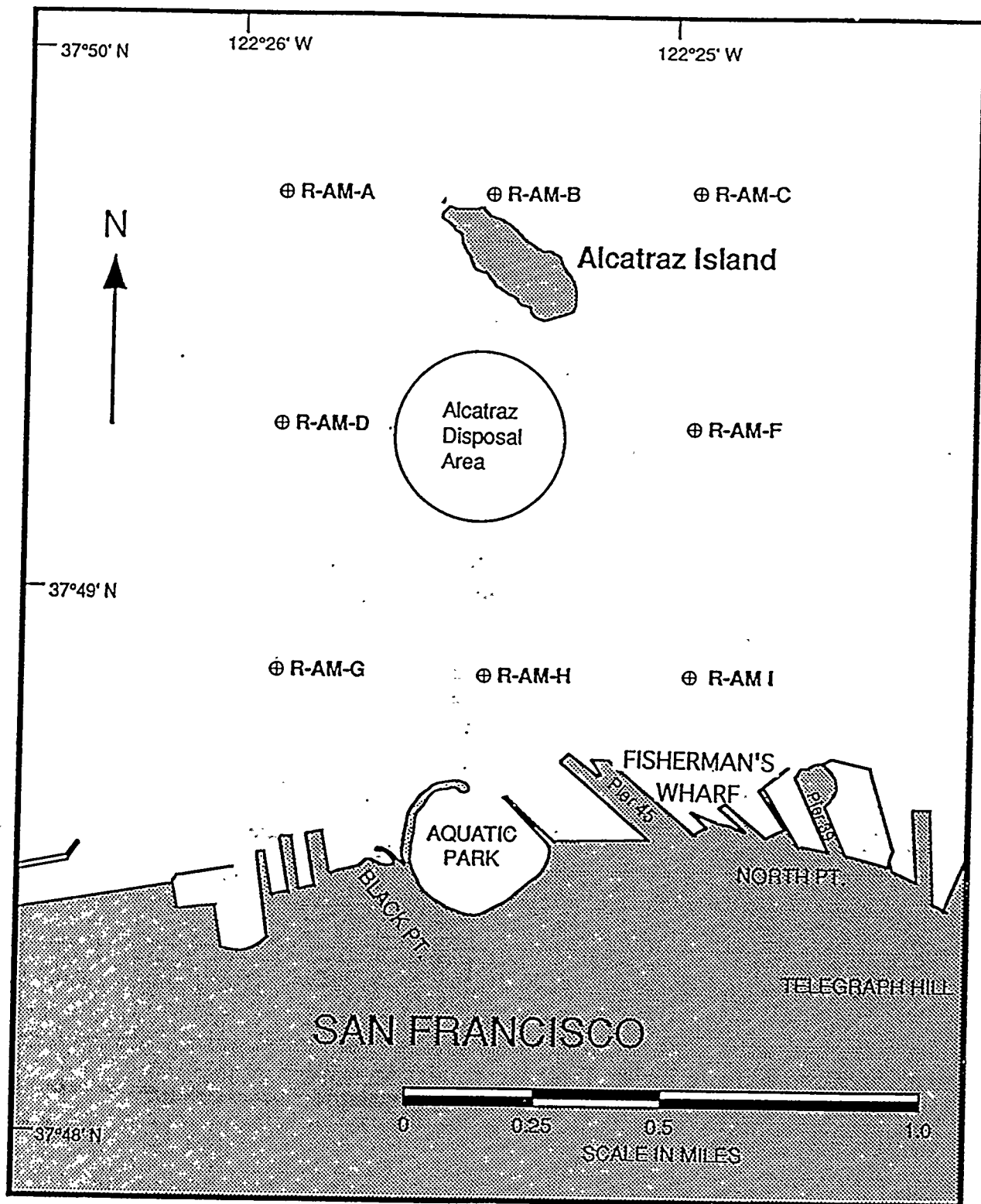


FIGURE 3.3. Location of Alcatraz Environs Reference Area (R-AM)

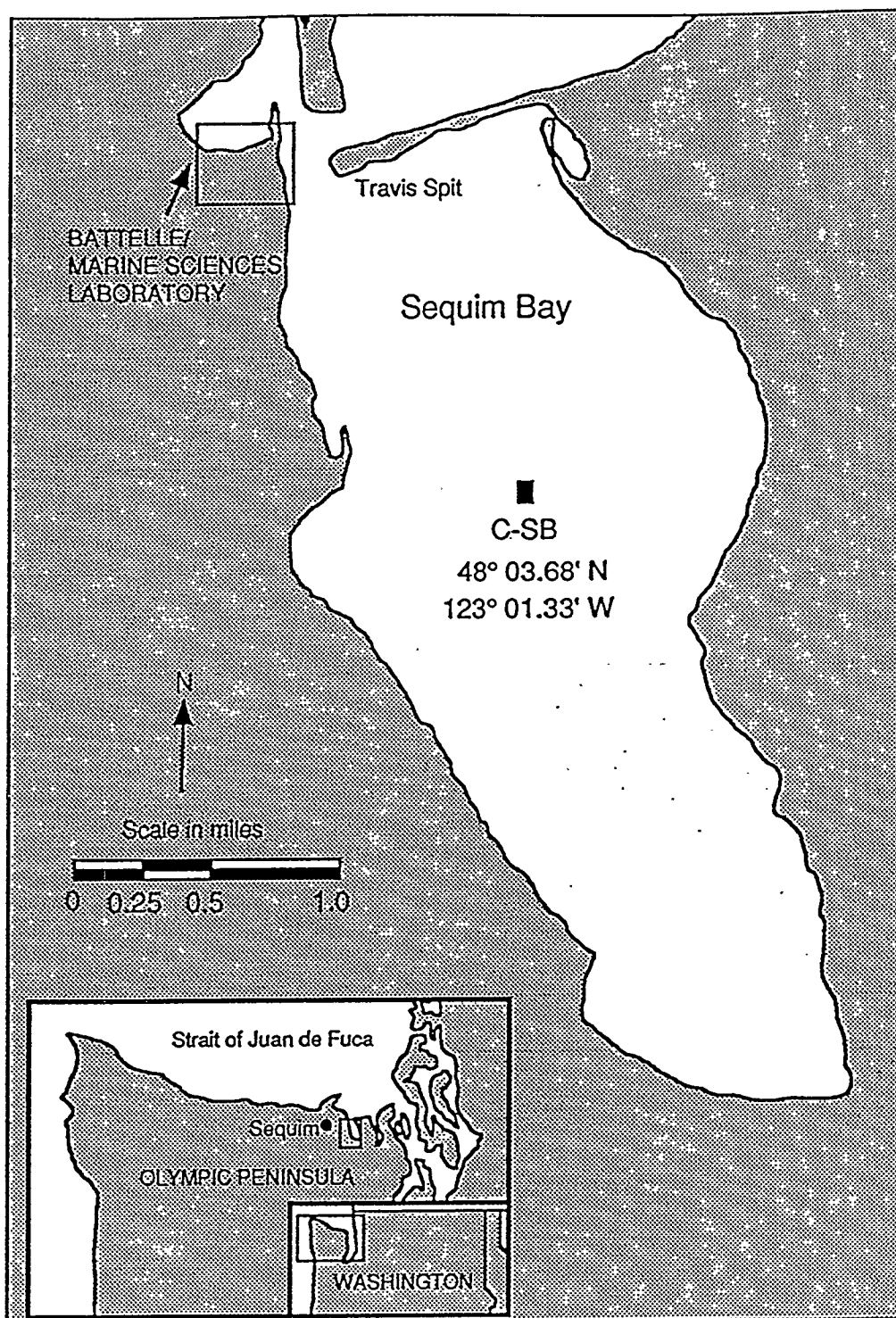


FIGURE 3.4. Location of *M. nasuta* Control (C-SB), Sequim Bay, Washington

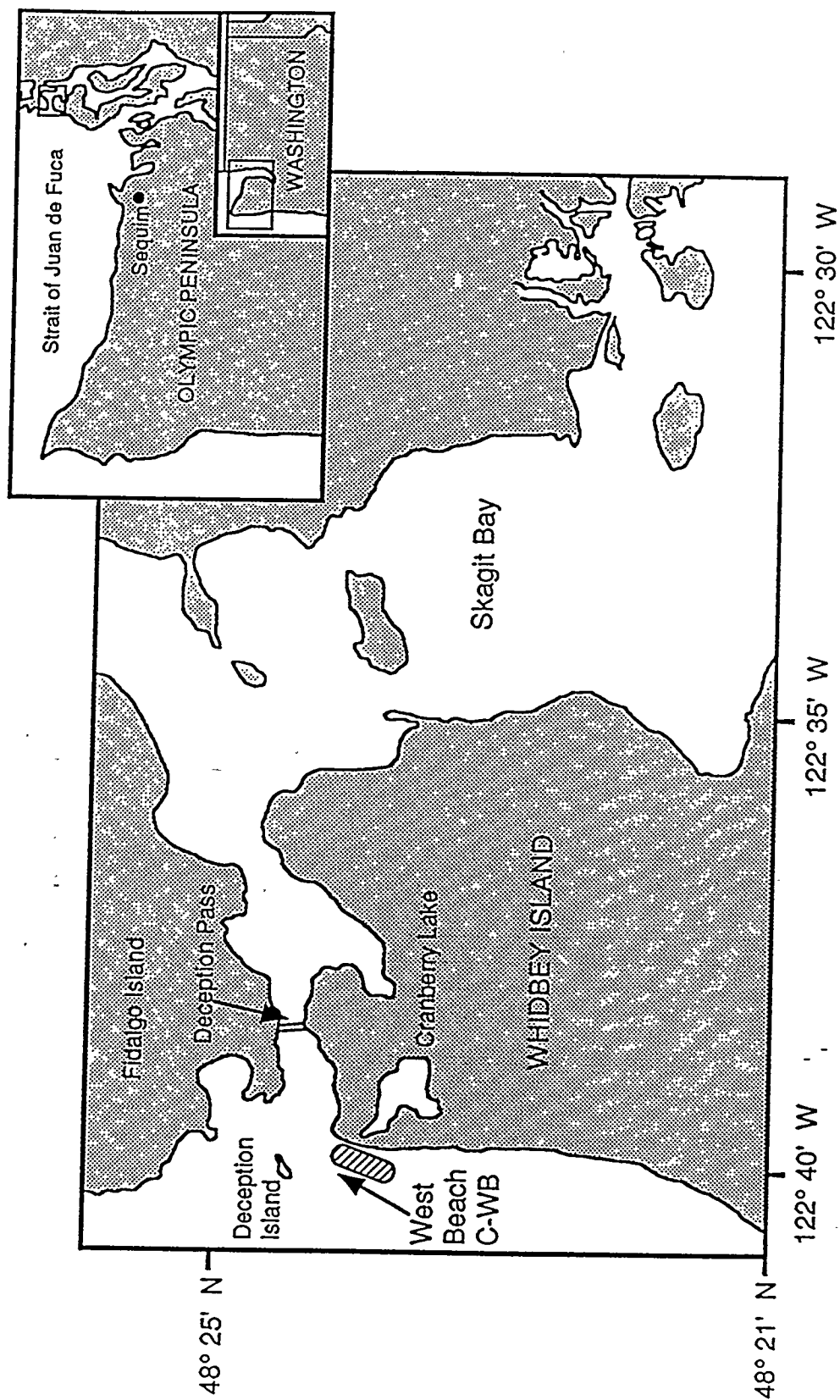


FIGURE 3.5. Location of *R. abronius* Control (C-WB), West Beach, Whidbey Island, Washington

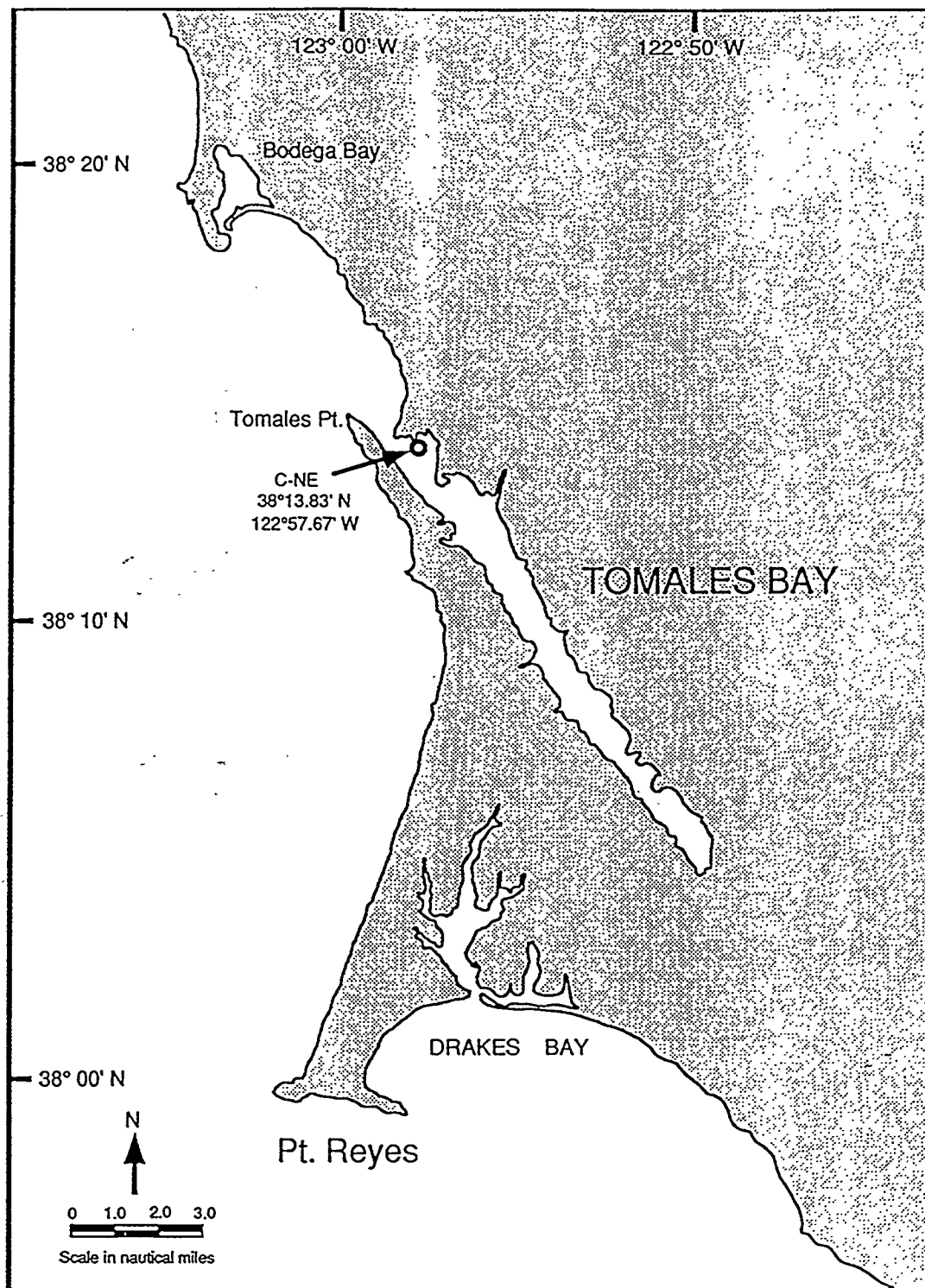


FIGURE 3.6. Location of *N. caecoides* Control (C-NE), Tomales Bay, California

control for the amphipod, *R. abronius*. Tomales Bay is the source of native sediment for the polychaete, *N. caecoides*, and was used in the 10-day toxicity test.

The sediment for the 1993 study was collected with a 4-in. core barrel. The standard procedure for processing sediment involves cutting the 4-in. core in half longitudinally, brushing off all in the Lexan shavings before opening the core, and then taking a pie-shaped wedge out of the sediment, using care to avoid processing any sediment that could have come in contact with the core tube. This is important because when each core is collected, the sediment is pushed up through the core tube and the upper, more contaminated, layers come in contact with the entire length of core. For the 1993 study, most of the sediment in the core tube was needed for testing; therefore, it is possible that the OBM sediments that came in contact with the core tube could have been processed and tested. The results obtained for the 1994 study, particularly for oil, grease, and pesticides, do not confirm those of the 1993 study. The 1994 data were collected using the 12-in. core barrel, and great care was taken to ensure that cross-contamination of YBM and OBM would not be an issue. For these reasons, the 1993 data should not be used to make decisions regarding sediment suitability.

3.2 GEOLOGIC DESCRIPTIONS

Marine sediment throughout Inner Richmond Harbor consists of two geologic units: the YBM and the underlying OBM (Goldman 1969). This report focuses on the OBM sediments that are fine-grained, consolidated, and generally olive or gray in color. It is believed that the OBM was deposited in shallow marine and terrestrial environments during the interglacial epoch immediately preceding the formation of the Wisconsin ice sheet, and was exposed and subjected to erosion during the first Wisconsinian glacial advance (Goldman 1969). While exposed, the OBM sediments desiccated and consolidated. The OBM is up to 200 ft thick, but appears to be about 50 ft thick in the Richmond area. The OBM in Richmond Harbor is underlain by interbedded sandstone and shale bedrock of the Franciscan Formation, which outcrops at Point Potrero (Helley and Lajoie 1979).

During the January 1993 study, sediment was included in the composite if more than 1.5 ft of OBM remained in the 4-in. core after the required sediment was removed for the other MSL projects. The OBM COMPs from the January 1994 and October 1994 studies were collected from the -40 ft MLLW to -42 ft MLLW section of the 12-in. cores. The OBM collected from the Inner Harbor Channel was composed of firm to hard homogeneous silts and clays. The silt and clay was generally dark gray or olive brown in color. Gypsum nodules up to 6 in. in diameter and roots were observed in some cores.

3.3 SEDIMENT SAMPLE PREPARATION

The core samples for each composite were combined, mixed, and homogenized into one sediment composite that was analyzed for selected chemical parameters and used in the biological tests. During the January 1993 study, sediment from the native controls for *R. abronius* and *M. nasuta* were also processed and sampled for chemical analysis. For the January 1994 and October 1994 studies, the three reference samples were collected and used for comparison purposes in the biological and chemical tests. An aliquot of the control samples collected for the January 1994 and October 1994 studies were archived for future potential chemical analysis, and the remainder was used for validating the biological tests. Table 3.2 presents the sediment treatment strategy and the appropriate chemistry and/or toxicity testing for the OBM sediment treatments.

3.4 SEDIMENT CHEMISTRY RESULTS

The OBM COMPs, control sediments, and reference sediments were analyzed for a variety of selected contaminants. The sediments collected in the January 1993 and October 1994 studies were analyzed for the following: conventional parameters (grain size, TOC, oil and grease, TPH, total solids and total volatile solids), PAHs, chlorinated pesticides, PCBs, metals, and butyltins. The sediments collected in the January 1994 study were only analyzed for PCBs, pesticides, and butyltins. These three parameters were analyzed again to confirm the results of the 1993 study. Section 3.4.1 summarizes the chemical attributes of OBM sediments. Section 3.4.2 is a comparison of OBM sediments with reference sediments to determine appropriate disposal of OBM sediments. The complete sediment chemistry results, and quality control summaries and results, are presented in Appendix A.

3.4.1 Physical and Chemical Attributes of OBM Sediments from Richmond Harbor

Older bay mud sediments are primarily fine-grained (82% to 85% silt or clay) with low concentrations of TOC (0.16% to 0.18%), oil and grease (undetected at 11 mg/kg 1994 data only), and TPH (undetected at 11 mg/kg) as shown in Table 3.3. The concentrations of total volatile solids (1.9% to 2.3%) are higher than would be predicted when compared with the low TOC content of this sediment. The OBM composite from the 1993 and 1994 studies had low total PAH concentrations (12 µg/kg to 57 µg/kg), undetected levels of pesticides (except 4,4'-DDD in January 1993), PCBs, and organotins. Metals were detected in the OBM composites from the 1993 and October 1994 studies at levels similar to those obtained for the control sediments (1993 study) and the reference sediments (October 1994 study).

TABLE 3.2. Summary of Sediment Treatment Strategy for Chemical and Biological Testing, Older Bay Mud Studies

Sediment Composite	Sediment Chemistry	Solid-Phase Toxicity	SPP Toxicity	Bioaccumulation Test
<u>January 1993</u>				
OBM COMP	YES	YES	YES	YES
C-WB	YES	YES	NO	NO
C-SPB	YES	YES	NO	NO
C-SB	YES	YES	NO	YES
<u>January 1994</u>				
OBM COMP	YES	YES	YES	YES
R-OS	YES	YES	NO	YES
R-BF	YES	YES	NO	YES
R-AM	YES	YES	NO	YES
C-SB	NO	YES	NO	YES
C-WB	NO	YES	NO	YES
C-NE	NO	YES	NO	YES
<u>October 1994</u>				
OBM COMP	YES	YES	NO	NO
R-OS	YES	YES	NO	NO
R-BF	YES	YES	NO	NO
R-AM	YES	YES	NO	NO
C-SB	NO	YES	NO	NO
C-WB	NO	YES	NO	NO
C-NE	NO	YES	NO	NO

3.4.2 Conventional Measurements

Conventional parameters measured in sediment are summarized in Table 3.4. The conventional results and quality control data for all the conventional parameters are presented in Appendix A, Tables A.1 through A.5. All sediment parameters are discussed on a dry weight basis.

The grain size results for the OBM COMP were similar for the January 1993 and October 1994 studies and showed that the OBM COMP was composed primarily of fine-grained sediments represented by 82% to 85% of the sediment in the silt and clay categories. Two of

TABLE 3.3. Physical and Chemical Results of the Older Bay Mud Sediments, the Older Bay Mud Studies

Analyte	January 1993	January 1994	October 1994	Mean
<u>Percent</u>				
gravel	0	NM(a)	0	0
sand	15	NM	18	16.5
silt	47	NM	56	51.5
clay	38	NM	26	32
TOC	0.18	NM	0.16	0.17
Solids	76.4	NM	74.1	75.3
TVS	2.29	NM	1.90	2.1
<u>mg/kg</u>				
oil & grease	501(b)	NM	11 U(c)	11 U
TPH	NM	NM	11 U	11 U
<u>µg/kg</u>				
TPAH	21.3	12	57	30.1
4,4'-DDD	1.85	0.13 U	0.22 U	NA(d)
<u>mg/kg</u>				
Ag	0.11	NM	0.11	0.11
As	3.30	NM	3.28	3.29
Cd	0.84	NM	0.56	0.70
Cr	148	NM	142	145
Cu	32.9	NM	27.4	30.2
Hg	0.057	NM	0.044	0.05
Ni	70.0	NM	62.7	66.5
Pb	14.6	NM	10.6	12.6
Se	0.30	NM	0.17 U	NA
Zn	72.4	NM	68.3	70.1

(a) NM Not measured for this study.

(b) Value probably reflects cross-contamination, and is not used for interpretation of results.

(c) U Undetected at or above the detection limit.

(d) NA Not applicable.

TABLE 3.4. Conventional Sediment Measurement Results for the Older Bay Mud Studies

Parameters	January 1993				October 1994					
	OBM	COMP	C-SB	C-WB	C-SPB	OBM	COMP	R-OS	R-BF	R-AM(a)
<u>Percent dry weight</u>										
Gravel	0		0	0	0(b)	0		0	0	0
Sand	15		33	99	12(b)	18		26	1	99
Silt	47		36	0	47(b)	56		53	39	0
Clay	38		31	1	42(b)	26		21	60	1
<u>Percent Total Solids</u>	76.4		31.6(b)	78.0	36.0	74.1		53.0	36.5	82.4(b)
<u>TOC for OBM</u>										
OBM-0.4% TOC(c)	0.18(b)		2.01	0.06	1.23	0.16		1.0	1.4	0.096(b)
OBM-0.6% TOC(c)	NA(d)		NA	NA	NA	0.24		NA	NA	NA
OBM-1.0% TOC(c)	NA		NA	NA	NA	0.27		NA	NA	NA
OBM-1.4% TOC(c)	NA		NA	NA	NA	0.39		NA	NA	NA
			NA	NA	NA	0.44		NA	NA	NA
<u>Percent TVS</u>	2.29		2.15(b)	0.496	2.08	1.9		2.4	2.3	0.6
<u>mg/kg dry weight</u>										
Oil and Grease	501		1380	327(b)	772	11 U		100	130	10 U
TPH	NA		NA	NA	NA	11 U		77	100	10 U

(a) Composite of R-AM stations excluding R-AM-F.

(b) Mean of replicate values.

(c) Prior to analysis, TOC was estimated in the OBM sediments; the actual values obtained from analysis are shown in the TOC column.

(d) NA Not applicable.

the control sediments were also fine-grained with 67% and 89% silt and clay for C-SB and C-SPB, respectively. The control C-WB and reference R-AM were $\geq 99\%$ sand. Two reference sediments, R-OS and R-BF, were composed primarily of silt and clay (74% and 99% fine-grained material, respectively).

The percentages of TOC found in the OBM COMP were comparable across studies and ranged from 0.16% to 0.18% dry weight. The percentages of TOC in the control sediments ranged from 0.06% in C-WB to 2.01% in C-SB. The reference sediments had percentages of TOC ranging from 0.096% in R-AM to 1.4% in R-BF. Fine-grained reference sediments had higher percentages of TOC than coarser-grained sediments. When OBM was amended with *Enteromorpha* spp. during the definitive feeding study, the percentages of TOC in the OBM sediments ranged from 0.24% to 0.44%.

Higher TVS percentages were found in the finer-grained sediments. The TVS percentages in the OBM COMP ranged from 1.9% to 2.29% dry weight, control sediments ranged from 0.5% in C-WB to 2.2% in C-SB, and reference sediments ranged from 0.6% in R-AM to 2.4% in R-OS.

The oil and grease concentration in the OBM COMP was 501 mg/kg in the January 1993 study and undetected in the October 1994 study. The concentration of oil and grease in the control sediments ranged from 327 mg/kg in C-WB to 1380 mg/kg in C-SB. The oil and grease concentrations in the reference sediments ranged from undetected in R-AM sediment to 130 mg/kg dry weight in R-BF. Oil and grease values for the OBM COMP are much higher in the January 1993 data relative to the 1994 data. Although the QA/QC data are acceptable, the oil and grease data for the 1993 study are probably cross-contaminated with YBM sediments and are not used to evaluate OBM sediments. The analysis performed in October 1994 shows that TPH was not detected in the OBM COMP and that the concentrations in the reference sediments ranged from undetected in R-AM sediments to 100 mg/kg dry weight in R-BF.

3.4.3 Polynuclear Aromatic Hydrocarbons

Sixteen PAHs were analyzed in the OBM COMP, the control sediments, and the reference sediments. The PAHs are reported in units of $\mu\text{g/kg}$ as LPAHs and HPAHs. The quality control summaries and results for PAH analysis are located in Appendix A, Tables A.6 through A.11.

Table 3.5 summarizes the LPAH, HPAH, and total PAH concentrations in sediments. The results of PAH analysis obtained for all three studies were similar, indicating that the PAH load is similar in OBM sediments throughout Richmond Harbor and has not increased substantially during these studies. The concentrations of LPAHs in the OBM COMP were either not detected or ranged from 8 $\mu\text{g/kg}$ to 29 $\mu\text{g/kg}$ dry weight. The HPAH concentrations in the OBM COMP

TABLE 3.5. Detected Total PAHs, Pesticides, PCBs, Metals, and Butyltins in the Three Older Bay Mud Studies

Parameters	Organic Concentrations in $\mu\text{g/kg}$ dry weight: Metals Concentrations in mg/kg dry weight									
	January 1993					January 1994				
	OBM COMPI(a)	C-SB	C-WB	C-SPB(a)	OBM COMP	R-OS	R-BF	R-AM	OBM COMP	October 1994 R-OS R-BF R-AM(b)
Total LPAH(c)	0	54	0	189	8	48	228	1177	29	65 355 538
Total HPAH(c)	21	216	0	1618	4	68	1527	2426	28	124 2116 1055
Total PAH(c)	21	270	0	1807	12	116	1755	3603	57	189 2471 1593
Aldrin	0.75 U(d)	1.4 U	1.0 U	1.6 U	0.05 U	0.06 U	5.07	0.04 U	0.18 U	0.25 U 2.15 1.32
4,4'-DDD	1.85	5.0 U	2.0 U	2.8 UJ(e)	0.13 U	0.18 U	1.05	0.45	0.22 U	0.31 U 5.19 0.21 U
4,4'-DDE	1.5 U	2.9 U	2.0 U	2.65 UJ	0.06 U	0.94	1.69	0.05 U	0.12 U	1.29 2.61 0.11 U
4,4'-DDT	1.11 UJ	2.9 U	2.0 U	3.2 U	0.38 U	0.51 U	12.1	0.52	0.63 U	0.87 U 1.19 U 0.58 U
Dieldrin	1.5 U	2.9 U	2.0 U	3.2 U	0.28 U	0.38 U	13.0	0.28 U	0.18 U	0.25 U 0.33 U 0.16 U
Aroclor-1254	15 U	29 U	15 U	32 U	20 U	20 U	23.3	20 U	20 U	20 U 46.8 20 U
Silver	0.11	0.18	0.01	0.30	NA(f)	0.556	0.530	0.052	0.11	0.44 0.44 0.026
Arsenic	3.30	10.4	2.15	13.0	NA	2.19 J(g)	11.4	7.7	3.28	5.39 9.4 5.88
Cadmium	0.84	0.93	0.11	0.24	NA	0.489	0.205	0.043	0.56	0.35 0.14 0.015
Chromium	148	96.0	234	166.5	NA	171	178	118	142	186 178 95.4
Copper	32.9	29.9	10.6	53.4	NA	23.5	57.3	10.0	27.4	31.8 60.6 4.30
Mercury	0.057	0.073	0.023	0.342	NA	0.084	0.373	0.039	0.044	0.123 0.315 0.049
Nickel	70.0	40.3	40.4	98.4	NA	68.1	103	38.0	62.7	77.3 111 33.6
Lead	14.6	8.90	6.10	28.1	NA	10.8	30.5	18.3	10.6	8.0 35.3 13.9
Selenium	0.30	0.86	0.13 U	0.22	NA	1.25	0.417	0.128 U	0.17 U	1.34 0.29 0.17 U
Zinc	72.4	83.0	43.3	122	NA	84.3	145	35.6	68.3	94.7 158 32.8
Tributyltin	8.0 U	8.0 U	8.0 U	3.8 J	0.40 U	0.40 U	0.40 U	0.40 U	0.48 U	0.62 2.27 0.48 U
Dibutyltin	2.2 BJ(h)	2.0 BJ	5.0 U	5.0 U	0.65 U	0.65 U	0.65 U	0.65 U	0.56 U	0.56 U 2.11 0.56 U

(a) Mean of replicate values.

(b) Composite of R-AM stations excluding R-AM-F.

(c) Values are the sum of detected values.

(d) U Undetected at or above the detection limit.

(e) UJ Undetected or detected below the method detection limit in all replicates.

(f) NA Not applicable.

(g) J Analyte was detected below the method detection limit (MDL), but above the instrument detection limit (IDL).

(h) BJ All replicates had analytes that were either detected below the method detection limit or present in the blank associated with the samples.

ranged from 4 µg/kg to 28 µg/kg dry weight. The highest total PAH concentrations in the control sediments was C-SPB (1807 µg/kg dry weight). In the January 1994 study, the highest total PAH concentrations in the reference sediments was found in R-AM (3603 µg/kg dry weight). Measurements of the individual stations comprising R-AM were also analyzed in 1993. Station R-AM-F had high levels of PAHs and was subsequently excluded from the R-AM composite tested in the October 1994 study. The reference sediment R-BF had the highest concentration of total PAHs of 2471 µg/kg dry weight for the October 1994 study.

3.4.4 Chlorinated Pesticides and Polychlorinated Biphenyls

Chlorinated pesticide and PCB concentrations that were detected in the sediment treatments are summarized in Table 3.5. All data associated with these summaries and related QA measurements can be found in Appendix A, Tables A.12 through A.18.

Nineteen chlorinated pesticides and four PCBs as Aroclors were analyzed in the sediment treatments. Most of the pesticide and PCB compounds were not detected in the OBM COMP above the target detection limits of 2 µg/kg and 20 µg/kg, respectively. The OBM COMP had detectable concentrations of 4,4'-DDD and 4,4'-DDT with values of 1.85 µg/kg (average of three replicates) and 1.11 µg/kg, respectively. The control sediment C-SPB had a mean detectable concentrations of 4,4'-DDE (2.7 µg/kg) and 4,4'-DDD (2.8 µg/kg). The three reference sediments had detected concentrations of one or more of the pesticides shown in Table 3.5, but at concentrations ≤13 µg/kg dry weight. No PCBs were detected in the OBM COMP or the control sediments above the detection limit of 20 µg/kg. Aroclor 1254 was detected in R-BF sediments for both the January 1994 and October 1994 studies.

3.4.5 Metals

Ten metals were analyzed in the sediment treatments. All results and quality control summaries are found in Appendix A, Tables A.19 and A.20. Table 3.5 shows that all 10 metals were detected in the OBM COMP, the control sediments, and the reference sediments. The metals results were similar for the OBM sediment analyzed during the 1993 and 1994 studies.

3.4.6 Butyltins

Butyltin concentrations are summarized in Table 3.5. All data associated with these summaries and related quality assurance measurements are found in Appendix A, Tables A.21 and A.22.

Tributyltin was not detected in the OBM COMP. Dibutyltin was either undetected or detected in the OBM COMP at an average concentration of 2.2 µg/kg; however, these levels are probably due to blank contamination. Tributyltin was present only in C-SPB at a concentration of 3.8 µg/kg. Dibutyltins were found in C-SB at 2.0 µg/kg and are also probably caused by blank contamination. The reference sediments R-OS and R-BF analyzed in the October 1994

study had detected concentrations of tributyltin and/or dibutyltin at concentrations ranging from 0.62 µg/kg to 2.27 µg/kg dry weight.

3.5 TOXICOLOGICAL TESTING RESULTS

In January 1993, several bioassays were conducted to assess the toxicity of potential contaminants from OBM sediments: a solid-phase test using *R. abronius*, two SPP tests using *H. costata* and the bivalve *M. galloprovincialis*, and a bioaccumulation test with *M. nasuta* was performed. In January 1994, three solid-phase tests using *M. nasuta*, *N. caecoides*, and *R. abronius*; three SPP tests using *C. stigmaeus*, *H. costata*, and *M. galloprovincialis*; and a bioaccumulation test with *M. nasuta* were performed. In October 1994, a solid-phase test using *N. caecoides* exposed to varying concentrations of TOC and food sources was performed. The appropriate controls for each species were also used in the toxicity tests to ensure test validity by acceptable control survival. Three reference sediments were tested concurrently with the OBM COMP for the January 1993 and 1994 studies. The reference results were compared with the OBM results to provide information regarding suitable disposal options for OBM sediments. The results of the bioassay testing are presented in Appendixes B through H.

3.5.1 10-Day Solid-Phase Static Test with *R. abronius*

The results of the 10-day solid-phase static test with *R. abronius* are presented in Table 3.6. The water quality data and test results are in Appendix B. The water quality data for the 10-day test and the 96-h Cd reference toxicant tests were all within the acceptable ranges established in the QAP, with the exception of salinity in one OBM COMP replicate and pH in a C-SB replicate. These water quality exceedences did not appear to affect the validity of the test. In the January 1993 study, survival of *R. abronius* exposed to the OBM COMP was 85% and the test was validated by 98% survival in the native control sediment C-WB. In the January 1994 study, survival of *R. abronius* exposed to the OBM COMP was 72% and the test was validated by 98% survival in the control sediment C-WB. Survival of *R. abronius* exposed to the three reference sediments ranged from 89% to 99%.

Results of the Dunn's Test showed that the difference in survival of *R. abronius* exposed to the OBM COMP tested in both studies was not statistically significant. The difference in survival in OBM COMP and in R-AM from the January 1993 study was statistically significant but since the difference in survival was not >20%, it is not considered acutely toxic. The OBM COMP from the January 1994 study was statistically significantly different and had a >20% difference in survival from both R-AM and R-BF.

TABLE 3.6. Summary Results of the 10-Day Solid-Phase Static Test with *R. abronius*

Sediment Treatment	Mean Percent Survival	Statistical Grouping
<u>January 1993</u>		
OBM COMP	85	ab
C-WB	98	NA(a)
<u>January 1994</u>		
OBM COMP	72	a
R-OS	89	b
R-BF	94	bc
R-AM	99	c
C-WB	98	NA
C-SB	93	NA
(a) NA Not applicable.		

The Cd reference toxicant test for the January 1993 and January 1994 studies produced LC₅₀ values of 0.65 mg/L and 0.90 mg/L, respectively. These values are similar to those from previous MSL studies that provided a reference toxicant range of 0.4 to 1.94 mg Cd/L.

3.5.2 10-Day Solid-Phase Flow-Through Test with *M. nasuta* and *N. caecoides*

The results of the 10-day solid-phase flow-through test with *M. nasuta* and *N. caecoides* are presented in Tables 3.7 and 3.8. The water quality data and test results are presented in Appendix C. The water quality data for the 10-day test were all within the acceptable ranges established in the QAP. *M. nasuta* exposed to the OBM COMP had a survival of 98%, which was not statistically significantly different from the survival in the three reference sediments (all had 100% survival). This test was validated by 100% survival of *M. nasuta* exposed to the native control sediment C-SB. The survival of *N. caecoides* exposed to the OBM COMP was 38%, whereas the survival in the reference sediments and control sediments ranged from 91% to 96%. The OBM COMP was acutely toxic to *N. caecoides*, shown by the statistically significant difference and the ≥10% difference in survival of the organisms after exposure to each of the three reference sediments.

3.5.3 28-Day Solid-Phase Flow-Through Bioaccumulation Test with *M. nasuta*

The results of the 28-day solid-phase flow-through test with *M. nasuta* are presented in Table 3.9. The water quality data and test results are presented in Appendix D. The water quality data for the 28-day test were all within the acceptable ranges established in the QAP, with the exception of temperature measurements in a few replicates that were as high as 17.3°C (target range of 13°C to 17°C). These exceedences did not appear to affect the validity of the

TABLE 3.7. Summary Results of the 10-Day Solid-Phase Flow-Through Test with *M. nasuta*

Sediment Treatment	Mean Percent Survival	Statistical Grouping
<u>January 1994</u>		
OBM COMP	98	a
R-OS	100	a
R-BF	100	a
R-AM	100	a
C-SB	100	NA(a)
C-NE	100	NA
(a) NA Not applicable.		

test. Statistical comparisons were not performed on this data, since the purpose of the 28-day solid-phase test was to provide information regarding the bioaccumulation potential of the OBM COMP. The January 1993 study results showed that survival of *M. nasuta* exposed to the OBM COMP was greater than 97%, with control survival exceeding 98%. In the January 1994 study, *M. nasuta* survival in the OBM COMP was 94% and the survival in the three reference sediments and one control sediment ranged from 91% to 96%.

3.5.4 48-Hour Suspended-Particulate-Phase Static Test with *M. galloprovincialis*

The results of the 48-h SPP static tests with *M. galloprovincialis* are summarized in Table 3.10. The water quality data and test results are presented in Appendix E. The water quality data for the SPP test and the reference toxicant test were all within acceptable ranges.

The mean percent survival was calculated by adding the number of normal D-shaped larvae, the abnormal larvae, and the other larvae and dividing the total by the average stocking density larval count. In the January 1993 study, the test was validated by 100% survival in the

TABLE 3.8. Summary Results of the 10-Day Solid-Phase Flow-Through Test with *N. caecoides*

Sediment Treatment	Mean Percent Survival	Statistical Grouping
<u>January 1994</u>		
OBM COMP	38	a
R-OS	96	b
R-BF	91	b
R-AM	96	b
C-NE	94	NA(a)
C-SB	96	NA

TABLE 3.9. Summary Results of the 28-Day Solid-Phase Flow-Through Test with *M. nasuta*

Sediment Treatment	Mean Percent Survival
<u>January 1993</u>	
OBM COMP	97
C-SB	99
<u>January 1994</u>	
OBM COMP	94
R-OS	96
R-BF	94
R-AM	94
C-SB	91

TABLE 3.10. Summary Results of the 48-Hour-Suspended-Particulate-Phase Test with *M. galloprovincialis*

Sediment Treatment	Percent SPP	Mean Proportion Survival	Mean Proportion Normal
<u>January 1993</u>			
OBM COMP	0	1.00	0.99
OBM COMP	10	0.99	0.98
OBM COMP	50	0.97	0.96
OBM COMP	100	1.00	1.00
<u>January 1994</u>			
OBM COMP	0	0.98	0.98
OBM COMP	10	0.96	0.95
OBM COMP	50	0.91	0.91
OBM COMP	100	0.98	0.96

control (0% SPP). Survival was 97% or greater for the 0%, 10%, 50%, and 100% SPP preparations. In the January 1994 study, the test was validated by 98% survival in the control (0% SPP). Survival was 91% or greater for all SPP preparations. These results indicate that the OBM COMP was not acutely toxic to *M. galloprovincialis*.

The mean percent normal development was calculated by dividing the normal D-shaped larvae by the average stocking density larval count. In the January 1993 study, the percentage of normal larvae was 96% or greater for all SPP preparations. In the January 1994 study, the percentage normal larvae was 91% or greater for all SPP preparations. These results indicate that the SPP made from the OBM COMP did not affect the development of *M. galloprovincialis* larvae.

The Cu reference toxicant tests for the January 1993 and January 1994 studies produced LC₅₀ values of 15.99 µg/L and 12.1 µg/L, respectively, and EC₅₀ values of 8.13 µg/L and 8.0 µg/L, respectively. These responses were within the MSL ranges previously established of 5.8 to 35 µg/L of copper for the LC₅₀ and 5.7 to 21 µg/L of copper for the EC₅₀, indicating comparable test organism sensitivity. The ammonia reference toxicant test for the January 1994 study produced an LC₅₀ value of 43.2 mg/L total ammonia and an EC₅₀ value of 2.76 mg/L total ammonia. A database of the response of *M. galloprovincialis* to ammonia is currently being established; therefore, an acceptable range of sensitivity has not been determined.

3.5.5 96-Hour Suspended-Particulate-Phase Static Test with *C. stigmaeus*

The results of the 96-h SPP test with *C. stigmaeus* are presented in Table 3.11. The water quality data and test results are presented in Appendix F. The water quality data for the test and reference toxicant tests were all within the acceptable ranges established in the QAP. The test was validated by 100% survival of *C. stigmaeus* in the 0% SPP. The survival of *C. stigmaeus* exposed to the OBM COMP was 100% in the 10% SPP, 98% in the 50% SPP, and 100% in the 100% SPP. An LC₅₀ value could not be calculated because there was not a 50% reduction in survival of *C. stigmaeus*.

The Cu reference toxicant test produced an LC₅₀ value of 0.88 mg/L Cu. These results are slightly below the LC₅₀ range established from previous MSL studies (1.2 to 1.6 mg/L Cu) indicating that the test organisms were slightly more sensitive than species used in previous studies. This does not appear to have affected the results, based on the high survival of *C. stigmaeus* in all concentrations of SPP.

The ammonia reference toxicant test produced an LC₅₀ value of 23.9 mg/L total ammonia. One other study has been conducted at the MSL with ammonia and *C. stigmaeus*; it resulted in an LC₅₀ value of 30.9 mg/L total ammonia.

TABLE 3.11. Summary Results of the 96-Hour Suspended-Particulate-Phase Test with *C. stigmaeus*

Sediment Treatment	Percent SPP	Mean Percent Survival
<u>January 1994</u>		
OBM COMP	0	100
OBM COMP	10	100
OBM COMP	50	98
OBM COMP	100	100

3.5.6 96-Hour Suspended-Particulate-Phase Static Test with *H. costata*

The results of the 96-h SPP test with *H. costata* are presented in Table 3.12. The water quality data and test results are presented in Appendix G. The water quality data for the test and reference toxicant tests were all within the acceptable ranges established in the QAP. In the January 1993 study, the OBM COMP had survival of 93% for the 10%, 50%, and 100% SPP, indicating no toxicity to any of the SPP concentrations. The *H. costata* test was validated by 93% survival in the control (0% SPP). In the January 1994 study, the OBM COMP had survival of 94% in the 10% SPP, 98% in the 50% SPP, and 96% in the 100% SPP, again indicating no toxicity to any of the SPP concentrations. The test was validated by 98% survival in the control (0% SPP).

TABLE 3.12. Summary Results of the 96-Hour Suspended-Particulate-Phase Test with *H. costata*

Sediment Treatment	Percent SPP	Mean Percent Survival
<u>January 1993</u>		
OBM COMP	0	93
OBM COMP	10	93
OBM COMP	50	93
OBM COMP	100	93
<u>January 1994</u>		
OBM COMP	0	98
OBM COMP	10	94
OBM COMP	50	98
OBM COMP	100	96

The Cu reference toxicant tests for the January 1993 and January 1994 studies produced LC₅₀ values of 120 µg/L Cu and 70.7 mg/L Cu, respectively. The results of the *H. costata* copper reference toxicant test conducted previously at the MSL produced an LC₅₀ value of 68.3 µg/L Cu. The ammonia reference toxicant test for the January 1994 study produced an LC₅₀ value of 39.8 mg/L total ammonia. Currently, there is no database of ammonia effects on *H. costata*.

3.5.7 10-Day Solid-Phase Flow-Through Feeding Tests with *N. caecoides*

The OBM COMP was acutely toxic to *N. caecoides* during the Richmond Harbor Project conducted in January 1994. The consolidated character of OBM and its relatively low TOC content (average of 0.17%) create an environment in which the organisms have a hard time burrowing into the sediment and finding an adequate food supply.

To further assess the toxicity of OBM sediments to *N. caecoides*, two tests, referred to as the preliminary test and the definitive test, were conducted in October 1994 using sediment from stations RC-12, RC-17, RC-21, and RC-30 that were composited to form one OBM COMP (Figure 1.1). The purpose of these tests was to determine whether the addition of a food source, or the softening of OBM sediments with filtered seawater, increases the survival of this test species exposed to OBM sediments.

3.5.7.1 Results of the Preliminary Feeding Study

The results of the preliminary feeding study are shown in Table 3.13. This test was validated by 99% survival of *N. caecoides* in their native control sediment (C-NE). The survival of *N. caecoides* in the OBM in the absence of additional food or water was 32%. The OBM sediment softened with the addition of seawater had an increase in survival to 77%. The addition of food to the OBM showed survival ranging from 63% to 87%.

A statistical analysis was performed that compared all the sediment treatments (with the exception of the control) with one another. The Dunn's test was conducted with an $\alpha = 0.05$ using the arcsine square-root transformation of the mean proportion surviving data. This comparison, shown in Table 3.13, indicates that there was a statistically significant decrease in survival of *N. caecoides* exposed to the untreated OBM COMP sediment when compared with all food types and methods, except OBM-0.8% TOC (sprinkled with tetramin). The 0.8% TOC (estimated measurement) appeared to be the optimum level of TOC for *N. caecoides*, as all three food types had greater than 85% survival using the mixed method. Two treatments, OBM-0.8% TOC (sprinkled with tetramin) and OBM-1.2% TOC (mixed with alfalfa), had *N. caecoides* survival values of 63% and 67%, respectively.

TABLE 3.13. Summary Results for the 10-Day *N. caecoides* Preliminary Test

Sediment Treatment	Mean Percent Survival	Statistical Grouping
OBM COMP	32	a
OBM-0.8% TOC (sprinkled with tetramin)	63	ab
OBM-1.2% TOC (mixed with alfalfa)	67	b
OBM with water	77	b
OBM-0.4% TOC (mixed with alfalfa)	81	b
OBM-0.8% TOC (mixed with tetramin)	85	bc
OBM-0.8% TOC (sprinkled with <i>Enteromorpha</i> spp.)	85	bc
OBM-0.8% TOC (mixed with alfalfa)	87	bc
OBM-0.8% TOC (mixed with <i>Enteromorpha</i> spp.)	87	bc
OBM-0.8% TOC (sprinkled with alfalfa)	87	bc
C-NE	99	c

3.5.7.2 Results of the Definitive Study

The *Enteromorpha* spp. was chosen as the food source for the definitive study using the roller method technique. This food source was chosen because it is found in nature and represents a potential food source most likely encountered by the test species. The experimental design included OBM sediment, OBM sediment softened with filtered seawater, and OBM mixed with food, to represent TOC concentrations (including TOC already present in the OBM sediment) of 0.4%, 0.6%, 1.0%, and 1.4%. These were estimated TOC levels based on the assumption that all of the *Enteromorpha* spp. added to the sediment was TOC. Aliquots of the OBM sediment mixed with *Enteromorpha* spp. were then sent to ARI for analysis of TOC. The actual TOC levels present in the OBM sediment were approximately half of the estimated values. The actual levels of TOC present in the OBM sediment and the results are shown in Table 3.14.

TABLE 3.14. Measurements of TOC in the OBM Sediments Mixed with Different Amounts of *Enteromorpha* spp.

Sediment Treatment	Estimate of % TOC	Actual % TOC
OBM COMP	0.08	0.16
OBM COMP + 2.45 g of <i>Enteromorpha</i> spp.	0.4	0.24
OBM COMP + 4.69 g of <i>Enteromorpha</i> spp.	0.6	0.27
OBM COMP + 9.15 g of <i>Enteromorpha</i> spp.	1.0	0.39
OBM COMP + 13.62 g of <i>Enteromorpha</i> spp.	1.4	0.44

The results of this study confirmed those of the preliminary study and suggest that when OBM is softened with seawater, *N. caecoides* are able to burrow into the sediment and their survival is increased to 65% (Table 3.15). The addition of food also enhanced *N. caecoides* survival in this softened sediment with a range in survival of 78% to 89%. This test was validated by 100% survival in the native control sediment C-NE.

The percent survival results of the definitive study were compared to the survival results from the OBM COMP and the reference treatments tested in January 1994 using the Bonferroni/Dunn multiple comparison test. This comparison showed that the OBM sediments, when softened and amended with food, were not acutely toxic to *N. caecoides* relative to any of the reference treatments.

3.6 TISSUE BIOACCUMULATION

The bioaccumulation potential of OBM sediments was evaluated with *M. nasuta* in a 28-day solid-phase test. The OBM COMP was tested with the *M. nasuta* control C-SB in January 1993, and with the three reference treatments (R-OS, R-BF, and R-AM) and C-SB in January 1994. The tissues of *M. nasuta* tested in January 1993 were analyzed for PAHs, pesticides, PCBs, metals, and butyltins. The tissues of *M. nasuta* tested in January 1994 were only analyzed for pesticides, PCBs, and butyltins. Complete tissue chemistry results in both wet and dry weight, quality control data and quality control summaries for *M. nasuta* are presented in Appendix I. Table 3.16 shows the mean tissue concentrations of the contaminants that were elevated above the reference treatments. Table 3.16 shows the test treatment mean and the magnitude of elevation above each of the reference treatment means.

3.6.1 Polynuclear Aromatic Hydrocarbon Bioaccumulation in *M. nasuta*

A total of 16 PAH compounds was analyzed in the tissues of *M. nasuta* exposed to the OBM COMP collected in January 1993. Complete *M. nasuta* tissue chemistry data in µg/kg for both wet and dry weight PAH concentrations, quality control data, and quality control summaries are contained in Appendix I, Tables I.1 through I.9. No PAH compounds were detected in the tissues of *M. nasuta* exposed to this composite.

3.6.2 Chlorinated Pesticides and Polychlorinated Biphenyls Bioaccumulation in *M. nasuta*

A total of 19 chlorinated pesticide and 4 PCBs as Aroclors were analyzed in the tissues of *M. nasuta*. Complete *M. nasuta* tissue chemistry data in µg/kg for both wet and dry weight pesticide and PCB concentrations, quality control data, and quality control summaries are contained in Appendix I, Tables I.10 through I.19. A summary of detectable tissue concentrations are presented in Table 3.15. The analytes 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, and dieldrin were statistically significantly elevated in the tissues of *M. nasuta*. The magnitudes of elevation of

TABLE 3.15. Summary of the 10-Day *N. caecoides* Results for the Definitive Test

Sediment Treatment	Mean Percent Survival	Statistical Grouping
October 1994 OBM COMP	20	a
January 1994 OBM COMP	38	ab
OBM with water	65	bc
OBM-0.44% TOC (mixed with <i>Enteromorpha</i> spp.)	78	cd
OBM-0.27% TOC (mixed with <i>Enteromorpha</i> spp.)	80	cd
OBM-0.39% TOC (mixed with <i>Enteromorpha</i> spp.)	87	cd
OBM-0.24% TOC (mixed with <i>Enteromorpha</i> spp.)	89	cd
R-BF	91	d
R-AM	96	d
R-OS	96	d
C-NE	100	NA(a)

(a) NA Not applicable.

4,4'-DDD in *M. nasuta* tissues exposed to the 1993 OBM COMP ranged from 87 times above R-BF to 112 times above R-OS. The *M. nasuta* tissues exposed to the 1994 OBM COMP had no detectable concentrations of any of the pesticides or PCBs analyzed.

3.6.3 Metals Bioaccumulation in *M. nasuta*

Metals bioaccumulation were only evaluated during the 1993 study. Ten metals were analyzed in the tissues of *M. nasuta* exposed to the 1993 OBM COMP, the three reference treatments, the control treatment C-SB, and *M. nasuta* background samples. Metals concentrations in the tissues of *M. nasuta*, expressed in mg/kg dry weight, are presented in Table 3.16. Complete *M. nasuta* tissue chemistry data in both wet and dry weight metals concentrations, quality control data, and quality control summaries are contained in Appendix I, Tables I.20 through I.22. All ten metals were detected in *M. nasuta* tissues. Four metals, Cd, Cr, Cu, and Hg, were statistically significantly higher in *M. nasuta* tissues exposed to OBM COMP relative to at least one reference treatment.

3.6.4 Butyltin Bioaccumulation in *M. nasuta*

Mean butyltin concentrations ($\mu\text{g/kg}$ dry weight) in *M. nasuta* tissues are summarized in Table 3.16. Complete *M. nasuta* tissue chemistry data in both wet and dry weight organotin concentrations, quality control data, and quality control summaries are contained in Appendix I, Tables I.23 and I.24. *M. nasuta* tissues exposed to the 1993 OBM COMP had TBT concentrations ranging from 50.7 $\mu\text{g/kg}$ to 63.6 $\mu\text{g/kg}$ and DBT concentrations ranging from

TABLE 3.16. Tissue Contaminant Concentrations in *M. nasuta* Tissues Exposed to the OBM Comp, Reference Sediments, and Control Sediments for Bioaccumulation Tests (bold indicates test treatment that was statistically significantly different compared to at least one of the three reference treatments)

Parameters	OBM Comp 1993	OBM Comp 1994	R-OS	R-BF	R-AM	C-SB	<i>M. nasuta</i> Background
4,4'-DDD	235.3	3.4 U	2.1 U	2.7 U	2.5 U	1.7 U	2.4 U
4,4'-DDE	4.9	1.01 U	0.66 U	2.27	2.06	0.50 U	2.90
4,4'-DDT	8.6	2.6 U	1.6 U	4.3	5.3	1.3 U	17.5
Dieldrin	18.7	2.3 U	1.5 U	1.9 U	1.7 U	1.2 U	1.6 U
Ag	0.402	---(a)	0.249	0.224	0.241	0.183	0.492
As	28.8	---	23.2	22.3	22.8	24.2	31.2
Cd	0.606	---	0.183	0.175	0.209	0.178	0.254
Cr	2.58	---	2.31	2.39	1.50	1.54	1.13
Cu	20.5	---	12.4	13.4	12.6	12.4	15.1
Hg	0.129	---	0.069	0.095	0.095	0.051	0.063
Ni	3.92	---	4.85	4.41	3.22	3.02	2.62
Pb	1.31	---	1.11	1.99	1.76	1.34	1.34
Se	1.80	---	1.84	1.58	1.57	1.65	1.79
Zn	109	---	97.0	103	99.0	89.4	107
Tributyltin	57.1	13.6	13.8	17.8	27.6	10.8	4.31
Dibutyltin	53.4	10.9	12.3	10.5	10.5	10.1	10.3

(a) --- Not analyzed.

35.0 µg/kg to 61.4 µg/kg. The elevation of both TBT and DBT levels in the 1993 OBM COMP over that of all three reference sediments was statistically significant. Due to the relatively high levels of butyltins in *M. nasuta* tissues, butyltins were reexamined in *M. nasuta* tissues for the January 1994 study. *M. nasuta* tissues exposed to the 1994 OBM COMP had TBT concentrations ranging from 11.5 µg/kg to 15.5 µg/kg, and DBT concentrations ranging from undetected at 9.58 µg/kg to 13.1 µg/kg. The increases in values in the 1994 OBM COMP compared with those of any of the reference sediments did not show statistically significant increases.

4.0 DISCUSSION

This section provides an overall summary of the results from chemical analyses and biological testing of the OBM sediments. The OBM composites tested in the 1993 and 1994 studies were compared, and each was compared with the reference data collected during the 1994 studies using the Dunn's multiple comparison test.

4.1 SEDIMENT CHEMISTRY

OBM sediments were fine-grained with a low TOC content (less than 0.2%) and a higher level of TVS than might be expected relative to the TOC concentrations. Concentrations of oil and grease and TPH were high in the 1993 study, but as stated in Section 3.4.1, those data are suspect and only the 1994 results should be used. The 1994 data showed that oil and grease, TPH, pesticides, PCBs, and butyltins were not detected in the OBM COMP. Metals were found in the OBM COMP at levels lower than or comparable to concentrations found in one or more reference treatment. Typical concentrations of OBM sediment constituents are shown in Table 3.1.

4.2 TOXICOLOGICAL EVALUATIONS

4.2.1 Deposited Sediment (Solid-Phase) Acute Toxicity

Deposited sediment toxicity was determined by exposing *R. abronius*, *M. nasuta* and *N. caecoides* to the OBM sediment, the native control sediments, and the reference sediments. The toxicity results from these tests are presented in Table 4.1. Sediments are considered toxic if mortality is statistically significantly higher and if it exceeds a reference sediment mortality by 20% or greater for *R. abronius*, and 10% or greater for *M. nasuta* or *N. caecoides*. The results of the *R. abronius* test showed that the OBM COMP from the 1993 study was not acutely toxic relative to any reference treatment. The OBM COMP tested in 1994 was acutely toxic to *R. abronius* relative to both of the in-bay reference treatments. The OBM COMP was not acutely toxic to *M. nasuta* relative to any reference treatment. The results of the *N. caecoides* test showed that the OBM COMP from the 1994 study was acutely toxic when compared with all three reference treatments. The feeding studies conducted in October 1994 showed that this toxicity was caused by a combination of the hard-packed nature of the OBM sediment and its relatively low TOC content. When the OBM was softened with seawater, survival increased to 65%; however, the sediment was still acutely toxic to *N. caecoides* relative to all three reference treatments. However, by first softening the OBM sediment and then amending with food, survival increased and the sediment was not considered acutely toxic to *N. caecoides* relative to any reference treatment.

TABLE 4.1. Summary of Solid-Phase Toxicity Results

Sediment Treatment	<i>R. abronius</i>		<i>M. nasuta</i>		<i>N. caecoides</i>	
	Percent Survival	Statistical Grouping	Percent Survival	Statistical Grouping	Percent Survival	Statistical Grouping
<u>January 1993</u>						
OBM COMP	85	ab	NA(a)	NA	NA	NA
C-WB	98	NA	NA	NA	NA	NA
<u>January 1994</u>						
OBM COMP	72	a	98	a	38	a
R-OS	89	b	100	a	96	b
R-BF	94	bc	100	a	91	b
R-AM	99	c	100	a	96	b
C-WB	98	NA	NA	NA	NA	NA
C-SB	93	NA	100	NA	96	NA
C-NE	NA	NA	100	NA	94	NA
(a) NA Not applicable.						

4.2.2 Water Column Effects

Estimates of water column toxicity were evaluated by exposing *M. galloprovincialis*, *C. stigmaeus*, and *H. costata* to three concentrations of SPP and a dilution water control (Sequim Bay seawater). Acute toxicity was determined by statistical comparison of the 0% and 100% SPP using a t-test and calculations of LC₅₀ and EC₅₀ values using the Trimmed Spearman-Kärber estimator. To calculate LC₅₀ or EC₅₀ values, there must be greater than 50% mortality or some other sublethal effect occurring in the sediment treatments. There was not a 50% decrease in survival for any test species or a 50% decrease in the percentage of normal larvae for *M. galloprovincialis* test (Table 4.2). Using 1991 *Implementation Manual* guidelines, the limiting permissible concentrations (LPC) of dissolved plus suspended contaminants cannot exceed 0.01 of the acutely toxic concentration at the boundaries of the disposal site after allowing 4 h for initial mixing. An LPC could not be calculated for OBM, since its SPP was not toxic to the test organisms.

4.3 BIOACCUMULATION RESULTS

The potential for bioaccumulation of contaminants was evaluated through a 28-day solid-phase exposure of *M. nasuta* to OBM sediments followed by chemical analysis of the tissues. *M. nasuta* tissue analyses for the 1993 study consisted of PAHs, pesticides, PCBs, metals, and butyltins. The analyses for the 1994 study consisted of pesticides, PCBs, and butyltins. PAHs

TABLE 4.2. Summary of the Suspended-Particulate-Phase Toxicity Test Results, Older Bay Mud Studies

Sediment Treatment	Percent SPP	48-h <i>M. galloprovincialis</i>		96-h <i>C. stigmaeus</i>	96-h <i>H. costata</i>
		Mean Percent Survival	Mean Percent Normal	Mean Percent Survival	Mean Percent Survival
<u>January 1993</u>					
OBM COMP	0	100	99	NA	93
OBM COMP	10	99	98	NA	93
OBM COMP	50	97	96	NA	93
OBM COMP	100	100	100	NA	93
<u>January 1994</u>					
OBM COMP	0	98	98	100	98
OBM COMP	10	96	95	100	94
OBM COMP	50	91	91	98	98
OBM COMP	100	98	96	100	96

and PCBs were not detected in the tissues of *M. nasuta* exposed to OBM COMP from either study relative to any of the reference treatment tissues; therefore, a statistical evaluation was not conducted. For the 1993 study, the concentrations of four pesticides (4,4'-DDD, 4,4'-DDE, 4,4'-DDT, and dieldrin) were statistically significantly elevated in *M. nasuta* tissues exposed to the OBM COMP. This is contrary to the results of the 1994 study, which showed no detectable concentrations of pesticides in *M. nasuta* tissues. All 10 metals were detected in the tissues of *M. nasuta* exposed to the OBM COMP. Four metals were statistically significantly elevated in *M. nasuta* tissues exposed to the OBM COMP relative to at least one reference treatment. The results of the 1993 study showed that both tributyltin and dibutyltin were statistically significantly elevated in *M. nasuta* tissue exposed to the OBM COMP. The confirmatory tests conducted in 1994 did not reflect the results of the 1993 study. In the 1994 studies, concentrations of tributyltin and dibutyltin were not statistically significantly elevated in *M. nasuta* tissues exposed to the OBM COMP relative to any reference treatment.

One possible explanation for the difference in bioaccumulation results of pesticides and butyltins between the January 1993 and 1994 data could be due to the potential cross-contamination of OBM with YBM. The sediments for the 1993 study were collected using the 4-in. core barrel and then used in the bioaccumulation test. The correct procedure for processing sediments from the 4-in. liner is to split the core vertically and then to remove sediment from the inside of the core, leaving the sediment in contact with the liner behind. However, some of the sediment that came in contact with the liner was inadvertently processed into the

OBM sediment and used for the bioaccumulation studies, as evidenced by the presence of oil and grease, some pesticides, and butyltins in the sediments and the elevation of these compounds in the tissues of *M. nasuta* relative to what was found in tissues exposed to the reference treatment. For the 1994 studies, the sediments were collected with the 12-in. barrel. Sediments were removed from this barrel by opening the hinged door, which cut the sediment sample in half. A wedge of sediment was then taken out of the middle of the sample. This method ensures that sediment in contact with the walls of the core barrel is not included in the sample.

The tissue contaminant levels from OBM were compared to existing Food and Drug Administration (FDA) limits (Table 4.3). *M. nasuta* tissue contaminant concentrations were orders of magnitude below the FDA action limits after exposure to OBM sediments.

4.4 CONCLUSIONS

Table 4.4 is a summary of the biological testing results for the OBM Study. No OBM sediment was acutely toxic to *M. nasuta*, *M. galloprovincialis*, *C. stigmaeus*, and *H. costata* relative to any reference treatment. The results from the 1994 study with *R. abronius* did show acute toxicity of OBM sediment relative to R-BF and R-AM. The *N. caecoides* definitive study showed that the hard-packed nature of the OBM sediment and its low TOC content contributed to its toxicity to *N. caecoides*. It is suspected that the lower *R. abronius* survival when exposed to OBM sediment could also be due to these factors.

Due to the potential cross-contamination problem discussed in Section 4.3, conclusions regarding the bioaccumulation potential of OBM sediments should be drawn from the 1994 data.

TABLE 4.3. Comparison of FDA Action Levels With Contaminant Levels in *M. nasuta* Exposed to OBM Sediments

Contaminant	FDA Action Level (mg/kg wet weight)	Maximum Concentration (mg/kg wet weight) in <i>M. nasuta</i> Tissues
Chlordane	0.3	0.013 U(a)
DDT + DDE	5.0	0.04
Dieldrin + Aldrin	0.3	0.003
Endrin	0.3	0.001 U
Heptachlor + Heptachlor Epoxide	0.3	0.001 U
Toxaphene	5.0	0.016 U
PCBs	2.0	0.0032 U

(a) U Undetected above given concentration.

TABLE 4.4. Summary of Statistically Significant Acute Toxicity and Bioaccumulation for the Older Bay Mud Study

Sediment Treatment	Acute Toxicity	<i>M. nasuta</i> Bioaccumulation				
		PAH	Pesticides	PCB	Metals	Tributyltins
Jan. 1993 OBM COMP	R(a)	---	DDD,DDE,DDT,Dieldrin	---	Cd, Cr, Cu, Hg	TBT, DBT
Jan. 1994 OBM COMP	R(c), N(d)	NA(e)	---	---	NA	---

(a) Rhepoxynius acutely toxic when compared with R-AM.
(b) --- Not statistically different when compared with the references.
(c) Rhepoxynius acutely toxic when compared with R-OS, R-BF, and R-AM.
(d) 10-day *Nephtys caecoides* test acutely toxic when compared with R-OS, R-BF, and R-AM.
(e) NA Not applicable; not analyzed for project.

only. Based on these data, PAHs, pesticides, PCBs, and butyltins were not present in the sediments or were present at low levels and did not bioaccumulate in *M. nasuta* tissues exposed to the OBM COMP relative to all three reference treatments. The metals were not tested in the 1994 bioaccumulation study; therefore, no definitive conclusions can be drawn regarding their potential to bioaccumulate into *M. nasuta* tissues. However, the metals were detected in the sediments at levels similar to those found in at least two of the three reference sediments. Therefore, bioaccumulation of metals in *M. nasuta* tissues exposed to OBM sediments would probably not have been statistically significantly different than the tissues exposed to the reference sediments, particularly R-OS and R-BF.

In summary, the two objectives of this study were to 1) determine whether OBM sediments could be left exposed after YBM sediments were dredged, and 2) determine whether OBM is acceptable for disposal at various disposal sites. OBM is primarily composed of silt and hard-packed clay, has a low percentage of TOC, and contains no detectable concentrations of oil and grease, TPH, pesticides, PCBs, and butyltins, and low concentrations of PAHs, and metals, indicating that OBM does not mix with the overlying YBM sediments. The OBM sediments in Richmond Harbor appear to be an effective barrier to the downward transport of contaminants that are associated with the YBM sediments. The OBM sediments did pass all toxicity testing relative to the ocean site. OBM sediments were acutely toxic to *R. abronius* relative to the in-bay sites. As stated earlier, this toxicity is probably caused by the physical nature of the sediment. The decisions regarding leaving OBM sediment exposed during dredging operations and determining suitable disposal sites for this sediment will lie with the regulatory agencies.

5.0 REFERENCES

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APPENDIX A

SEDIMENT CHEMISTRY AND QUALITY ASSURANCE DATA

QA/QC SUMMARY

PROGRAM	Older Bay Mud Study
LABORATORY:	Soil Technology, Bainbridge Island, Washington
MATRIX:	Sediment
PARAMETER:	Grain Size
METHOD	Grain size was measured for 4 fractions (gravel, sand, silt and clay) using a combination of sieve and pipette techniques following ASTM Methods D-2217 for wet sieving and D-422 modified. Tabular results are presented as fractional percent for each category.
HOLDING TIMES	The holding time of 6 months was met for all grain size analyses.
DETECTION LIMITS	Target detection limits of 1% by weight were met for each sediment sample.
METHOD BLANKS	Not applicable.
MATRIX SPIKES	Not applicable.
REPLICATES	One sample, C-SPB, was analyzed in duplicate. Precision was measured by calculating the relative percent difference (RPD) between the replicate results. The RPDs ranged from 0% to 9% which is within the QA/QC limits of $\pm 20\%$ established for precision. One QC sample was analyzed in triplicate. Precision was measured by calculating the relative standard deviation (RSD) among the replicate results. The RSD's ranged from 0% to 2% which is within the QA/QC limits of $\pm 20\%$ established for precision.
SRMs	Not applicable.

REFERENCES

- ASTM (American Society for Testing and Materials). 1972. *Determination of Soil Constants and Standard Method for Particle-Size analysis of Soils (16 fractions)*. Method D-422, American Society for Testing and Materials, Philadelphia, Pennsylvania.
- ASTM (American Society for Testing and Materials). 1985. *Standard Method for Wet Preparation of Soil Samples for Particle-size Analysis*. Method D-2217, American Society for Testing and Materials, Philadelphia, Pennsylvania.

QA/QC SUMMARY

**PROGRAM
LABORATORY:**

Older Bay Mud Study
Global Geochemistry, Canoga Park, California and
Analytical Resources, Inc., Seattle, Washington

**MATRIX:
PARAMETER:**

Sediment
Total Organic Carbon

METHOD BY GLOBAL

TOC was analyzed using EPA method 9060 (EPA 1986). Samples were dried and ball milled prior to analysis using a carbon analyzer. Global analyzed TOC for the January 1993 and 1994 studies.

METHOD BY ARI

ARI's method follows Plumb 1981, which involves direct combustion at 850°C in a resistance furnace. ARI analyzed TOC for the October 1994 study.

HOLDING TIMES

The holding time of 6 months was met for all TOC analyses.

DETECTION LIMITS

Target detection limits of 0.1% were met for each sample.

METHOD BLANKS

Three method blanks were analyzed with the sediment samples. TOC was detected in the blanks and ranged from 0.0018% to 0.003% which was below the target detection limit.

MATRIX SPIKES

One sample, R-AM, was spiked in duplicate. Matrix spike recoveries were 96% and 107% which is within the QA/QC recovery range of 50% to 150%. Precision was measured by comparing the relative percent difference (RPD) between percent recoveries. The RPD was 10% indicating acceptable precision.

REPLICATES

One sample, OBM COMP, was analyzed in duplicate. Precision was measured by calculating the relative percent difference (RPD) between the replicate results. The RPD was 6%, which is within the $\pm 10\%$ QA/QC limit established for precision.

One sample, R-AM, was analyzed in triplicate. Precision was measured by calculating the relative standard deviation (RSD) among replicate results. The RSD was 13% which is slightly higher than the $\pm 10\%$ QA/QC limit established for precision.

SRMs

Two SRMs, MESS-1 and NBS 2704, were analyzed for TOC. Although MESS-1 is not certified for TOC, in-house values have been 2.6 ± 0.2 . The MESS-1 value reported in this study was within this range. NBS 2074 is certified at 3.35%. The values reported here are within 30% of the certified value.

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.

Plumb, R. H. 1981. *Procedures for Handling and Chemical Analysis of Sediment and Water Samples*. U.S. Army Engineer Waterways Experiment Station, Vicksburg, Mississippi.

QA/QC SUMMARY

PROGRAM	Older Bay Mud Study
LABORATORY:	Battelle Marine Sciences Laboratory, Sequim, Washington
MATRIX:	Sediment
PARAMETER:	Total Volatile Solids
METHOD	Total volatile solids were analyzed according to EPA Method 160.4 (EPA 1979) by heating dried solids to 550°C for 1 hour and measuring the weight percentage lost during this process.
HOLDING TIMES	A holding time is not specified for TVS analyses.
DETECTION LIMITS	Target detection limits of 0.1% by weight were met for each sediment sample.
METHOD BLANKS	Two method blanks were analyzed with the sediment samples. The blank level was undetected at 1.0%.
MATRIX SPIKES	Not applicable.
REPLICATES	Two samples, C-SB and R-AM, were analyzed in duplicate. Precision was measured by calculating the relative percent difference (RPD) between the replicate results. The RPD ranged from 6% to 18%, which is within the $\pm 30\%$ QA/QC Limit established for precision.
SRMs	Not applicable.
REFERENCES	EPA (U.S. Environmental Protection Agency). 1979. <i>Methods for Chemical Analysis of Water and Wastes</i> . EPA-600 4-79-020 Method 160.4, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

QA/QC SUMMARY

PROGRAM
LABORATORY:
MATRIX:
PARAMETER:

Older Bay Mud Study
Analytical Resources, Inc., Seattle, Washington
Sediment
Oil and Grease

METHOD

Oil and grease was analyzed using EPA Method 413.2. A 20-g sample was dried and extracted with Freon. The samples were then analyzed using infrared spectrophotometry.

HOLDING TIMES

The 30-day holding time specified in the QA/QC Plan was met for all sediment samples analyzed for the January 1993 and 1994 studies. The samples analyzed in October 1994 had to be reanalyzed to obtain acceptable method blank results. This reanalysis occurred within 60 days of sample receipt at the laboratory.

DETECTION LIMITS

Target detection limits of 20 mg/kg were met for each sediment sample.

METHOD BLANKS

Two method blanks were analyzed with the sediment samples. The blank levels were undetected at up to 13 mg/kg which is below the target detection limit of 20 mg/kg.

MATRIX SPIKES

Two samples, C-WB and R-AM, were spiked with oil and grease. Matrix spike recoveries ranged from 99% to 121% which is within the QA/QC recovery range of 50% to 150%. Precision was measured by comparing the relative percent difference (RPD) between matrix spike and matrix spike duplicate recoveries. The RPD was 6% indicating acceptable precision.

REPLICATES

One sample, C-WB, was analyzed in duplicate. Precision was measured by calculating the relative percent difference (RPD) between the replicate results. The RPD was 26%, which is within the $\pm 30\%$ QA/QC limit established for precision.

One sample, R-AM, was analyzed in triplicate. Precision was measured by calculating the relative standard deviation among triplicate results. All of the triplicate values were undetected; therefore, an RSD could not be calculated.

SRMs

Not applicable.

REFERENCES

EPA (U.S. Environmental Protection Agency). 1979. *Methods for Chemical Analysis of Water and Wastes*. EPA-600 4-79-020 Method 413.2, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

QA/QC SUMMARY

PROGRAM
LABORATORY:
MATRIX:
PARAMETER:

Older Bay Mud Study
Analytical Resources, Inc., Seattle, Washington
Sediment
Total Petroleum Hydrocarbons

METHOD

Total petroleum hydrocarbons comprise the nonpolar mineral fraction of total oil and grease that is not removed by silica gel absorption. An aliquot of sample material was dried with anhydrous sodium sulfate and extracted with Freon. Silica gel was added to the extract to remove the more polar animal and vegetable based oils. The extract was shaken, allowed to settle, removed, and scanned from 4000 to 600 cm^{-1} by infrared spectrophotometry (IR). The peak height measured at 2930 cm^{-1} was used to quantify the concentration of hydrocarbons in the sample.

HOLDING TIMES

The 30-day holding time specified in the QA/QC Plan was met for all sediment samples analyzed for the January 1993 and 1994 studies. The samples analyzed in October 1994 had to be reanalyzed to obtain acceptable method blank results. This reanalysis occurred within 60 days of sample receipt at the laboratory.

DETECTION LIMITS

Target detection limits of 20 mg/kg were met for each sediment sample.

METHOD BLANKS

One procedural blank was processed with each batch of samples. No hydrocarbons were detected in the method blanks at concentrations above the detection limit.

MATRIX SPIKES

One matrix spike (MS) was analyzed with each batch of samples. The percent recovery was 95% which is within the QA/QC recovery range of 50% to 150%.

REPLICATES

One sample, R-AM, was analyzed in triplicate. Precision was measured by calculating the relative standard deviation among triplicate results. All of the triplicate values were undetected; therefore, an RSD could not be calculated.

SRMs

Not applicable.

REFERENCES

EPA (U.S. Environmental Protection Agency). 1979. *Methods for Chemical Analysis of Water and Wastes*. EPA-600 4-79-020 Method 413.2, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

QA/QC SUMMARY

**PROGRAM
LABORATORY:**

Older Bay Mud Study
Analytical Resources, Inc., Seattle, Washington, and
Battelle Marine Sciences Laboratory, Sequim, Washington
Sediment

**MATRIX:
PARAMETER:**

Polynuclear Aromatic Hydrocarbons (PAHs)

METHOD

PAHs were analyzed using EPA SW 846 Method 8270 for samples analyzed at ARI and were analyzed using the National Ocean and Atmospheric Administration (NOAA) Status and Trends method (Krahn et al. 1988) for samples analyzed at the MSL.

HOLDING TIMES

In the January 1993 study, sediment samples were received at ARI, extracted, and analyzed by GC/MS within 30 days which is within EPA's recommended holding time of 40 days for organics (EPA 1986).

In the January 1994 and October 1994 studies, sediment samples were stored at Battelle at -20°C until ready for extraction and were analyzed within 60 days (January) and 30 days (October) of receipt of the samples.

DETECTION LIMITS

Target detection limits of 20 µg/kg were met for each sediment sample.

METHOD BLANKS

Four method blanks were analyzed with the sediment samples. Six PAHs (naphthalene, phenanthrene, fluoranthene, pyrene, benzo(a)anthracene, and benzo(a)pyrene) were detected in the blanks at concentrations of ≤11.5 µg/kg. Some sediment concentrations were flagged with a "B" flag to indicate that a specific analyte was detected in the sample at concentrations less than five times the value in the associated method blank.

MATRIX SPIKES

Three samples, C-WB, QC sample, and OBM Comp, were spiked with PAHs. Matrix spike recoveries ranged from 79% to 681%. Four percent recoveries were outside the QA/QC recovery range of 40% to 120%. Precision was measured by calculating the relative percent difference (RPD) between matrix spike and matrix spike duplicate recoveries. The RPDs were 0% which indicates excellent precision.

REPLICATES

One sample, OBM COMP, was analyzed in duplicate. Precision was measured by calculating the RPD between the replicate results. All detected values were <10 times the MDL; therefore, the precision criterion does not apply.

Three samples, QC sample, R-AM, and QC sample, were analyzed in triplicate. Precision was measured by calculating the RSDs among the replicate results. The RSDs ranged from 2% to 81%. Fourteen of the calculated RSD's were outside the QA/QC limit of <30% established for precision.

QA/QC SUMMARY (contd)

SRMs

Four SRMs, SQ-1 and 1941a, were analyzed with the sediment samples. One LPAH value and ten HPAH values were outside of the QA/QC goal of $\leq 30\%$, indicating acceptable accuracy of the method.

SURROGATES

For the January 1993 study, prior to extraction, two compounds, diphenyl-d10 and p-Terphenyl-d14 were added to the sediment samples to assess the efficiency of the method. Recoveries ranged from 50% to 83% which were within the QA/QC range of 40% to 120%.

In the January and October 1994 studies, five isotopically labelled compounds were added prior to extraction to assess the efficiency of the method. These were d8-Naphthalene, d8-Acenaphthene, d12-Chrysene, d10-Pyrene (January), d10-Perylene (October), and d14-Dibenzo(a,h)anthracene. Recoveries of all surrogates were within the quality control limits of 40% to 120%.

REFERENCES

EPA (U.S. Environmental Protection Agency). 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SWP-846 Method 8270. EPA-955-001-00000, U.S. Government Printing Office, Washington D.C.

Krahn, M.M., C.A. Wigren, R.W. Pearch, L.K. Moore, R.G. Bogar, W.D. MacLeod, Jr., S.L. Chan, and D.W. Brown. 1988. "A Rapid High-Performance Liquid Chromatographic Method for Isolating Organic Contaminants from Tissue and Sediment Extracts." NOAA Technical Memorandum NMFS F/NWC-153, Silver Spring, Maryland.

QA/QC SUMMARY

**PROGRAM
LABORATORY:**

Older Bay Mud Study
Analytical Resources, Inc., Seattle, Washington, and
Battelle Marine Sciences Laboratory, Sequim, Washington

**MATRIX:
PARAMETER:**

Sediment
Pesticide

METHOD

For the January 1993 study, pesticides were analyzed by ARI using EPA SW 846 Method 8080.

For the January and October 1994 studies, pesticides were analyzed by Battelle Marine Sciences Laboratory. Sediment samples were extracted with methylene chloride using a roller under ambient conditions following a procedure based on EPA method 3510 and 8080 (EPA 1986) and NOAA status and trends methodology (Krahn et al. 1988). Samples were then cleaned using silica/alumina (5% deactivated) chromatography followed by HPLC cleanup (Krahn et al. 1988). Modifications in the clean-up method were required in the January study to adequately recover the phthalate ester compounds required by the project. The Si column clean-up step was either eliminated or the extracts were eluted through silica and alumina, but twice the usual amount of methylene chloride was used. 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 ID).

HOLDING TIMES

In the January 1993 study, sediment samples were received at ARI, extracted, and analyzed by GC/MS within 30 days which is within EPA's recommended holding time of 40 days for organics (EPA 1986).

In the January 1994 and October 1994 studies, sediment samples were stored at Battelle at -20°C until ready for extraction and were analyzed within 60 days (January) and 30 days (October) of receipt of the samples.

DETECTION LIMITS

Target detection limits of 2 µg/kg were set for 19 pesticides. Ten of the pesticides met the detection limit requirements. The remaining nine pesticides had detection limits ranging from undetected at 0.75 µg/kg to 11 µg/kg. Toxaphene detection limits ranged from undetected at 75 µg/kg to 160 µg/kg.

METHOD BLANKS

Six method blanks were analyzed with the sediment samples. For all of the blanks except Blank 3, the levels of pesticides were below the detection limit for every pesticide except toxaphene. The levels of pesticides in Blank 3 were undetected at a higher level due to the fact that only one-half the sample weight was used to calculate the blank concentrations. All of the sediment samples were undetected, no corrective action was taken.

QA/QC SUMMARY (contd)

MATRIX SPIKES

Four samples, C-WB, QC sample, QC sample, and OBM Comp, were spiked with pesticides. Matrix spike recoveries ranged from 74% to 135%. All but one of the percent recoveries exceeded the QA/QC recovery range of 40% to 120%. Precision was measured by calculating the relative percent difference (RPD) between matrix spike and matrix spike duplicate recoveries. The RPDs ranged from 0% to 3% which indicates excellent precision.

REPLICATES

Three samples, OBM COMP, C-SPB, and QC sample, were analyzed in duplicate. Precision was measured by calculating the RPD between the replicate results. The RPD's ranged from 0% to 30%, which is within the QA/QC limit of $\leq 30\%$ established for precision.

Three samples, QC sample, R-AM, and QC sample, were analyzed in triplicate. Precision was measured by calculating the RSD between the replicates. The RSD's ranged from 4% to 26%, which is within the QA/QC limit of $\leq 30\%$ established for precision.

SRMs

Not applicable.

SURROGATES

For the January 1993 study, prior to extraction, two compounds decachlorobiphenyl and tetrachlorometacylene were added to the sediment samples to assess the efficiency of the method. Recoveries ranged from 10% to 114%, with only four surrogate values exceeding the QA/QC range of 40% to 120%.

REFERENCES

EPA (U.S. Environmental Protection Agency). 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SWP-846 Method 8080. EPA-955-001-00000, U.S. Government Printing Office, Washington D.C.

Krahn, M.M., C.A. Wigren, R.W. Pearch, L.K. Moore, R.G. Bogar, W.D. MacLeod, Jr., S.L. Chan, and D.W. Brown. 1988. "A Rapid High-Performance Liquid Chromatographic Method for Isolating Organic Contaminants from Tissue and Sediment Extracts." NOAA Technical Memorandum NMFS F/NWC-153, Silver Spring, Maryland.

QA/QC SUMMARY

**PROGRAM
LABORATORY:**

Older Bay Mud Study
Analytical Resources, Inc., Seattle, Washington, and
Battelle Marine Sciences Laboratory, Sequim, Washington
Sediment

**MATRIX:
PARAMETER:**

Polychlorinated Biphenyls (PCBs)

METHOD

For the January 1993 study, PCBs were analyzed using EPA SW 846 Method 8080.

For the January and October 1994 studies, PCBs were analyzed by Battelle Marine Sciences Laboratory. Sediment samples were extracted with methylene chloride using a roller under ambient conditions following a procedure based on EPA method 3510 and 8080 (EPA 1986) and NOAA status and trends method (Krahn et al. 1988). Samples were then cleaned using silica/alumina (5% deactivated) chromatography followed by HPLC cleanup (Krahn et al. 1988). Modifications in the clean-up method were required in the January study to adequately recover the phthalate ester compounds required by the project. The Si column clean-up step was either eliminated or the extracts were eluted through silica and alumina, but twice the usual amount of methylene chloride was used. 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 ID).

HOLDING TIMES

In the January 1993 study, sediment samples were received at ARI, extracted, and analyzed by GC/MS within 30 days which is within EPA's recommended holding time of 40 days for organics (EPA 1986).

In the January 1994 and October 1994 studies, sediment samples were stored at the MSL at -20°C until ready for extraction and were analyzed within 60 days (January) and 30 days (October) of receipt of the samples.

DETECTION LIMITS

Target detection limits of 20 µg/kg were met for OBM COMP and the reference samples. Some of the other samples had higher detection limits due to limited sample volume.

METHOD BLANKS

Six method blanks were analyzed with the sediment samples. The levels of PCBs in all blanks except Blank 3 were below the detection limits. The levels of PCBs in Blank 3 were undetected at a higher level because only one-half the sample weight was used to calculate the blank concentrations. All of the sediment samples were undetected, no corrective action was taken.

MATRIX SPIKES

Three samples QC sample, QC sample, and OBM COMP were spiked with Aroclor 1254. Matrix spike recoveries ranged from 98% to 110% which is within the QA/QC limits of 50% to 150%.

QA/QC SUMMARY (contd)

REPLICATES

Three samples, OBM COMP, C-SPB, and QC sample, were analyzed in duplicate. Precision was measured by calculating the relative percent difference (RPD) between the replicate results. Calculations could not be performed for most Aroclors because all replicate values were undetected. The RPD for Aroclor 1254 was 30% which is within the QA/QC limit of $\leq 30\%$ established for precision.

Three samples, QC sample, R-AM, and QC sample, were analyzed in triplicate. Precision was measured by calculating the relative standard deviation (RSD) among the replicate results. Only the RSD for Aroclor 1254 could be calculated at 10% which is within the QA/QC limit of $\leq 30\%$ established for precision.

SRMs

Not applicable.

SURROGATES

For the January 1993 study, prior to extraction, two compounds, decachlorobiphenyl and tetrachlorometacylene were added to the sediment samples to assess the efficiency of the method. Recoveries ranged from 10% to 114%, with only four surrogate values exceeding the QA/QC range of 40% to 120%.

For the January and October 1994 studies, 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). Recoveries of these compounds were within the QC guidelines of 40% to 120% for all samples analyzed.

REFERENCES

EPA (U.S. Environmental Protection Agency). 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SWP-846 Method 8080. EPA-955-001-00000, U.S. Government Printing Office, Washington D.C.

Krahn, M.M., C.A. Wigren, R.W. Pearch, L.K. Moore, R.G. Bogar, W.D. MacLeod, Jr., S.L. Chan, and D.W. Brown. 1988. "A Rapid High-Performance Liquid Chromatographic Method for Isolating Organic Contaminants from Tissue and Sediment Extracts." NOAA Technical Memorandum NMFS F/NWC-153, Silver Spring, Maryland.

QA/QC SUMMARY

PROGRAM
LABORATORY:
MATRIX:
PARAMETER:

Older Bay Mud Study
Battelle Marine Sciences Laboratory, Sequim, Washington
Sediment
Metals

METHOD

A total of 10 metals was analyzed for silver (Ag), arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), nickel (Ni), lead (Pb), selenium (Se) and zinc (Zn). Six metals (As, Cr, Cu, Ni, Pb, and Zn) were analyzed by energy diffusive x-ray fluorescence (XRF) following the method of a PNL SOP. Three metals (Ag, Cd, and Se) were analyzed using Zeeman Graphite Furnace Atomic Absorption (GFAA) spectrometry following the EPA Method 200.9. The metal Hg was analyzed using cold-vapor atomic absorption spectroscopy (CVAA) according to the method of Bloom and Crecelius (1983).

HOLDING TIMES

Samples were received in good condition and were placed into Battelle's log-in system. Samples were frozen to -80°C and subsequently freeze-dried within approximately 7 days of sample receipt. Samples were all analyzed within 180 days of collection. The metal Hg was analyzed within the 28 day holding time.

The samples from the January and October 1994 studies were frozen to -80°C and subsequently freeze-dried within approximately 14 days of sample receipt. Samples were analyzed within 180 days of sample receipt with the exception of Hg, which was analyzed within 30 days of receipt.

DETECTION LIMITS

Target detection limits were met for all metals with the exception of Hg. The achieved detection limits for Hg were 0.02 mg/kg, slightly above the target of 0.01 mg/kg. Mercury was detected in all samples above the achieved detection limit.

METHOD BLANKS

Two method blanks were analyzed for Ag, Cd, Se and Hg with each batch of sediment samples. Silver was detected in Blanks 1 and 2 at the target detection limit and Cd was detected in Blank 2 at 0.032 mg/kg. All metals data are blank corrected; therefore, no data was flagged. Method blanks are not analyzed by XRF.

MATRIX SPIKES

Two samples, C-SPB and QC sample, were spiked with Ag, Cd, Hg and Se. Matrix spike recoveries ranged from 77% to 117%, which is within the QA/QC limits of 75% to 125%. Samples for XRF are analyzed whole and cannot be spiked.

QA/QC SUMMARY METALS (contd)

REPLICATES

One sample, C-SPB, was digested in duplicate and analyzed. Precision was measured by calculating the relative percent difference (RPD) between the replicate results. The RPDs ranged from 0% to 23%. All metals were within the QA/QC limits of $\pm 20\%$ with the exception of Pb which had an RPD of 23%. Samples for XRF are not processed, limiting the chance for contamination or loss due to handling. The exceedance for Pb is most likely due to nonhomogeneity of the sample.

One sample, QC sample, was analyzed in triplicate. Precision was measured by calculating the relative standard deviation (RSD) among the replicate results. The RSD's ranged from 0% to 43% with one RSD outside the QA/QC limit of $\leq 20\%$ established for precision.

SRMs

One SRM, 1646 (an estuarine sediment obtained from the National Institute for Standards and Technology, NIST), was analyzed for all metals and were within $\pm 30\%$ of the certified value, indicating acceptable accuracy.

One SRM, BEST-1 (1646), was analyzed for Hg and was within the $\pm 30\%$ of the certified value, indicating acceptable accuracy.

REFERENCES

Bloom, N.S., and E.A. Creclius. 1983. "Determination of Mercury in Seawater at Sub-Nanogram per Liter Levels." *Marine Chemistry* 21:337-390.

EPA (U.S. Environmental Protection Agency). 1991. *Methods for the Determination of Metals in Environmental Samples*. EPA-600/4-91-010 Methods 200.8, 200.9, Environmental Services Division, Monitoring Management Branch, Cincinnati, Ohio.

QA/QC SUMMARY

PROGRAM	Older Bay Mud Study
LABORATORY:	Battelle Marine Sciences Laboratory, Sequim, Washington
MATRIX:	Sediment
PARAMETER:	Butyltins
METHOD	Butyltin analyses were performed following the method of Unger et al. (1986).
HOLDING TIMES	Samples were placed into Battelle log-in system and stored at approximately -20° C until extraction. Samples were extracted and extracts were analyzed by GC/FPD within EPAs recommended holding time of 40 days for organic extracts (EPA 1986).
DETECTION LIMITS	Target detection limits of 10 µg/kg dry weight were met for all butyltin compounds. Actual detection limits ranged from 5 to 8 µg/kg for non-detected analytes. These detection limits represent quantitation limits (LOQs) defined as 10 times the standard deviation of results from 7 replicate low level matrix spikes. Note that some values were flagged with a "J" flag indicating the levels present were below the LOQ but above the MDL. The MDLs are defined as 3 times the standard deviation of 7 replicate spike results
METHOD BLANKS	Four method blanks were analyzed with the sediment samples. Tributyltin was detected in Blank 4 at 0.55 µg/kg. Dibutyltin was detected in Blank 1 just below the LOQ at 4.3 µg/kg and at 0.65 µg/kg in Blank 3. These values are less than the target detection limit of 10 µg/kg; therefore, no data was flagged and no other corrective actions were taken.
SURROGATES	One compound, Triphenyltin chloride, is added prior to extraction to assess the efficiency of the method. This compound also is used as an internal standard as all data is corrected for the recovery of the compound. Recoveries ranged from 78% to 113%, within the QA/QC limits of 40% to 120%.
MATRIX SPIKES	One sample, C-WB, was spiked in duplicate with mono-, di- and tributyltin. Matrix spike recoveries ranged from 83% to 94% for the di- and tributyltins, which were within the QA/QC limits of 40% to 120%. Relative Percent Differences between MS and MSD recoveries ranged from 2% to 7%, within the ±30% QA/QC limit for all butyltins, indicating acceptable precision. Three samples, QC sample, R-AM, and QC sample, were spiked with butyltins. Matrix spike recoveries ranged from 53% to 128% with one recovery outside the QA/QC limits of 40% to 120%.

QA/QC SUMMARY BUTYLTINS (contd)

REPLICATES

One sample, C-SPB, was extracted in duplicate. Precision was measured by calculating the RPDs between the replicate results. The RPD for tributyltin was 0%, indicating acceptable precision. The RPDs could not be calculated due to undetected values in the replicates.

Three samples, QC sample, QC sample, and R-OS, were analyzed in triplicate. Precision was measured by calculating the RSDs among the replicate results. The RSDs ranged from 10% to 12% which are within the QA/QC acceptable limit of $\leq 20\%$ established for precision.

SRMs

PACS-1, a marine sediment obtained from the National Research Council of Canada (NRCC) was analyzed with each batch of sediment samples. The TBT value for Batch 1 and the DBT value for Batch 3 were below $\pm 30\%$ of the certified value. Historically, results obtained for dibutyltin and tributyltin from analysis of PACS-1 have been closer to the lower certified range. This has been corroborated by other laboratories.

REFERENCES

Unger, M.A., W.G. MacIntyre, J. Greaves, and R.J. Huggett. 1986. "GC Determination of Butyltins in Natural Waters by Flame Photometric Detection of Hexyl Derivatives with Mass Spectrometric Confirmation." *Chemosphere*. 15(4):461-470.

EPA (U.S. Environmental Protection Agency). 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SW-846. EPA-955-001-00000, U.S. Government Printing Office, Washington D.C.

TABLE A.1. Summary of Sediment Grain Size Including Quality Control Data, Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Batch</u>	<u>Total Percent (Dry Weight)</u>			
		<u>Gravel</u> <u>>2000 μm</u>	<u>Sand</u> <u>62.5-</u> <u>2000 μm</u>	<u>Silt</u> <u>3.9-</u> <u>62.5 μm</u>	<u>Clay</u> <u><3.9 μm</u>
<u>January 1993</u>					
OBM COMP	1	0	15	47	38
C-SB	1	0	33	36	31
C-WB	1	0	99	0	1
C-SPB, Replicate 1	1	0	12	46	42
C-SPB, Replicate 2	1	0	11	48	41
<u>October 1994</u>					
OBM COMP	2	0	18	56	26
R-OS	2	0	26	53	21
R-BF	2	0	1	39	60
R-AM	2	0	99	0	1
<u>Quality Control Data</u>					
<u>Analytical Replicates</u>					
<u>January 1993</u>					
C-SPB, Replicate 1	1	0	12	46	42
C-SPB, Replicate 2	1	0	11	48	41
RPD		0%	9%	4%	2%
I-Stat		0.00	0.04	0.02	0.01
<u>October 1994</u>					
QC Sample, Replicate 1	2	0	4	49	47
QC Sample, Replicate 2	2	0	4	48	48
QC Sample, Replicate 3	2	0	4	47	49
RSD		NA ^(a)	0%	2%	2%

(a) NA Not applicable.

TABLE A.2. Sediment Total Organic Carbon (TOC), Total Percent Solids, and Total Volatile Solids (TVS), Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Batch</u>	<u>Percent dry weight</u>		
		<u>Total Solids</u>	<u>TOC</u>	<u>TVS</u>
Target DL ^(a)		1.0	0.1	0.1
<u>January 1993</u>				
OBM COMP, Replicate 1	1	76.38	0.17	2.29
OBM COMP, Replicate 2	1	NA ^(b)	0.18	NA
C-SB, Replicate 1	1	31.58	2.01	2.21
C-SB, Replicate 2	1	31.57	NA	2.09
C-WB	1	77.97	0.06	0.496
C-SPB	1	35.99	1.23	2.08
<u>October 1994</u>				
OBM COMP	2	74.1	0.16	1.9
OBM-0.4% TOC ^(c)	2	NA	0.24	NA
OBM-0.6% TOC ^(c)	2	NA	0.27	NA
OBM-1.0% TOC ^(c)	2	NA	0.39	NA
OBM-1.4% TOC ^(c)	2	NA	0.44	NA
R-OS	2	53.0	1.0	2.4
R-BF	2	36.5	1.4	2.3
R-AM, Replicate 1	2	82.1	0.096	0.5
R-AM, Replicate 2	2	82.6	0.096	0.6

(a) DL Detection limit.

(b) NA Not applicable.

(c) Prior to analysis, TOC was estimated in the OBM sediments; the actual values obtained from analysis are shown in the TOC column.

TABLE A.3. Quality Control Data for Sediment Total Organic Carbon (TOC), Total Percent Solids, and Total Volatile Solids (TVS), Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Batch</u>	<u>Percent dry weight</u>		
		<u>Total Solids</u>	<u>TOC</u>	<u>TVS</u>
<u>Method Blank</u>				
Blank-1	1	<1.0	0.003	<1.0
Blank-2	1	NA ^(a)	0.003	NA
Blank	2	<1.0	0.0018	<1.0
<u>Matrix Spike</u>				
R-AM	2	NA	0.0962	NA
R-AM, MS		NA	0.681	NA
Concentration Recovered		NA	0.585	NA
Amount Spiked		NS ^(b)	0.547	NS
Percent Recovery		NA	107%	NA
R-AM	2	NA	0.0962	NA
R-AM, MSD		NA	0.782	NA
Concentration Recovered		NA	0.686	NA
Amount Spiked		NS	0.711	NS
Percent Recovery		NA	96%	NA
RPD		NA	10%	NA
I-Stat		NA	0.05	NA
<u>Standard Reference Material</u>				
Consensus value MESS-1		NA	2.60	NA
		NA	±0.20	NA
MESS-1	1	NA	2.55 ^(c)	NA
MESS-1	2	NA	2.80 ^(c)	NA
Certified value SRM NBS 2704		NA	3.35	NA
NBS 2704	2	NA	2.87	NA
NBS 2704	2	NA	3.37	NA

TABLE A.3. (contd)

<u>Sediment Treatment</u>	<u>Batch</u>	<u>Percent dry weight</u>		
		<u>Total Solids</u>	<u>TOC</u>	<u>TVS</u>
<u>Analytical Replicates</u>				
OBM COMP, Replicate 1	1	NA	0.17	NA
OBM COMP, Replicate 2	1	NA	0.18	NA
RPD		NA	6%	NA
I-Stat		NA	0.03	NA
C-SB, Replicate 1	1	31.58	NA	2.21
C-SB, Replicate 2	1	31.57	NA	2.09
RPD		0%	NA	6%
I-Stat		0.00	NA	0.03
R-AM, Replicate 1	2	82.1	0.096	0.5
R-AM, Replicate 2	2	82.6	0.096	0.6
R-AM, Replicate 3	2	NA	0.12	NA
RSD/RPD		0.6%	13%	18%

(a) NA Not applicable.

(b) NS Not spiked.

(c) MESS-1 not certified for TOC, but frequent analysis at MSL indicates a value of 2.6%.

TABLE A.4. Sediment Oil and Grease and Total Petroleum Hydrocarbons (TPH)
Results, Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Batch</u>	<u>mg/kg dry weight</u>	
		<u>Oil and Grease</u>	<u>TPH</u>
Target DL ^(a)		20	20
<u>January 1993</u>			
OBM COMP	1	501	NA ^(b)
C-SB	1	1380	NA
C-WB, Replicate 1	1	284	NA
C-WB, Replicate 2	1	370	NA
C-SPB	1	772	NA
<u>October 1994</u>			
OBM COMP	2	11 U ^(c)	11 U
R-OS	2	100	77
R-BF	2	130	100
R-AM	2	10 U	10 U

(a) Detection Limit

(b) NA Not applicable.

(c) U Undetected at or above detection limit.

TABLE A.5. Quality Control Data for Sediment Oil and Grease and Total Petroleum Hydrocarbons (TPH) Analyses, Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Batch</u>	<u>mg/kg dry weight</u>	
		<u>Oil and Grease</u>	<u>TPH</u>
<u>Method Blank</u>			
Blank	1	10	NA ^(a)
Blank	2	13	11
<u>Matrix Spike</u>			
C-WB, Replicate 1	1	284	NA
C-WB, Replicate 1 MS		8930	NA
Concentration Recovered		8646	NA
Amount Spiked		7570	NS ^(b)
Percent Recovery		114%	NA
C-WB, Replicate 1	1	284	NA
C-WB, Replicate 1 MSD		6200	NA
Concentration Recovered		5916	NA
Amount Spiked		4900	NS
Percent Recovery		121%	NA
RPD		6%	NA
I-Stat		0.03	NA
R-AM	2	11 U	11 U
R-AM, MS		12700	10600
Concentration Recovered		12700	10600
Amount Spiked		12800	11100
Percent Recovery		99%	95%
<u>Analytical Replicates</u>			
C-WB, Replicate 1	1	284	NA
C-WB, Replicate 2	1	370	NA
RPD		26%	NA
I-Stat		0.13	NA
R-AM, Replicate 1	2	11 U	11 U
R-AM, Replicate 2	2	11 U	11 U
R-AM, Replicate 3	2	10 U	10 U
RSD		NA	NA

(a) NA Not applicable.

(b) NS Not spiked.

(c) U Undetected at or above detection limit.

TABLE A.6. Sediment Total Polynuclear Aromatic Hydrocarbons (PAHs),
Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Batch</u>	<u>μg/kg dry weight</u>		
		<u>Total Low Molecular Weight PAHs</u>	<u>Total High Molecular Weight PAHs</u>	<u>Total PAHs</u>
<u>January 1993</u>				
OBM COMP, Replicate 1	1	0	24	24
OBM COMP, Replicate 2	1	0	18	18
C-SB	1	54	216	270
C-WB	1	0	0	0
C-SPB	1	188	1618	1806
<u>January 1994</u>				
OBM COMP	2	8	4	12
R-OS	3	48	68	116
R-BF	3	228	1527	1755
R-AM, Replicate 1	3	1507	3229	4736
R-AM, Replicate 2	3	773	1772	2545
R-AM, Replicate 3	3	1251	2277	3528
<u>October 1994</u>				
OBM COMP	4	29	28	57
R-OS	4	65	124	189
R-BF	4	355	2116	2471
R-AM	4	538	1055	1593

TABLE A.7. Sediment Low Molecular Weight Polynuclear Aromatic Hydrocarbons (LPAHs), Older Bay Mud Study

Sediment Treatment	Batch	LPAHs ($\mu\text{g/kg}$ dry weight)					
		Napthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
Target DL ^(a)		20	20	20	20	20	20
<u>January 1993</u>							
OBM COMP, Replicate 1	1	13 U ^(b)	13 U	13 U	13 U	13 U	13 U
OBM COMP, Replicate 2	1	13 U	13 U	13 U	13 U	13 U	13 U
C-SB	1	3.5 M ^(c)	20 U	20 U	4.3 M	39	7.5 J ^(d)
C-WB	1	13 U	13 U	13 U	13 U	13 U	13 U
C-SPB	1	11 J	10 J	6.9 J	11 J	110	40
<u>January 1994</u>							
OBM COMP	2	3.94 B ^(e)	1.47 U	1.62 ^(f)	1.08 U	2.11	1.77 U
R-OS	3	12.3	2.37 ^(f) B	0.98 U	5.67	18.9 B	8.90
R-BF	3	40.1	15.3	12.2	13.4	112	34.7
R-AM, Replicate 1	3	46.0	63.6	32.4	92.2	925	348
R-AM, Replicate 2	3	29.6	32.6	37.8	44.3	456	173
R-AM, Replicate 3	3	38.1	56.6	20.5 ^(f)	94.0	783	259
<u>October 1994</u>							
OBM COMP	4	9.29 B	3.27 ^(f)	4.87	3.59 U	11.4 B	5.15 U
R-OS	4	16.5 B	2.97 U	6.80	6.18	27.9 B	7.91 ^(f)
R-BF	4	55.0	19.8 ^(f)	14.7	18.7	189	57.5
R-AM	4	28.8 B	24.0	9.39	30.2	296	150

- (a) DL Detection limit.
(b) U Undetected at or above detection limit.
(c) M Indicates an estimated value of analyte found and confirmed by analyst, but with low spectral match parameters.
(d) J Analyte detected below method detection limit (MDL), but above instrument detection limit (IDL).
(e) B Analyte detected in sample is less than five times the value in associated method blank.
(f) Ratio of confirmation ion between the primary and secondary column is outside of the theoretical ratio of 20% established for EPA-CLP programs.

TABLE A.8. Sediment High Molecular Weight Polynuclear Aromatic Hydrocarbons (HPAHs),
Older Bay Mud Study

Sediment Treatment	Batch	HPAH ($\mu\text{g/kg}$ dry weight)									
		Fluor- anthene	Pyrene	Benzo[a] anthra- cene	Chrysene	Benzo[b] fluor- anthene	Benzo[k] fluor- anthene	Benzo[a] pyrene	Indeno [1,2,3- c,d] pyrene	Dibenzo- [a,h] anthra- cene	Benzo- [g,h,i] perylene
Target DL ^(a)		20	20	20	20	20	20	20	20	20	20
January 1993											
OBM COMP, Replicate 1	1	13 U ^(b)	3.3 J ^(c)	2.5 M ^(d)	2.6 J	6.3 J	6.3 J	3.4 J	13 U	13 U	13 U
OBM COMP, Replicate 2	1	13 U	2.6 J	1.8 M	1.7 J	4.9 J	4.9 J	2.3 J	13 U	13 U	13 U
C-SB	1	34	30	14 J	25	21	21	16 J	22	15 J	18 J
C-WB	1	13 U	13 U	13 U	13 U	13 U	13 U	13 U	13 U	13 U	13 U
C-SPB	1	220	230	96	130	220	220	180	190	32	100
January 1994											
OBM COMP	2	2.50 ^(e) B ^(f)	1.52 U	1.37 ^(e) B	1.39 U	0.64 U	1.40 U	1.52 U	2.97 U	4.28 U	4.65 U
R-OS	3	11.3 B	7.39	6.88	4.55	9.30	3.41	2.58	10.1	6.02 U	12.2
R-BF	3	199	241	94.7	103	200	66.7	175	210	23.7	214
R-AM, Replicate 1	3	616	751	386	382	290	105	308	182	44.3	165
R-AM, Replicate 2	3	322	395	194	190	167	59.8	185	121	23.5	115
R-AM, Replicate 3	3	452	545	261	255	200	71.3	215	129	30.4	118
October 1994											
OBM COMP	4	8.64 ^(e) B	11.2 B	4.61 ^(e)	0.78 U	1.49 U	2.52 U	3.49 ^(e) B	0.90 U	1.14 U	0.82 U
R-OS	4	22.7 B	27.1 B	9.85	8.10	18.7	3.72 U	8.73 B	10.9	4.50 ^(e)	13.5
R-BF	4	287	337	114	147	280	110	250	279	29.8	282
R-AM	4	199	238	113	133	96.5	42.7	105	60.8	14.0	53.0

- (a) DL Detection limit.
 (b) U Undetected at or above detection limit.
 (c) J Analyte detected below method detection limit (MDL), but above instrument detection limit.
 (d) M Indicates an estimated value of analyte found and confirmed by analyst, but with low spectral match parameters.
 (e) Ratio of confirmation ion between the primary and secondary column is outside of the theoretical ratio of 20% established for EPA-CLP programs.
 (f) B Analyte detected in sample is less than five times the value in associated method blank.

TABLE A.9. Quality Control Data for Sediment Low Molecular Weight Polynuclear Aromatic Hydrocarbons (LPAHs), Dry Weight, Older Bay Mud Study

Sediment Treatment	Batch	LPAHs (µg/kg dry weight)					
		Naptha- lene	Acenaph- thylene	Acenaph- thene	Fluorene	Phenan- threne	Anthra- cene
Method Blank							
Blank	1	10 U ^(a)	10 U	10 U	10 U	10 U	10 U
Blank	2	2.70	1.92 U	0.92 U	1.41 U	1.83 U	2.33 U
Blank	3	5.78	2.49 ^(b)	0.93 U	1.43 U	6.32 ^(b)	2.36 U
Blank	4	8.11 ^(b)	2.16 U	1.94 U	3.86 U	11.3	5.54 U
Matrix Spike							
C-WB	1	NA ^(c)	NA	13 U	NA	NA	NA
C-WB MS		NA	NA	309	NA	NA	NA
Concentration Recovered		NA	NA	309	NA	NA	NA
Amount Spiked		NS ^(d)	NS	392	NS	NS	NS
Percent Recovery		NA	NA	79%	NA	NA	NA
C-WB	1	NA	NA	13 U	NA	NA	NA
C-WB MSD		NA	NA	311	NA	NA	NA
Concentration Recovered		NA	NA	311	NA	NA	NA
Amount Spiked		NS	NS	394	NS	NS	NS
Percent Recovery		NA	NA	79%	NA	NA	NA
RPD		NA	NA	0%	NA	NA	NA
I-Stat		NA	NA	0.00	NA	NA	NA
QC Sample	2	18.2	7.82	4.48	6.57	60.8	17.4
QC Sample, MS		63.0	47.0	49.4	52.5	126	73.2
Concentration Recovered		44.8	39.2	44.9	45.9	65.2	55.8
Amount Spiked		42.0	42.0	42.0	42.0	42.0	42.0
Percent Recovery		107%	93%	107%	109%	155% ^(e)	133% ^(e)
QC Sample	3	36.4	11.4	5.09	12.4 ^(b)	113	41.9
QC Sample, MS		54.6	44.2	33.0	45.0	197	132
Concentration Recovered		18.2	32.8	27.9	32.6	84.0	90.1
Amount Spiked		28.5	28.5	28.5	28.5	28.5	28.5
Percent Recovery		64%	115%	98%	114%	295% ^(e)	316% ^(e)
OBM COMP	4	9.29	3.27 ^(b)	4.87	3.59 U	11.4	5.15 U
OBM COMP, MS		29.5	22.1	24.8	20.1	28.7	19.3
Concentration Recovered		20.2	18.8	19.9	20.1	17.3	19.3
Amount Spiked		22.0	22.0	22.0	22.0	22.0	22.0
Percent Recovery		92%	86%	91%	91%	79%	88%
Standard Reference Material							
SQ-1		99	NC ^(f)	120	120	130	100
Non-certified value		±4	NC	±7	±7	±9	±7
SQ-1	1	72	NA	100	100	160	110
Certified Value		1010	NC	NC	97.3	489	184
NIST 1941a		±140	NC	NC	±8.6	±23	±14
SRM 1941a	2	749	NA	NA	83.8	455	193
SRM 1941a	3	865	NA	NA	90.6	508	208
SRM 1941a	4	893	NA	NA	92.3	608	242 ^(g)

TABLE A.9. (contd)

Sediment Treatment	Batch	LPAHs ($\mu\text{g/kg}$ dry weight)					
		Naptha-lene	Acenaph-thylene	Acenaph-thene	Fluorene	Phenan-threne	Anthra-cene
<u>Analytical Replicates</u>							
OBM COMP, Replicate 1	1	13 U	13 U	13 U	13 U	13 U	13 U
OBM COMP, Replicate 2	1	13 U	13 U	13 U	13 U	13 U	13 U
RPD		NA	NA	NA	NA	NA	NA
I-Stat		NA	NA	NA	NA	NA	NA
QC Sample, Replicate 1	2	40.6	15.5	9.76	18.0	112	64.1
QC Sample, Replicate 2	2	43.1	17.2	13.0	22.3	139	84.4
QC Sample, Replicate 3	2	42.9	26.5	12.1	33.9	314	268
RSD		3%	30%	14%	33% ^(h)	58% ^(h)	81% ^(h)
R-AM, Replicate 1	3	46.0	63.6	32.4	92.2	925	348
R-AM, Replicate 2	3	29.6	32.6	37.8	44.3	456	173
R-AM, Replicate 3	3	38.1	56.6	20.5 ^(b)	94.0	783	259
RSD		22%	32% ^(h)	29%	37% ^(h)	33% ^(h)	34% ^(h)
QC Sample, Replicate 1	4	12.5 B ^(t)	2.46 U	5.98	6.03	18.4 B	6.31 U
QC Sample, Replicate 2	4	11.8 ^(b) B	2.43 U	6.25	5.97	16.2 B	6.23 U
QC Sample, Replicate 3	4	15.7 B	2.31 U	5.48	5.60	19.2 B	5.92 U
RSD		16%	NA	7%	4%	9%	NA

(a) U Undetected at or above detection limit.

(b) Ratio of confirmation ion between the primary and secondary column is outside of the theoretical ratio of 20% established for EPA-CLP programs.

(c) NA Not applicable.

(d) NS Not spiked.

(e) Outside quality control criteria (40%-120%) for matrix spike recoveries.

(f) NC Not certified.

(g) Outside quality control criteria ($\pm 30\%$) for SRMs.(h) Exceeds quality control criteria ($\leq 30\%$) for precision.

(i) B Analyte detected in sample at less than five times the value in associated method blank.

**TABLE A.10. Quality Control Data for Sediment High Molecular Weight Polynuclear Aromatic Hydrocarbons (HPAHs),
Older Bay Mud Study**

Sediment Treatment	HPAHs (µg/kg dry weight)										
	Batch	Fluor- anthene	Pyrene	Benzo[a] anthra- cene	Chrysene	Benzo[b] fluor- anthene	Benzo[k] fluor- anthene	Benzo[a] pyrene	Indeno [1,2,3- c,d] pyrene	Dibenzo- [a,h] anthra- cene	Benzo [g,h] perylene
Method Blank											
Blank	1	10 U ^(e)	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
Blank	2	2.85	1.99 U	1.78	1.83 U	0.84 U	1.84 U	1.99 U	3.90 U	5.62 U	6.10 U
Blank	3	4.52 ^(b)	2.02 U	1.01 U	1.85 U	0.85 U	1.86 U	2.02 U	3.95 U	5.69 U	6.17 U
Blank	4	8.65 ^(b)	11.5 ^(b)	0.76 U	0.84 U	1.60 U	2.70 U	2.93 ^(b)	0.97 U	1.22 U	0.88 U
Matrix Spike											
C-WB	1	13 U	NA ^(c)	13 U	NA	NA	NA	NA	NA	NA	NA
C-WB, MS		317	NA	319	NA	NA	NA	NA	NA	NA	NA
Concentration Recovered		317	NA	319	NA	NA	NA	NA	NA	NA	NA
Amount Spiked		392	NS ^(d)	392	NS	NS	NS	NS	NS	NS	NS
Percent Recovery		81%	NA	81%	NA	NA	NA	NA	NA	NA	NA
C-WB	1	13 U	NA	13 U	NA	NA	NA	NA	NA	NA	NA
C-WB, MSD		332	NA	321	NA	NA	NA	NA	NA	NA	NA
Concentration Recovered		332	NA	321	NA	NA	NA	NA	NA	NA	NA
Amount Spiked		394	NS	394	NS	NS	NS	NS	NS	NS	NS
Percent Recovery		84%	NA	81%	NA	NA	NA	NA	NA	NA	NA
RPD		4%	NA	0%	NA	NA	NA	NA	NA	NA	NA
I-Stat		0.02	NA	0.00	NA	NA	NA	NA	NA	NA	NA
QC Sample	2	105	142	51.1	56.0	110	35.0	104	97.3	12.8	107
QC Sample, MS		150	189	98.9	100	157	81.2	149	142	58.6	147
Concentration Recovered		45.0	47.0	47.8	44.0	47.0	46.2	45.0	44.7	45.8	40.0
Amount Spiked		42.0	42.0	42.0	42.0	42.0	42.0	42.0	42.0	42.0	42.0
Percent Recovery		107%	112%	114%	105%	112%	110%	107%	106%	109%	95%
QC Sample	3	85.9	109	54.1	54.6	42.6	15.9	44.4	32.3	7.84	30.2
QC Sample, MS		236	303	189	238	203	84.8	217	182	52.9	175
Concentration Recovered		150	194	135	183	160	68.9	173	150	45.1	145
Amount Spiked		28.5	28.5	28.5	28.5	28.5	28.5	28.5	28.5	28.5	28.5
Percent Recovery		527% ^(e)	681% ^(e)	473% ^(e)	644% ^(e)	563% ^(e)	242% ^(e)	606% ^(e)	525% ^(e)	158% ^(e)	508% ^(e)

TABLE A.10. (contd)

HPAHs (µg/kg dry weight)													
Sediment Treatment	Batch	Fluor-anthene	Pyrene	Benzo[a]anthracene	Chrysene	Benzo[b]fluor-anthene		Benzo[k]fluor-anthene	Benzo[a]pyrene	Indeno[1,2,3-c,d]pyrene	Dibenzo-[a,h]anthracene		Benzo perylene
						anthene	anthene						
Matrix Spike (cont'd)													
OBM COMP	4	8.64 ^(b)	11.2 U	4.61 ^(b)	0.78 U	1.49 U	2.52 U	3.49 ^(b)	0.90 U	1.14 U	0.82 U		
OBM COMP, MS		26.8	27.1	22.2	23.8	24.2	28.9	21.2	22.1	22.2	21.4		
Concentration Recovered		18.2	27.1	17.6	23.8	24.2	28.9	17.7	22.1	22.2	21.4		
Amount Spiked		22.0	22.0	22.0	22.0	22.0	22.0	22.0	22.0	22.0	22.0	22.0	
Percent Recovery		83%	123% ^(e)	80%	108%	110%	131% ^(e)	81%	100%	101%	97%		
Standard Reference Material													
SQ-1	1	130	110	110	150	NC	NC	130	NC	74	NC		
Non-certified value		±13	±8	±9	±9			±13		±7			
SQ1		140	110	110	120	NA	NA	120	NA	100 ^(f)	NA		
Certified value	2	981	811	427	380	740	361	628	501	73.9	525		
NIST 1941a		±78	±24	±25	±24	±110	±18	±52	±72	±9.7	±67		
SRM NIST 1941a		718	609	371	483	1010 ^(f)	327	508	489	123 ^(f)	453		
SRM NIST 1941a		768 ^(f)	625	421	519 ^(f)	1010 ^(f)	343	519	532	130 ^(f)	476		
SRM NIST 1941a	4	921	739	442	603 ^(f)	1100 ^(f)	382	579	580	128 ^(f)	503		
Analytical Replicates													
OBM COMP, Replicate 1	OBM COMP, Replicate 2	13 U	3.3 J ^(g)	2.5 M ^(h)	2.6 J	6.3 J	6.3 J	3.4 J	13 U	13 U	13 U		
		13 U	2.6 J	1.8 M	1.7 J	4.9 J	4.9 J	2.3 J	13 U	13 U	13 U		
RPD		NA	24%	33% ⁽ⁱ⁾	42% ⁽ⁱ⁾	25%	25%	39% ⁽ⁱ⁾	NA	NA	NA		
I-Stat		NA	0.12	0.16	0.21	0.12	0.12	0.19	NA	NA	NA		
QC Sample, Replicate 1	2	312	480	200	324	637	222	447	291	81.9	293		
QC Sample, Replicate 2	2	235	438	194	332	685	236	466	297	80.2	296		
RPD		28%	9%	3%	2%	7%	6%	4%	2%	2%	1%		
I-Stat		0.14	0.05	0.02	0.01	0.04	0.03	0.02	0.01	0.01	0.01		

TABLE A.10. (contd)

		HPAHs (µg/kg dry weight)									
Sediment Treatment	Batch	Fluor-anthrene	Pyrene	Benzo[a]anthracene	Chrysene	Benzo[b]fluor-anthrene	Benzo[k]fluor-anthrene	Benzo[a]pyrene	Indeno[1,2,3-c,d]pyrene	Dibenzo-[a,h]anthracene	Benzo[g,h]perylene
Analytical Replicates (contd)											
QC Sample, Replicate 1	2	166	309	106	153	314	104	232	190	36.2	202
QC Sample, Replicate 2	2	193	352	164	243	452	163	344	252	55.0	260
QC Sample, Replicate 3	2	335	541	228	260	420	150	343	245	51.6	251
RSD		39% ^(f)	31% ^(f)	37% ^(f)	26%	18%	22%	21%	15%	21%	13%
R-AM, Replicate 1	3	616	751	386	382	290	105	308	182	44.3	165
R-AM, Replicate 2	3	322	395	194	190	167	59.8	185	121	23.5	115
R-AM, Replicate 3	3	452	545	261	255	200	71.3	215	129	30.4	118
RSD		32% ^(f)	32% ^(f)	5%	35% ^(f)	29%	30%	27%	23%	32% ^(f)	21%
QC Sample, Replicate 1	4	14.4 B ^(k)	16.7 B	6.36 ^(b)	3.69 ^(b)	8.87 ^(b)	3.08 U	3.81 B ^(b)	5.11 ^(b)	1.39 U	5.86 ^(b)
QC Sample, Replicate 2	4	12.6 B ^(b)	16.1 B	6.23 ^(b)	3.61 ^(b)	8.83 ^(b)	3.04 U	3.96 B ^(b)	4.92 ^(b)	1.38 U	6.44 ^(b)
QC Sample, Replicate 3	4	14.2 B ^(b)	16.0 B	5.88 ^(b)	3.74 ^(b)	7.77 ^(b)	2.89 U	2.25 U	4.55 ^(b)	1.31 U	5.54 ^(b)
RSD		7%	2%	4%	2%	7%	NA	NA	NA	NA	8%

(a) U Undetected at or above detection limit.

(b) Ratio of confirmation ion between the primary and secondary column is outside of the theoretical ratio of 20% established for EPA-CLP programs.

(c) NA Not applicable.

(d) NS Not spiked.

(e) Outside quality control criteria (40% - 120%) for matrix spike recoveries.

(f) Outside quality control criteria (±30%) for SRMs.

(g) J Analyte detected below method detection limit (MDL), but above instrument detection limit (IDL).

(h) M Indicates an estimated value of analyte found and confirmed by analyst but with low spectral match parameters.

(i) Precision criteria does not apply because sample results are <10 times the MDL.

(j) Exceeds quality control criteria (≤30%) for precision.

(k) B Analyte detected in sample at less than five times the value in the associated method blank.

TABLE A.11. Surrogate Percent Recoveries for Polynuclear Aromatic Hydrocarbons (PAHs) Including Quality Control Data for Sediment, Older Bay Mud Study

Sediment Treatment	Batch	Surrogate Percent Recoveries				
		Diphenyl d10	p-Terphenyl d14			
January 1993						
OBM COMP, Replicate 1	1	50	65			
OBM COMP, Replicate 2	1	65	77			
C-SB	1	55	76			
C-WB	1	70	74			
C-SPB	1	70	60			
Quality Control Data						
Method Blank						
Blank	1	75	84			
Matrix Spike						
C-WB MS	1	73	80			
C-WB MSD	1	67	83			
Standard Reference Material						
SQ1	1	66	67			
	Batch	Naphthalene d8	Acenaphthene d10	Chrysene d12	Perylene d12	Dibenzo(a,h,i)anthracene d14
January 1994						
OBM COMP	2	69	72	84	ND ^(a)	79
R-OS	3	63	67	95	ND	90
R-BF	3	69	73	96	ND	83
R-AM, Replicate 1	3	71	72	94	ND	84
R-AM, Replicate 2	3	65	67	85	ND	75
R-AM, Replicate 3	3	69	71	93	ND	84
October 1994						
OBM COMP	4	62	63	66	63	65
R-OS	4	64	67	65	66	64
R-BF	4	44	48	71	67	69
R-AM	4	48	57	76	70	75
Quality Control Data						
Method Blank						
Blank	2	84	81	90	ND	75
Blank	3	85	78	80	ND	69
Blank	4	46	56	60	62	55
Matrix Spike						
QC Sample	2	69	73	86	ND	82
QC Sample, MS	2	74	77	64	ND	82
QC Sample	3	54	69	85	ND	82
QC Sample, MS	3	76	75	95	ND	87
OBM COMP	4	62	63	66	63	65
OBM COMP, MS	4	58	61	64	61	64

TABLE A.11. (contd)

Sediment Treatment	Batch	Naph- thalene d8	Acenaph- thene d10	Chrysene d12	Perylene d12	Dibenzo(a,h,i) anthracene d14
<u>Analytical Replicates</u>						
QC Sample, Replicate 1	2	75	78	89	ND	80
QC Sample, Replicate 2	2	65	68	87	ND	75
QC Sample, Replicate 1	2	68	75	95	ND	86
QC Sample, Replicate 2	2	56	65	83	ND	76
QC Sample, Replicate 3	2	72	75	91	ND	83
R-AM, Replicate 1	3	71	72	94	ND	84
R-AM, Replicate 2	3	65	67	85	ND	75
R-AM, Replicate 3	3	69	71	93	ND	84
QC Sample, Replicate 1	4	69	72	64	70	67
QC Sample, Replicate 2	4	63	66	66	65	64
QC Sample, Replicate 3	4	65	68	65	70	63

(a) ND No data.

TABLE A.12. Sediment Chlorinated Pesticide Results (alphabetical, Aldrin - 4,4'-DDT), Older Bay Mud Study

Sediment Treatment	Batch	Pesticides (µg/kg dry weight)										
		Aldrin	Alpha-BHC	Beta-BHC	Delta-BHC	Gamma-BHC	Alpha-chlordane	Gamma-chlordane	T chlordane	4,4'-DDD	4,4'-DDE	4,4'-DDT
Target DL ^(a)		2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
January 1993												
OBM COMP, Replicate 1	2	0.75 U ^(b)	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	ND ^(c)	1.4 J ^(d)	1.5 U	1.5 U
OBM COMP, Replicate 2	2	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	ND	2.3	1.5 U	0.71J
C-SB	1	1.4 U	1.4 U	1.4 U	1.1	1.4 U	1.4 U	1.4 U	ND	5.0 U	2.9 U	2.9 U
C-WB	1	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	ND	2.0 U	2.0 U	2.0 U
C-SPB, Replicate 1	3	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	ND	3.2 U	3.2 U	3.2 U
C-SPB, Replicate 2	3	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	ND	2.4 J	2.1 J	3.2 U
January 1994												
OBM COMP	4	0.05 U	0.91 U	0.91 U	0.91 U	0.05 U	ND	ND	30 U	0.13 U	0.06 U	0.38 U
R-OS	5	0.06 U	1.22 U	1.22 U	1.22 U	0.06 U	ND	ND	30 U	0.18 U	0.94	0.51 U
R-BF	5	5.07	1.78 U	1.78 U	1.78 U	4.83	ND	ND	30 U	1.05	1.69	12.1
R-AM, Replicate 1	5	0.04 U	0.90 U	0.90 U	0.90 U	0.05 U	ND	ND	30 U	0.56	0.06 U	0.66
R-AM, Replicate 2	5	0.04 U	0.88 U	0.88 U	0.88 U	0.05 U	ND	ND	30 U	0.66	0.05 U	0.46
R-AM, Replicate 3	5	0.04 U	0.87 U	0.87 U	0.87 U	0.05 U	ND	ND	30 U	0.13 U	0.05 U	0.45
October 1994												
OBM COMP	6	0.18 U	0.30 U	0.30 U	0.30 U	0.19 U	ND	ND	30 U	0.22 U	0.12 U	0.63 U
R-OS	6	0.25 U	0.42 U	0.42 U	0.42 U	0.26 U	ND	ND	30 U	0.31 U	1.29	0.87 U
R-BF	6	2.15	0.57 U	0.57 U	0.57 U	0.35 U	ND	ND	30 U	5.19	2.61	1.19 U
R-AM	6	1.32	0.28 U	0.28 U	0.28 U	0.17 U	ND	ND	30 U	0.21 U	0.11 U	0.58 U

(a) DL Detection limit.
 (b) U Undetected at or above detection limit.
 (c) ND No data.
 (d) J Analyte detected below method detection limit (MDL), and above instrument detection limit (IDL).

**TABLE A.13. Sediment Chlorinated Pesticide Results (alphabetical, Dieldrin - Toxaphene),
Older Bay Mud Study**

Sediment Treatment	Batch	Dieldrin	Pesticides ($\mu\text{g/kg}$ dry weight)							Toxa- phene
			Endo- Sulfan I	Endo- Sulfan II	Endo- sulfan Sulfate	Endrin	Endrin Aldehyde	Hepta- chlor	Hepta- chlor epoxide	
Target DL ^(a)		2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	30
<u>January 1993</u>										
OBM COMP, Replicate 1	2	1.5 U ^(b)	0.75 U	1.5 U	1.5 U	1.5 U	1.5 U	0.75 U	0.75 U	75 U
OBM COMP, Replicate 2	2	1.5 U	0.75 U	1.5 U	1.5 U	1.5 U	1.5 U	0.75 U	0.75 U	75 U
C-SB	1	2.9 U	1.4 U	2.9 U	2.9 U	2.9 U	2.9 U	1.4 U	1.4 U	140 U
C-WB	1	2.0 U	1.0 U	2.0 U	2.0 U	2.0 U	2.0 U	1.0 U	1.0 U	40 U
C-SPB, Replicate 1	3	3.2 U	1.6 U	3.2 U	3.2 U	3.2 U	3.2 U	1.6 U	1.6 U	160 U
C-SPB, Replicate 2	3	3.2 U	1.6 U	3.2 U	3.2 U	3.2 U	3.2 U	1.6 U	1.6 U	160 U
<u>January 1994</u>										
OBM COMP	4	0.28 U	0.91 U	0.91 U	0.91 U	0.91 U	0.91 U	0.34 U	0.21 U	30 U
R-OS	5	0.38 U	1.22 U	1.22 U	1.22 U	1.22 U	1.22 U	0.45 U	0.28 U	30 U
R-BF	5	13.0	1.78 U	1.78 U	1.78 U	14.7	1.78 U	2.59	0.89	30 U
R-AM, Replicate 1	5	0.28 U	0.90 U	0.90 U	0.90 U	0.90 U	0.90 U	0.33 U	0.20 U	30 U
R-AM, Replicate 2	5	0.28 U	0.88 U	0.88 U	0.88 U	0.88 U	0.88 U	0.33 U	0.20 U	30 U
R-AM, Replicate 3	5	0.27 U	0.87 U	0.87 U	0.87 U	0.87 U	0.87 U	0.32 U	0.20 U	30 U
<u>October 1994</u>										
OBM COMP	6	0.18 U	0.30 U	0.30 U	0.30 U	0.30 U	0.30 U	0.06 U	0.26 U	30 U
R-OS	6	0.25 U	0.42 U	0.42 U	0.42 U	0.42 U	0.42 U	0.08 U	0.36 U	30 U
R-BF	6	0.33 U	0.57 U	0.57 U	0.57 U	0.57 U	0.57 U	0.11 U	0.50 U	30 U
R-AM	6	0.16 U	0.28 U	0.28 U	0.28 U	0.28 U	0.28 U	0.05 U	0.24 U	30 U

(a) DL Detection limit.

(b) U Undetected at or above detection limit.

TABLE A.14. Quality Control Data for Sediment Chlorinated Pesticides Results (alphabetical, Aldrin - 4,4'-DDT),
Older Bay Mud Study

Sediment Treatment	Batch	Aldrin	Pesticides (µg/kg dry weight)										4,4'-DDD	4,4'-DDE	4,4'-DDT
			Alpha-BHC	Beta-BHC	Delta-BHC	Gamma-BHC	Alpha-chlordane	Gamma-chlordane	T chlordane	4,4'-DDD	4,4'-DDE	4,4'-DDT			
Method Blank															
Blank	1	1.0 U ^(a)	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	ND ^(b)	2.0 U	2.0 U	2.0 U	
Blank	2	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	ND	1.5 U	1.5 U	1.5 U	
Blank	3	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	ND	3.2 U	3.2 U	3.2 U	
Blank	4	0.06 U	1.20 U	1.20 U	1.20 U	0.06 U	0.06 U	ND	ND	ND	30 U	0.17 U	0.07 U	0.50 U	
Blank	5	0.06 U	1.21 U	1.21 U	1.21 U	0.06 U	0.06 U	ND	ND	ND	30 U	0.18 U	0.08 U	0.50 U	
Blank	6	0.23 U	0.39 U	0.39 U	0.39 U	0.24 U	0.24 U	ND	ND	ND	30 U	0.29 U	0.16 U	0.83 U	
Matrix Spike															
C-WB	1	1.0 U	NA (c)	NA	NA	1.0 U	1.0 U	NA	NA	NA	NA	NA	NA	2.0 U	
C-WB MS		2.99	NA	NA	NA	3.1	3.1	NA	NA	NA	NA	NA	NA	6.25	
Concentration Recovered		2.99	NA	NA	NA	3.1	3.1	NA	NA	NA	NA	NA	NA	6.25	
Amount Spiked		3.92	NS (d)	NS	NS	3.92	3.92	NS	NS	NS	NS	NS	NS	7.84	
Percent Recovery		76%	NA	NA	NA	79%	79%	NA	NA	NA	NA	NA	NA	80%	
C-WB	1	1.0 U	NA	NA	NA	1.0 U	1.0 U	NA	NA	NA	NA	NA	NA	2.0 U	
C-WB MSD		3.04	NA	NA	NA	3.17	3.17	NA	NA	NA	NA	NA	NA	6.45	
Concentration Recovered		3.04	NA	NA	NA	3.17	3.17	NA	NA	NA	NA	NA	NA	6.45	
Amount Spiked		3.94	NS	NS	NS	3.94	3.94	NS	NS	NS	NS	NS	NS	7.89	
Percent Recovery		77%	NA	NA	NA	80%	80%	NA	NA	NA	NA	NA	NA	82%	
RPD		1%	NA	NA	NA	2%	2%	NA	NA	NA	NA	NA	NA	3%	
I-Stat		0.01	NA	NA	NA	0.01	0.01	NA	NA	NA	NA	NA	NA	0.01	
QC Sample	4	1.26	NA	NA	NA	0.06 U	0.06 U	NA	NA	NA	NA	NA	NA	0.73	
QC Sample, MS		4.37	NA	NA	NA	4.07	4.07	NA	NA	NA	NA	NA	NA	20.0	
Concentration Recovered		3.11	NA	NA	NA	4.07	4.07	NA	NA	NA	NA	NA	NA	19.3	
Amount Spiked		4.20	NS	NS	NS	4.20	4.20	NS	NS	NS	NS	NS	NS	16.8	
Percent Recovery		74%	NA	NA	NA	97%	97%	NA	NA	NA	NA	NA	NA	115%	
QC Sample	5	0.04 U	NA	NA	NA	0.04 U	0.04 U	NA	NA	NA	NA	0.12 U	NA	NA	
QC Sample, MS		2.89	NA	NA	NA	2.48	2.48	NA	NA	NA	NA	11.1	NA	NA	
Concentration Recovered		2.89	NA	NA	NA	2.48	2.48	NA	NA	NA	NA	11.1	NA	NA	
Amount Spiked		2.85	NS	NS	NS	2.85	2.85	NS	NS	NS	NS	11.6	NS	NS	
Percent Recovery		101%	NA	NA	NA	87%	87%	NA	NA	NA	NA	96%	NA	NA	

TABLE A.14. (contd)

Sediment Treatment	Batch	Aldrin	Pesticides (µg/kg dry weight)										T chlordane	4,4'- DDD	4,4'- DDE	4,4'- DDT
			Alpha- BHC	Beta- BHC	Delta- BHC	Gamma- BHC	Alpha- chlordane	Gamma- chlordane	4,4'- chlordane	4,4'- DDE	4,4'- DDT					
Matrix Spike (contd)																
OBM COMP	6	0.18 U	NA	NA	NA	0.19 U	NA	NA	NA	NA	NA	NA	NA	NA	0.63 U	0.63 U
OBM COMP, MS		1.80	NA	NA	NA	1.75	NA	NA	NA	NA	NA	NA	NA	NA	NA	11.9
Concentration Recovered		1.80	NA	NA	NA	1.75	NA	NA	NA	NA	NA	NA	NA	NA	NA	11.9
Amount Spiked		2.20	NS	NS	NS	2.20	NS	NS	NS	NS	NS	NS	NS	NS	NS	8.80
Percent Recovery		82%	NA	NA	NA	80%	NA	NA	NA	NA	NA	NA	NA	NA	135% ^(e)	135% ^(e)
Standard Reference Material																
Certified Value NIST 1941a		NC ^(f)	NC	NC	NC	NC	NC	NC	NC	NC	NC	5.06 ±0.58	6.59 ±0.56	NC	NC	NC
SRM NIST 1941a	4	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	5.55	8.35	NA	NA	NA
SRM NIST 1941a	5	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	7.97 ^(g)	7.72	NA	NA	NA
SRM NIST 1941a	6	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	5.80	8.85 ^(g)	NA	NA	NA
Analytical Replicates																
OBM COMP, Replicate 1	2	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	1.4 J ^(h)	1.5 U	1.5 U	1.5 U	1.5 U	1.5 U
OBM COMP, Replicate 2	2	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	2.3	1.5 U	0.71 J	1.5 U	0.71 J	0.71 J
RPD		NA	NA	NA	NA	NA	NA	NA	NA	NA	49% ⁽ⁱ⁾	NA	NA	NA	NA	NA
I-Stat		NA	NA	NA	NA	NA	NA	NA	NA	NA	0.24	NA	NA	NA	NA	NA
C-SPB, Replicate 1	3	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	3.2 U	3.2 U	3.2 U	3.2 U	3.2 U	3.2 U
C-SPB , Replicate 2	3	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	2.4 J	2.1 J	3.2 U	2.1 J	3.2 U	3.2 U
RPD		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
I-Stat		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
QC Sample, Replicate 1	4	3.39	1.59 U	1.59 U	1.59 U	1.59 U	0.14	NA	NA	NA	267 ^(j)	34.6 ^(j)	304 ^(j)	34.6 ^(j)	304 ^(j)	304 ^(j)
QC Sample, Replicate 2	4	3.78	1.49 U	1.49 U	1.49 U	1.49 U	0.14	NA	NA	NA	298 ^(j)	37.5 ^(j)	412 ^(j)	37.5 ^(j)	412 ^(j)	412 ^(j)
RPD		11%	NA	NA	NA	0%	NA	NA	NA	NA	11%	8%	30%	8%	30%	30%
I-Stat		0.05	NA	NA	NA	0.00	NA	NA	NA	NA	0.05	0.04	0.15	0.04	0.15	0.15

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(b) ND No data.

(b) ND No data.
(c) NA Not applicable.

(c) NA Not applicable.

(d) NS Not spiked.

(e) Outside quality of

(f) NC Not certified.

(g) Outside quality control.

saturnus quality control criteria (±30%) for SMS.

(h) J Analyte detected below method detection limit (MDL), but above instrument detection limit (IDL).

(ii) Precision criteria does not apply because sample results are <10 times the method detection limit (MDL). Sample was diluted and analyzed due to low concentration.

J) Sample was diluted and analyzed due to analyte concentrations above the instrument linear range of calibration.

TABLE A.15. Quality Control Data for Sediment Chlorinated Pesticide Results (alphabetical, Dieldrin - Toxaphene),
Older Bay Mud Study

Sediment Treatment	Batch	Dieldrin	Pesticides (µg/kg dry weight)					Hepta-chlor epoxide	Toxa-phene	
			Endo-sulfan I	Endo-sulfan II	Endo-sulfan Sulfate	Endrin	Endrin Aldehyde			
Method Blank										
Blank	1	2.0 U ^(a)	1.0 U	2.0 U	2.0 U	2.0 U	2.0 U	1.0 U	1.0 U	40 U
Blank	2	1.5 U	0.75 U	1.5 U	1.5 U	1.5 U	1.5 U	0.75 U	0.75 U	75 U
Blank	3	3.2 U	1.6 U	3.2 U	3.2 U	3.2 U	3.2 U	1.6 U	1.6 U	160 U
Blank	4	0.37 U	1.20 U	1.20 U	1.20 U	1.20 U	1.20 U	0.44 U	0.27 U	30 U
Blank	5	0.38 U	1.21 U	1.21 U	1.21 U	1.21 U	1.21 U	0.45 U	0.27 U	30 U
Blank	6	0.23 U	0.39 U	0.39 U	0.39 U	0.39 U	0.39 U	0.07 U	0.34 U	30 U
Matrix Spike										
C-WB	1	2.0 U	NA ^(b)	NA	NA	2.0 U	2.0 U	1.0 U	NA	NA
C-WB MS		7.10	NA	NA	NA	5.81	5.81	3.10	NA	NA
Concentration Recovered		7.10	NA	NA	NA	5.81	5.81	3.10	NA	NA
Amount Spiked		7.84	NS ^(c)	NS	NS	7.84	7.84	3.92	NS	NS
Percent Recovery		91%	NA	NA	NA	74%	74%	79%	NA	NA
C-WB	1	2.0 U	NA	NA	NA	2.0 U	2.0 U	1.0 U	NA	NA
C-WB MSD		7.26	NA	NA	NA	6.03	6.03	3.12	NA	NA
Concentration Recovered		7.26	NA	NA	NA	6.03	6.03	3.12	NA	NA
Amount Spiked		7.89	NS	NS	NS	7.89	7.89	3.94	NS	NS
Percent Recovery		92%	NA	NA	NA	76%	76%	79%	NA	NA
RPD		2%	NA	NA	NA	3%	3%	0%	NA	NA
I-Stat		0.01	NA	NA	NA	0.02	0.02	0.00	NA	NA
QC Sample	4	1.06	NA	NA	NA	1.25 U	1.25 U	0.46 U	NA	NA
QC Sample, MS		14.9	NA	NA	NA	15.7	15.7	4.38	NA	NA
Concentration Recovered		13.8	NA	NA	NA	15.7	15.7	4.38	NA	NA
Amount Spiked		16.8	NS	NS	NS	16.0	16.0	4.20	NS	NS
Percent Recovery		82%	NA	NA	NA	98%	98%	104%	NA	NA
QC Sample	5	0.26 U	NA	NA	NA	0.83 U	0.83 U	0.31	NA	NA
QC Sample, MS		9.98	NA	NA	NA	11.8	11.8	2.78	NA	NA
Concentration Recovered		9.98	NA	NA	NA	11.8	11.8	2.47	NA	NA
Amount Spiked		11.6	NS	NS	NS	11.6	11.6	2.85	NS	NS
Percent Recovery		86%	NA	NA	NA	102%	102%	87%	NA	NA

TABLE A.15. (contd)

Sediment Treatment	Batch	Dieldrin	Pesticides (µg/kg dry weight)							Hepta-chlor epoxide	Toxa-phene
			Endo-sulfan I	Endo-sulfan II	Endo-sulfan Sulfate	Endrin	Endrin Aldehyde	Hepta-chlor			
Matrix Spike (contd)											
OBM COMP	6	0.18 U	NA	NA	NA	0.30 U	NA	0.06 U	NA	NA	NA
OBM COMP, MS		8.40	NA	NA	NA	10.4	NA	1.81	NA	NA	NA
Concentration Recovered		8.40	NA	NA	NA	10.4	NA	1.81	NA	NA	NA
Amount Spiked		8.80	NS	NS	NS	8.80	NS	2.20	NS	NS	NS
Percent Recovery		95%	NA	NA	NA	118%	NA	82%	NA	NA	NA
Standard Reference Material											
SRM data not available.											
Analytical Replicates											
OBM COMP, Replicate 1	2	1.5 U	0.75 U	1.5 U	1.5 U	1.5 U	1.5 U	0.75 U	0.75 U	75 U	75 U
OBM COMP, Replicate 2	2	1.5 U	0.75 U	1.5 U	1.5 U	1.5 U	1.5 U	0.75 U	0.75 U	75 U	75 U
RPD		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
I-Stat		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
C-SPB, Replicate 1	3	3.2 U	1.6 U	3.2 U	3.2 U	3.2 U	3.2 U	1.6 U	1.6 U	160 U	160 U
C-SPB, Replicate 2	3	3.2 U	1.6 U	3.2 U	3.2 U	3.2 U	3.2 U	1.6 U	1.6 U	160 U	160 U
RPD		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
I-Stat		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
QC Sample, Replicate 1	4	22.3	1.59 U	1.59 U	1.59 U	1.59 U	1.59 U	0.59 U	1.88	30 U	30 U
QC Sample, Replicate 2	4	25.9	1.49 U	1.49 U	1.49 U	1.49 U	1.49 U	0.55 U	2.07	30 U	30 U
RPD		15%	NA	NA	NA	NA	NA	NA	10%	NA	NA
I-Stat		0.07	NA	NA	NA	NA	NA	NA	0.05	NA	NA
QC Sample, Replicate 1	4	9.60	1.30 U	1.30 U	1.30 U	1.30 U	1.30 U	0.48 U	1.50	30 U	30 U
QC Sample, Replicate 2	4	8.69	1.52 U	1.52 U	1.52 U	1.52 U	1.52 U	0.56 U	1.56	30 U	30 U
QC Sample, Replicate 3	4	9.22	1.54 U	1.54 U	1.54 U	1.54 U	1.54 U	0.57 U	1.86	30 U	30 U
RSD		5%	NA	NA	NA	NA	NA	NA	12%	NA	NA

TABLE A.15. (contd)

Sediment Treatment	Batch	Pesticides (µg/kg dry weight)								
		Dieldrin	Endo-sulfan			Endrin	Endrin Aldehyde	Hepta-chlor	Hepta-chlor epoxide	Toxa- phene
			Endo-sulfan I	Endo-sulfan II	Sulfate					
Analytical Replicates (contd)										
R-AM, Replicate 1	5	0.28 U	0.90 U	0.90 U	0.90 U	0.90 U	0.90 U	0.33 U	0.20 U	30 U
R-AM, Replicate 2	5	0.28 U	0.88 U	0.88 U	0.88 U	0.88 U	0.88 U	0.33 U	0.20 U	30 U
R-AM, Replicate 3	5	0.27 U	0.87 U	0.87 U	0.87 U	0.87 U	0.87 U	0.32 U	0.20 U	30 U
RSD		NA	NA	NA	NA	NA	NA	NA	NA	NA
QC Sample, Replicate 1	6	0.35 U	0.59 U	0.59 U	0.59 U	0.59 U	0.59 U	0.11 U	0.52 U	30 U
QC Sample, Replicate 2	6	0.34 U	0.59 U	0.59 U	0.59 U	0.59 U	0.59 U	0.11 U	0.51 U	30 U
QC Sample, Replicate 3	6	0.33 U	0.56 U	0.56 U	0.56 U	0.56 U	0.56 U	0.10 U	0.49 U	30 U
RSD		NA	NA	NA	NA	NA	NA	NA	NA	NA

(a) U Undetected at or above detection limit.

(b) NA Not applicable.

(c) NS Not spiked.

TABLE A.16. Sediment Polychlorinated Biphenyl (PCB) Results, Older Bay Mud Study

Sediment Treatment	Batch	PCBs ($\mu\text{g/kg}$ dry weight)			
		Aroclor 1242/1016	Aroclor 1248	Aroclor 1254	Aroclor 1260
Target DL ^(a)		20	20	20	20
<u>January 1993</u>					
OBM COMP, Replicate 1	2	15 U ^(b)	15 U	15 U	15 U
OBM COMP, Replicate 2	2	15 U	15 U	15 U	15 U
C-SB	1	29 U	60 U	29 U	29 U
C-WB	1	15 U	15 U	15 U	15 U
C-SPB, Replicate 1	3	32 U	32 U	32 U	32 U
C-SPB, Replicate 2	3	32 U	32 U	32 U	32 U
<u>January 1994</u>					
OBM COMP	4	20 U	20 U	20 U	20 U
R-OS	5	20 U	20 U	20 U	20 U
R-BF	5	20 U	20 U	23.3	20 U
R-AM, Replicate 1	5	20 U	20 U	20 U	20 U
R-AM, Replicate 2	5	20 U	20 U	20 U	20 U
R-AM, Replicate 3	5	20 U	20 U	20 U	20 U
<u>October 1994</u>					
OBM COMP	6	20 U	20 U	20 U	20 U
R-OS	6	20 U	20 U	20 U	20 U
R-BF	6	20 U	20 U	46.8	20 U
R-AM	6	20 U	20 U	20 U	20 U

(a) DL Detection limit.

(b) U Undetected at or above detection limit.

TABLE A.17. Quality Control Data for Sediment Polychlorinated Biphenyl (PCB) Results, Older Bay Mud Study

Sediment Treatment	Batch	PCBs (μg/kg dry weight)			
		Aroclor 1242/1016	Aroclor 1248	Aroclor 1254	Aroclor 1260
<u>Method Blank</u>					
Blank	1	15 U ^(a)	15 U	15 U	15 U
Blank	2	15 U	15 U	15 U	15 U
Blank	3	32 U	32 U	32 U	32 U
Blank	4	20 U	20 U	20 U	20 U
Blank	5	20 U	20 U	20 U	20 U
Blank	6	20 U	20 U	20 U	20 U

Matrix Spike

QC Sample	4	NA ^(b)	NA	20 U	NA
QC Sample, MS		NA	NA	92.1	NA
Concentration Recovered		NA	NA	92.1	NA
Amount Spiked		NS ^(c)	NS	84.0	NS
Percent Recovery		NA	NA	110%	NA
QC Sample	5	NA	NA	20 U	NA
QC sample, MS		NA	NA	55.8	NA
Concentration Recovered		NA	NA	55.8	NA
Amount Spiked		NS	NS	57.0	NS
Percent Recovery		NA	NA	98%	NA
OBM COMP	6	NA	NA	20 U	NA
OBM COMP, MS		NA	NA	44.5	NA
Concentration Recovered		NA	NA	44.5	NA
Amount Spiked		NS	NS	44.0	NS
Percent Recovery		NA	NA	101%	NA

Standard Reference Material

SRM data not available for PCBs.

Analytical Replicates

OBM COMP, Replicate 1	2	15 U	15 U	15 U	15 U
OBM COMP, Replicate 2	2	15 U	15 U	15 U	15 U
RPD		NA	NA	NA	NA
I-Stat		NA	NA	NA	NA

TABLE A.17. (contd)

Sediment Treatment	Batch	PCBs ($\mu\text{g/kg}$ dry weight)			
		Aroclor 1242/1016	Aroclor 1248	Aroclor 1254	Aroclor 1260
C-SPB, Replicate 1	3	32 U	32 U	32 U	32 U
C-SPB, Replicate 2	3	32 U	32 U	32 U	32 U
RPD		NA	NA	NA	NA
I-Stat		NA	NA	NA	NA
QC Sample, Replicate 1	4	20 U	20 U	155	20 U
QC Sample, Replicate 2	4	20 U	20 U	210	20 U
RPD		NA	NA	30%	NA
I-Stat		NA	NA	0.15	NA
QC Sample, Replicate 1	4	20 U	20 U	93.8	20 U
QC Sample, Replicate 2	4	20 U	20 U	82.7	20 U
QC Sample, Replicate 3	4	20 U	20 U	101	20 U
RSD		NA	NA	10%	NA
R-AM, Replicate 1	5	20 U	20 U	20 U	20 U
R-AM, Replicate 2	5	20 U	20 U	20 U	20 U
R-AM, Replicate 3	5	20 U	20 U	20 U	20 U
RSD		NA	NA	NA	NA
QC Sample, Replicate 1	6	20 U	20 U	20 U	20 U
QC Sample, Replicate 2	6	20 U	20 U	20 U	20 U
QC Sample, Replicate 3	6	20 U	20 U	20 U	20 U
RSD		NA	NA	NA	NA

(a) U Undetected at or above detection limit.

(b) NA Not applicable.

(c) NS Not spiked.

TABLE A.18. Surrogate Percent Recoveries and Quality Control Data for Chlorinated Pesticides and Polychlorinated Biphenyls (PCBs) in Sediment, Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Batch</u>	<u>Surrogate Percent Recoveries</u>			
		<u>Decachloro-biphenyl (DCBP)</u>	<u>Tetrachloro-Metaxylene (TCMX)</u>	<u>PCB 103</u>	<u>PCB 198</u>
<u>January 1993</u>					
OBM COMP, Replicate 1	2	110	81	NA ^(a)	NA
OBM COMP, Replicate 2	2	113	80	NA	NA
C-SB	1	80	84	NA	NA
C-WB	1	92	85	NA	NA
C-SPB, Replicate 1	3	13 ^(b)	10 ^(b)	NA	NA
C-SPB, Replicate 2	3	69	77	NA	NA
<u>January 1994</u>					
OBM COMP	4	NA	NA	70	72
R-OS	5	NA	NA	68	70
R-BF	5	NA	NA	65	94
R-AM, Replicate 1	5	NA	NA	65	76
R-AM, Replicate 2	5	NA	NA	61	70
R-AM, Replicate 3	5	NA	NA	65	73
<u>October 1994</u>					
OBM COMP	6	NA	NA	70	63
R-OS	6	NA	NA	81	67
R-BF	6	NA	NA	77	61
R-AM	6	NA	NA	81	58
<u>Quality Control Data</u>					
<u>Method Blank</u>					
Blank	1	91	88	NA	NA
Blank	2	114	81	NA	NA
Blank	3	13 ^(b)	10 ^(b)	NA	NA
Blank	4	NA	NA	76	78
Blank	5	NA	NA	74	75
Blank	6	NA	NA	64	62
<u>Matrix Spike</u>					
C-WB MS	1	96	90	NA	NA
C-WB MSD	1	97	87	NA	NA

TABLE A.18. (contd)

<u>Sediment Treatment</u>	<u>Batch</u>	<u>Surrogate Percent Recoveries</u>			
		<u>Decachloro- biphenyl (DCBP)</u>	<u>Tetrachloro- Metaxylene (TCMX)</u>	<u>PCB 103</u>	<u>PCB 198</u>
<u>Matrix Spike (contd)</u>					
QC Sample	4	NA	NA	65	71
QC Sample, MS	4	NA	NA	65	71
QC Sample	5	NA	NA	66	67
QC Sample, MS	5	NA	NA	66	71
OBM COMP	6	NA	NA	70	63
OBM COMP, MS	6	NA	NA	70	62

Standard Reference Material

SRM data not available for PCBs.

Analytical Replicates

QC Sample, Replicate 1	4	NA	NA	65	83
QC Sample, Replicate 2	4	NA	NA	59	75
QC Sample, Replicate 1	4	NA	NA	60	81
QC Sample, Replicate 2	4	NA	NA	55	70
QC Sample, Replicate 3	4	NA	NA	62	78
R-AM, Replicate 1	5	NA	NA	65	76
R-AM, Replicate 2	5	NA	NA	61	70
R-AM, Replicate 3	5	NA	NA	65	73
QC Sample, Replicate 1	6	NA	NA	79	64
QC Sample, Replicate 2	6	NA	NA	75	61
QC Sample, Replicate 3	6	NA	NA	78	65

(a) NA Not applicable.

(b) Recovery outside of quality control range (40%-120%).

TABLE A.19. Sediment Metal Results, Dry Weight, Older Bay Mud Study

Sediment Treatment	Batch	Metals (mg/kg dry weight)									
		Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
Target DL ^(a)		0.02	2.5	0.1	33	5.5	0.01	7.5	6.2	0.20	7.8
<u>January 1993</u>											
OBM COMP	1	0.11	3.30	0.84	148	32.9	0.057	70.0	14.6	0.30	72.4
C-SB	1	0.18	10.4	0.93	96.0	29.9	0.073	40.3	8.90	0.86	83.0
C-WB	1	0.01	2.15	0.11	234	10.6	0.023	40.4	6.10	0.13 U ^(b)	43.3
C-SPB, Replicate 1	1	0.29	12.0	0.24	160	53.7	0.336	97.3	31.4	0.22	119
C-SPB, Replicate 2	1	0.30	14.0	0.24	173	53.1	0.348	99.5	24.8	0.22	125
<u>October 1994</u>											
OBM COMP	2	0.11	3.28	0.56	142	27.4	0.044	62.7	10.6	0.17 U	68.3
R-OS	2	0.44	5.39	0.35	186	31.8	0.123	77.3	8.0	1.34	94.7
R-BF	2	0.44	9.4	0.14	178	60.6	0.315	111	35.3	0.29	138
R-AM	2	0.026	5.88	0.015	95.4	4.30	0.049	33.6	13.9	0.17 U	32.8

(a) DL Detection limit.

(b) U Undetected at or above detection limit.

TABLE A.20. Quality Control Data for Sediment Metals, Dry Weight, Older Bay Mud Study

Sediment Treatment	Batch	Metals (mg/kg dry weight)									
		Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
Method Blank											
Blank 1	1	0.02	NA ^(a)	0.01 U ^(b)	NA	NA	0.02 U	NA	NA	0.13 U	NA
Blank 2	2	0.026	NA	0.032	NA	NA	0.002 U	NA	NA	0.17 U	NA
Matrix Spike											
C-SPB	1	0.30	NA	0.24	NA	NA	0.342	NA	NA	0.22	NA
C-SPB MS		2.87	NA	3.16	NA	NA	2.85	NA	NA	2.49	NA
Concentration Recovered		2.57	NA	2.92	NA	NA	2.51	NA	NA	2.27	NA
Amount Spiked		2.50	NS ^(c)	2.50	NS	NS	2.50	NS	NS	2.50	NS
Percent Recovery		103%	NA	117%	NA	NA	100%	NA	NA	91%	NA
QC Sample	2	0.100	NA	0.207	NA	NA	0.058	NA	NA	0.17 U	NA
QC Sample, MS		2.39	NA	2.29	NA	NA	1.60	NA	NA	2.13	NA
Concentration Recovered		2.29	NA	2.08	NA	NA	1.54	NA	NA	2.13	NA
Amount Spiked		2.00	NS	2.00	NS	NS	2.00	NS	NS	2.00	NS
Percent Recovery		115%	NA	104%	NA	NA	77%	NA	NA	107%	NA
Standard Reference Material											
Certified value 1646		NC ^(d)	11.6	0.36	76	18	0.063	32	28.2	NC	138
		NC	±1.3	±0.07	±3	±3	±0.012	±3	±1.8	NC	±6
1646	1	NA	11.4	0.42	86	19.3	0.074	33.6	27.3	NA	127
1646	2	NA	11.2	0.32	70.6	20.2	0.066	29.4	29.6	NA	138
Certified value BEST-1		NC	NC	NC	NC	NC	0.092 ±0.009	NC	NC	NC	NC
BEST-1	2	NA	NA	NA	NA	NA	0.090	NA	NA	NA	NA
Analytical Replicates											
C-SPB, Replicate 1	1	0.29	12.0	0.24	160	53.7	0.336	97.3	31.4	0.22	119
C-SPB, Replicate 2	1	0.30	14.0	0.24	173	53.1	0.348	99.5	24.8	0.22	125
RPD		3%	15%	0%	8%	1%	4%	2%	23% ^(e)	0%	5%
I-Stat		0.02	0.08	0.00	0.04	0.01	0.02	0.01	0.12	0.00	0.02

TABLE A.20. (contd)

Sediment Treatment	Batch	Metals (mg/kg dry weight)									
		Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
<u>Analytical Replicates</u>											
QC Sample, Replicate 1	2	0.10	10.0	0.21	202	43.2	0.029	94.7	10.7	0.17	92.4
QC Sample, Replicate 2	2	0.10	9.0	0.20	174	39.3	0.073	98.1	11.9	0.17	95.3
QC Sample, Replicate 3	2	0.10	11.2	0.21	201	41.1	0.071	99.9	9.2	0.17	95.1
RSD		0%	11%	3%	8%	5%	43%(e)	3%	13%	NA	2%

- (a) NA Not applicable.
 (b) U Undetected at or above detection limit.
 (c) NS Not spiked.
 (d) NC Not certified.
 (e) Value exceeds relative precision goal of ≤20%.

TABLE A.21. Sediment Butyltin Results, Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Batch</u>	<u>Surrogate Percent Recovery</u>	<u>Butyltins ($\mu\text{g/kg}$ dry weight)</u>	
			<u>Tri-butyltin</u>	<u>Di-butyltin</u>
Target DL ^(a)			10	10
<u>January 1993</u>				
OBM COMP	1	113	8.0 U ^(b)	2.2 B ^(c) J ^(d)
C-SB	1	78	8.0 U	2.0 BJ
C-WB	1	81	8.0 U	5.0 U
C-SPB, Replicate 1	1	100	3.8 J	5.0 U
C-SPB, Replicate 2	1	107	3.8 J	5.0 U
<u>January 1994</u>				
OBM COMP	2	80	0.40 U	0.65 U
R-OS	3	99	0.40 U	0.65 U
R-BF	3	102	3.82	3.38
R-AM	3	104	0.40 U	0.65 U
<u>October 1994</u>				
OBM COMP	4	94	0.48 U	0.56 U
R-OS, Replicate 1	4	98	0.67	0.56 U
R-OS, Replicate 2	4	103	0.54	0.56 U
R-OS, Replicate 3	4	98	0.66	0.56 U
R-BF	4	104	2.27	2.11
R-AM	4	93	0.48 U	0.56 U

(a) DL Detection limit.

(b) U Undetected at or above detection limit.

(c) B Analyte detected in sample at less than five times the value in associated method blank.

(d) J Analyte detected below method detection limit (MDL), but above instrument detection limit (IDL).

TABLE A.22. Quality Control Data for Sediment Butyltin Results,
Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Batch</u>	<u>Surrogate Percent Recovery</u>	<u>Butyltins (μg/kg dry weight)</u>	
			<u>Tri-butyltin</u>	<u>Di-butyltin</u>
<u>Method Blank</u>				
Blank	1	110	8.0 U ^(a)	4.3 J ^(b)
Blank	2	91	0.40 U	0.65 U
Blank	3	100	0.40 U	0.65
Blank	4	101	0.55	0.56 U
<u>Matrix Spike</u>				
C-WB	1	81	8.0 U	5.0 U
C-WB MS		98	27.4	26.8
Concentration Recovered		NA ^(c)	27.4	26.8
Amount Spiked		NS ^(d)	31.5	31.5
Percent Recovery		NA	87%	85%
C-WB	1	81	8.0 U	5.0 U
C-WB MSD		107	29.3	26.1
Concentration Recovered		NA	29.3	26.1
Amount Spiked		NS	31.3	31.3
Percent Recovery		NA	94%	83%
RPD		NA	7%	2%
I-Stat		NA	0.04	0.01
QC Sample	2	97	8.52	7.48
QC Sample, MS		90	64.6	44.0
Concentration Recovered		NA	56.1	36.5
Amount Spiked		NS	43.9	43.9
Percent Recovery		NA	128% ^(e)	83%
R-AM	3	104	0.40 U	0.65 U
R-AM, MS		103	31.9	31.7
Concentration Recovered		NA	31.9	31.7
Amount Spiked		NS	30.9	30.9
Percent Recovery		NA	103%	103%
QC Sample	4	92	0.49	0.56 U
QC Sample, MS		85	45.1	26.6
Concentration Recovered		NA	44.6	26.6
Amount Spiked		NS	50.0	50.0
Percent Recovery		NA	89%	53%

TABLE A.22. (contd)

<u>Sediment Treatment</u>	<u>Batch</u>	<u>Surrogate Percent Recovery</u>	<u>Butyltins (μg/kg dry weight)</u>	
			<u>Tri-butyltin</u>	<u>Di-butyltin</u>
<u>Standard Reference Material</u>				
Certified Value PACS-1		NA	1270	1160
		NA	±220	±180
PACS-1	1	102	798 ^(f)	827
PACS-1	2	99	1110	1210
PACS-1	3	103	1060	659 ^(f)
PACS-1	4	96	897	1080
<u>Analytical Replicates</u>				
C-SPB, Replicate 1	1	100	3.8 J	5.0 U
C-SPB, Replicate 2	1	107	3.8 J	5.0 U
RPD		NA	0%	NA
I-Stat		NA	0.00	NA
QC Sample, Replicate 1	2	107	9.32	9.30
QC Sample, Replicate 2	2	91	7.48	7.74
QC Sample, Replicate 3	2	100	8.98	8.11
RSD		NA	11%	10%
QC Sample, Replicate 1	3	91	0.40 U	0.65 U
QC Sample, Replicate 2	3	94	0.40 U	0.65 U
QC Sample, Replicate 3	3	105	0.40 U	0.65 U
RSD		NA	NA	NA
R-OS, Replicate 1	4	98	0.67	0.56 U
R-OS, Replicate 2	4	103	0.54	0.56 U
R-OS, Replicate 3	4	98	0.66	0.56 U
RSD		NA	12%	NA

(a) U Undetected at or above detection limit.

(b) J Analyte detected below method detection limit (MDL), but above instrument detection limit (IDL).

(c) NA Not applicable.

(d) NS Not spiked.

(e) Outside quality control criteria (40%-120%) for matrix spike recoveries.

(f) Outside quality control criteria ($\pm 30\%$) for SRMs.

APPENDIX B

BIOASSAY RESULTS FOR 10-DAY SOLID-PHASE, STATIC TEST
AND 96-HOUR REFERENCE TOXICANT TEST FOR *RHEPOXYNIUS abronius*

TABLE B.1. Test Results for 10-Day *R. abronius* Solid-Phase, Static Test, Older Bay Mud Study

Sediment Treatment	Replicate	R. abronius			Mean Proportion Surviving	Standard Deviation
		Live ^(a)	Dead or Missing	Proportion Surviving		
January 1993						
OBM COMP	1	17	3	0.85	0.85	0.06
OBM COMP	2	18	2	0.90		
OBM COMP	3	15	5	0.75		
OBM COMP	4	17	3	0.85		
OBM COMP	5	18	2	0.90		
C-WB	1	19	1	0.95	0.98	0.03
C-WB	2	20	0	1.00		
C-WB	3	20	0	1.00		
C-WB	4	20	0	1.00		
C-WB	5	19	1	0.95		
January 1994						
OBM COMP	1	14	6	0.70	0.72	0.08
OBM COMP	2	13	7	0.65		
OBM COMP	3	17	3	0.85		
OBM COMP	4	13	7	0.65		
OBM COMP	5	15	5	0.75		
R-OS	1	17	3	0.85	0.89	0.07
R-OS	2	19	1	0.95		
R-OS	3	19	1	0.95		
R-OS	4	16	4	0.80		
R-OS	5	18	2	0.90		
R-BF	1	17	3	0.85	0.94	0.05
R-BF	2	19	1	0.95		
R-BF	3	20	0	1.00		
R-BF	4	19	1	0.95		
R-BF	5	19	1	0.95		
R-AM	1	20	0	1.00	0.99	0.02
R-AM	2	20	0	1.00		
R-AM	3	20	0	1.00		
R-AM	4	20	0	1.00		
R-AM	5	19	1	0.95		
C-WB	1	20	0	1.00	0.98	0.03
C-WB	2	19	1	0.95		
C-WB	3	20	0	1.00		
C-WB	4	20	0	1.00		
C-WB	5	19	1	0.95		
C-SB	1	18	2	0.90	0.93	0.07
C-SB	2	18	2	0.90		
C-SB	3	20	0	1.00		
C-SB	4	17	3	0.85		
C-SB	5	20	0	1.00		
October 1994						
OBM COMP	1	13	7	0.65	0.68	0.10
OBM COMP	2	15	5	0.75		
OBM COMP	3	16	4	0.80		
OBM COMP	4	13	7	0.65		
OBM COMP	5	11	9	0.55		

TABLE B.1. (contd)

Sediment Treatment	Replicate	<i>R. abronius</i>			Mean Proportion Surviving	Standard Deviation
		Live ^(a)	Dead or Missing	Proportion Surviving		
R-OS	1	19	1	0.95	0.94	0.05
R-OS	2	17	3	0.85		
R-OS	3	19	1	0.95		
R-OS	4	20	0	1.00		
R-OS	5	19	1	0.95		
R-BF	1	19	1	0.95	0.84	0.11
R-BF	2	17	3	0.85		
R-BF	3	15	5	0.75		
R-BF	4	19	1	0.95		
R-BF	5	14	6	0.70		
R-AM	1	17	3	0.85	0.97	0.07
R-AM	2	20	0	1.00		
R-AM	3	20	0	1.00		
R-AM	4	20	0	1.00		
R-AM	5	20	0	1.00		
C-WB	1	19	1	0.95	0.98	0.03
C-WB	2	19	1	0.95		
C-WB	3	20	0	1.00		
C-WB	4	20	0	1.00		
C-WB	5	20	0	1.00		
C-SB	1	18	2	0.90	0.89	0.07
C-SB	2	17	3	0.85		
C-SB	3	17	3	0.85		
C-SB	4	20	0	1.00		
C-SB	5	17	3	0.85		

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

TABLE B.2. Water Quality Summary for 10-Day *R. abronius* Solid-Phase, Static Test, Older Bay Mud Study

Sediment Treatment	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)	
	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	≥6.0	NA ^(a)	28.0	32.0
<u>January 1993</u>								
OBM COMP	15.4	16.2	7.98	8.13	7.2	8.2	30.5	31.0
C-WB	15.4	16.2	7.88	8.17	7.1	8.1	30.0	31.0
<u>January 1994</u>								
OBM COMP	14.6	16.1	7.89	8.10	7.2	8.6	31.0	32.5 ^(b)
R-OS	14.6	15.9	7.49	8.01	7.4	8.4	31.0	32.0
R-BF	14.7	15.9	7.49	7.92	7.5	8.5	30.0	32.0
R-AM	14.6	15.7	7.49	7.97	7.3	8.5	31.0	32.0
C-WB	14.6	16.0	7.67	7.99	7.3	8.3	31.5	32.0
C-SB	14.6	15.9	7.76	8.40 ^(b)	7.2	8.4	31.0	32.0
<u>October 1994</u>								
OBM COMP	15.0	15.8	7.98	8.18	7.6	8.0	31.0	33.0 ^(b)
R-OS	15.0	15.9	7.94	8.11	7.5	7.9	31.0	33.0 ^(b)
R-BF	15.1	15.8	7.86	8.09	7.4	7.8	31.0	32.5 ^(b)
R-AM	15.0	15.9	7.87	8.10	7.4	7.9	30.5	33.0 ^(b)
C-WB	15.0	15.9	7.80	8.09	7.3	7.9	31.0	32.5 ^(b)
C-SB	15.0	15.9	7.81	8.19	7.3	7.9	31.0	33.0 ^(b)

(a) NA Not applicable.

(b) Data point out of range.

TABLE B.3. Ammonia Measurements in Overlying Water for 10-Day
R. abronius Solid-Phase, Static Test, Older Bay Mud Study

Sediment Treatment	Ammonia (mg/L)				
	Day 0	Day 1	Day 3	Day 7	Day 10
<u>January 1994</u>					
OBM COMP	1.11	1.96	2.14	3.34	1.32
R-OS	0.65	1.09	0.272	1.48	1.42
R-BF	0.78	1.43	1.74	0.700	1.23
R-AM	0.42	0.493	0.261	0.896	1.19
C-WB	0.82	0.947	0.802	1.31	1.45
C-SB	0.76	1.94	0.844	1.14	1.30
<u>October 1994</u>					
OBM COMP	0.734	0.639	0.723	1.13	0.380
R-OS	0.447	0.396	0.579	1.12	0.566
R-BF	2.71	2.83	2.60	1.90	0.630
R-AM	0.140	0.180	0.177	0.350	0.295
C-WB	0.347	0.330	0.778	1.39	0.564
C-SB	1.67	1.61	1.23	1.08	0.597

TABLE B.4. Water Quality Measurements of Interstitial Water for 10-Day *R. abronius* Solid-Phase, Static Test, Older Bay Mud Study

Sediment Treatment	pH		Salinity (o/oo)		Porewater Ammonia (mg/L)	
	Min	Max	Min	Max	Day 0	Day 10
<u>January 1994</u>						
OBM COMP	8.08	8.30	32.0	32.0	2.53	2.49
R-OS	7.87	8.00	32.0	32.0	3.61	1.05
R-BF	7.61	7.96	32.0	32.0	3.38	0.66
R-AM	7.92	7.93	32.0	32.0	3.25	1.03
C-WB	7.71	7.71	32.0	32.0	4.99	NI ^(a)
C-SB	7.82	7.85	32.0	32.0	4.22	1.07

	pH		Salinity (o/oo)		Porewater Ammonia (mg/L)	
	Day 0	Day 10	Day 0	Day 10	Day 0	Day 10
<u>October 1994</u>						
OBM COMP	8.31	8.25	32.0	31.5	1.14	0.637
R-OS	7.95	7.76	32.0	32.5	1.18	1.01
R-BF	7.45	7.46	31.5	32.0	6.98	2.11
R-AM	7.84	7.47	32.0	32.0	0.476	0.156
C-WB	7.79	7.60	31.0	32.0	2.20	2.71
C-SB	7.71	7.30	32.0	32.0	2.46	1.08

(a) NI Not initiated; not enough water in sediment sample.

TABLE B.5. Test Results for 96-Hour *R. abronius* Cadmium Reference Toxicant Test, Older Bay Mud Study

Cadmium Concentration (mg/L)	Replicate	<i>R. abronius</i>			Mean Proportion Surviving	Standard Deviation
		Live ^(a)	Dead or Missing	Proportion Surviving		
<u>January 1993</u>						
0	1	18	2	0.90	0.92	0.03
0	2	18	2	0.90		
0	3	19	1	0.95		
0.5	1	13	7	0.65	0.60	0.05
0.5	2	12	8	0.60		
0.5	3	11	9	0.55		
1	1	3	17	0.15	0.22	0.06
1	2	5	15	0.25		
1	3	5	15	0.25		
2	1	3	17	0.15	0.05	0.09
2	2	0	20	0.00		
2	3	0	20	0.00		
4	1	0	20	0.00	0.00	0.00
4	2	0	20	0.00		
4	3	0	20	0.00		
<u>January 1994</u>						
0.00	1	19	1	0.95	0.93	0.08
0.00	2	17	3	0.85		
0.00	3	20	0	1.00		
0.38	1	15	5	0.75	0.50	0.25
0.38	2	10	10	0.50		
0.38	3	5	15	0.25		
0.75	1	11	9	0.55	0.63	0.10
0.75	2	12	8	0.60		
0.75	3	15	5	0.75		
1.50	1	5	15	0.25	0.18	0.06
1.50	2	3	17	0.15		
1.50	3	3	17	0.15		
3.00	1	0	20	0.00	0.00	0.00
3.00	2	0	20	0.00		
3.00	3	0	20	0.00		
<u>October 1994</u>						
0.00	1	19	1	0.95	0.45	0.35
0.25	1	15	5	0.75		
0.50	1	12	8	0.60		
1.00	1	10	10	0.50		
1.50	1	5	15	0.25		
2.00	1	2	18	0.10		
4.00	1	0	20	0.00		

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

TABLE B.6. Water Quality Summary for 96-Hour *R. abronius* Cadmium Reference Toxicant Test, Older Bay Mud Study

Cadmium Concentration (mg/L)	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)	
	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	≥6.0	NA ^(a)	28.0	32.0
<u>January 1993</u>								
0	15.6	16.2	7.92	8.08	7.1	8.0	30.5	30.5
0.5	15.6	16.1	7.93	8.07	7.1	7.9	30.5	30.5
1	15.6	15.9	7.82	8.04	7.1	8.0	30.5	30.5
2	15.6	16.1	7.87	8.01	7.2	7.9	30.5	30.5
4	15.6	16.1	7.80	7.93	7.2	8.0	30.5	30.5
<u>January 1994</u>								
0.00	15.1	15.5	7.83	7.96	7.4	8.4	31.5	32.0
0.38	15.1	15.5	7.83	7.92	7.3	8.6	31.5	32.0
0.75	15.1	15.5	7.84	7.93	7.6	8.6	31.0	32.0
1.50	15.1	15.4	7.79	7.91	7.7	8.7	31.5	32.0
3.00	15.2	15.5	7.79	7.85	7.4	8.7	31.5	32.0
<u>October 1994</u>								
0.00	15.1	15.5	7.78	8.09	7.7	7.8	31.5	32.0
0.25	15.2	15.5	7.73	8.09	7.7	8.0	31.5	32.0
0.50	15.2	15.4	7.78	8.08	7.8	8.1	31.5	32.0
1.00	15.1	15.5	7.77	8.05	7.8	8.0	31.5	32.0
1.50	15.2	15.5	7.71	8.04	7.9	8.1	31.5	32.0
2.00	15.1	15.5	7.62	8.01	7.8	8.0	31.5	32.0
4.00	15.1	15.5	7.60	7.96	7.9	8.0	31.5	32.0

(a) NA Not applicable.

TABLE B.7. Test Results for 96-Hour *R. abronius* Ammonia Reference Toxicant Test, Older Bay Mud Study

Ammonia Concentration mg/L	Live ^(a)	Dead or Missing	Proportion Surviving
0	20	0	1.00
10	20	0	1.00
20	20	0	1.00
40	19	1	0.95
80	13	7	0.65
120	5	15	0.25
160	0	20	0.00

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

TABLE B.8. Water Quality Summary for 96-Hour *R. abronius* Ammonia Reference Toxicant Test, Older Bay Mud Study

Ammonia Concentration mg/L	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)		Ammonia (mg/L)	
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	6.0	NA ^(a)	28.0	32.0	NE ^(b)	NE
0	15.0	15.6	7.71	8.05	7.7	7.8	31.5	32.0	0.212	0.419
10	15.0	15.6	7.65	8.04	7.8	8.0	31.5	32.0	9.15	10.5
20	14.9	15.6	7.65	8.05	7.7	8.0	31.0	32.0	17.3	21.7
40	14.9	15.5	7.60	8.08	7.8	8.1	31.0	32.0	34.7	40.0
80	14.9	15.5	7.59	8.06	7.8	8.0	31.5	32.0	66.2	78.6
120	15.0	15.5	7.60	8.03	7.8	8.1	31.5	32.0	99.5	126
160	15.0	15.5	7.45	8.01	7.8	8.1	31.5	32.0	132	179

(a) NA Not applicable.

(b) NE Not established.

APPENDIX C

BIOASSAY RESULTS FOR 10-DAY SOLID-PHASE, FLOW-THROUGH TEST
WITH *MACOMA nasuta* AND *NEPHTYS caecoides*

TABLE C.1. Test Results for 10-Day *M. nasuta* Solid-Phase, Flow-Through Test, Older Bay Mud Study

Sediment Treatment	Replicate	M. nasuta		Proportion Surviving	Mean	Standard Deviation
		Live ^(a)	Dead or Missing		Proportion Surviving	
<u>January 1994</u>						
OBM COMP	1	25	0	1.00		
OBM COMP	2	25	0	1.00		
OBM COMP	3	24	1	0.96		
OBM COMP	4	25	0	1.00		
OBM COMP	5	24	1	0.96	0.98	0.02
R-OS	1	25	0	1.00		
R-OS	2	25	0	1.00		
R-OS	3	25	0	1.00		
R-OS	4	25	0	1.00		
R-OS	5	25	0	1.00	1.00	0.00
R-BF	1	25	0	1.00		
R-BF	2	25	0	1.00		
R-BF	3	25	0	1.00		
R-BF	4	25	0	1.00		
R-BF	5	25	0	1.00	1.00	0.00
R-AM	1	25	0	1.00		
R-AM	2	25	0	1.00		
R-AM	3	25	0	1.00		
R-AM	4	NI ^(b)	NI	NA ^(c)		
R-AM	5	25	0	1.00	1.00	0.00
C-SB	1	20	0	1.00		
C-SB	2	20	0	1.00		
C-SB	3	20	0	1.00		
C-SB	4	20	0	1.00		
C-SB	5	20	0	1.00	1.00	0.00
C-NE	1	20	0	1.00		
C-NE	2	20	0	1.00		
C-NE	3	20	0	1.00		
C-NE	4	20	0	1.00		
C-NE	5	20	0	1.00	1.00	0.00

(a) Survival based on initial exposure of either 20 or 25 organisms per replicate.

(b) NI Not initiated; insufficient organisms to test all five replicates.

(c) NA Not applicable.

TABLE C.2. Test Results for 10-Day *N. caecoides* Solid-Phase, Flow-Through Test, Older Bay Mud Study

		<i>N. caecoides</i>			Mean	
Sediment Treatment	Replicate	Live ^(a)	Dead or Missing	Proportion Surviving	Proportion Surviving	Standard Deviation
<u>January 1994</u>						
OBM COMP	1	6	14	0.30		
OBM COMP	2	11	9	0.55		
OBM COMP	3	4	16	0.20		
OBM COMP	4	11	9	0.55		
OBM COMP	5	6	14	0.30	0.38	0.16
R-OS	1	19	1	0.95		
R-OS	2	20	0	1.00		
R-OS	3	19	1	0.95		
R-OS	4	18	2	0.90		
R-OS	5	20	0	1.00	0.96	0.04
R-BF	1	17	3	0.85		
R-BF	2	19	1	0.95		
R-BF	3	17	3	0.85		
R-BF	4	18	2	0.90		
R-BF	5	20	0	1.00	0.91	0.07
R-AM	1	19	1	0.95		
R-AM	2	20	0	1.00		
R-AM	3	19	1	0.95		
R-AM	4	NI ^(b)	NI	NA ^(c)		
R-AM	5	19	1	0.95	0.96	0.03
C-NE	1	18	2	0.90		
C-NE	2	20	0	1.00		
C-NE	3	18	2	0.90		
C-NE	4	18	2	0.90		
C-NE	5	20	0	1.00	0.94	0.05
C-SB	1	20	0	1.00		
C-SB	2	19	1	0.95		
C-SB	3	19	1	0.95		
C-SB	4	19	1	0.95		
C-SB	5	19	1	0.95	0.96	0.02

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

(b) NI Not initiated; insufficient organisms for all five replicates.

(c) NA Not applicable.

TABLE C.3. Water Quality Summary for 10-Day *M. nasuta*/*N. caecoides* Solid-Phase, Flow-Through Test, Older Bay Mud Study

Sediment Treatment	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)	
	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	5.0	NA (a)	28.0	32.0
<u>January 1994</u>								
OBM COMP	14.3	15.6	7.32	7.87	7.4	8.6	30.5	32.0
R-OS	14.2	15.5	7.40	7.90	7.4	8.5	30.5	32.0
R-BF	14.2	15.5	7.32	7.89	7.3	8.5	30.5	32.0
R-AM	14.3	15.6	7.43	8.00	7.5	8.5	31.0	32.0
C-NE	14.3	15.6	7.34	8.20	7.1	8.5	30.5	32.0
C-SB	14.2	15.6	7.42	7.89	7.2	8.4	30.5	32.0

(a) NA Not applicable.

TABLE C.4. Ammonia Measurements in Overlying Water for 10-Day *M. nasuta*/
N. caecoides Solid-Phase, Flow-Through Test, Older Bay Mud Study

Sediment Treatment	Ammonia (mg/L)			
	Day 1	Day 3	Day 7	Day 10
<u>January 1994</u>				
OBM COMP	0.134	0.159	0.372	0.177
R-OS	0.149	0.348	0.352	0.232
R-BF	0.177	0.143	0.352	0.178
R-AM	0.118	0.300	0.364	0.125
C-NE	0.137	0.150	0.361	0.133
C-SB	0.127	0.358	0.373	0.144

APPENDIX D

BIOASSAY RESULTS FOR 28-DAY SOLID-PHASE,
FLOW-THROUGH TEST WITH *MACOMA nasuta*

TABLE D.1. Test Results for 28-Day *M. nasuta* Solid-Phase, Flow-Through Test,
Older Bay Mud Study

Sediment Treatment	Replicate	<i>M. nasuta</i>		Proportion Surviving	Mean	Standard Deviation
		Live ^(a)	Dead or Missing		Proportion Surviving	
<u>January 1993</u>						
OBM COMP	1	29	1	0.97		
OBM COMP	2	30	0	1.00		
OBM COMP	3	28	2	0.93		
OBM COMP	4	30	0	1.00		
OBM COMP	5	29	1	0.97	0.97	0.03
C-SB	1	30	0	1.00		
C-SB	2	30	0	1.00		
C-SB	3	28	2	0.93		
C-SB	4	30	0	1.00		
C-SB	5	30	0	1.00	0.99	0.03
<u>January 1994</u>						
OBM COMP	1	25	0	1.00		
OBM COMP	2	22	3	0.88		
OBM COMP	3	25	0	1.00		
OBM COMP	4	22	3	0.88		
OBM COMP	5	24	1	0.96	0.94	0.06
R-OS	1	25	0	1.00		
R-OS	2	23	2	0.92		
R-OS	3	22	3	0.88		
R-OS	4	25	0	1.00		
R-OS	5	25	0	1.00	0.96	0.06
R-BF	1	25	0	1.00		
R-BF	2	25	0	1.00		
R-BF	3	23	2	0.92		
R-BF	4	22	3	0.88		
R-BF	5	23	2	0.92	0.94	0.05
R-AM	1	23	2	0.92		
R-AM	2	24	1	0.96		
R-AM	3	23	2	0.92		
R-AM	4	25	0	1.00		
R-AM	5	23	2	0.92	0.94	0.04

TABLE D.1. (contd)

Sediment Treatment	Replicate	<i>M. nasuta</i>		Proportion Surviving	Mean Proportion Surviving	Standard Deviation
		Live ^(a)	Dead or Missing			
C-SB	1	23	2	0.92		
C-SB	2	22	3	0.88		
C-SB	3	24	1	0.96		
C-SB	4	24	1	0.96		
C-SB	5	21	4	0.84	0.91	0.05

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

TABLE D.2. Water Quality Summary for 28-Day *M. nasuta* Solid-Phase Flow-Through Test, Older Bay Mud Study

Sediment Treatment	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)	
	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	≥6.0	NA ^(a)	28.0	32.0
<u>January 1993</u>								
OBM COMP	15.1	17.3 ^(b)	7.73	7.92	7.4	9.7	30.0	31.0
C-SB	15.1	17.2 ^(b)	7.62	7.91	7.1	9.2	30.0	31.0
<u>January 1994</u>								
OBM COMP	11.0 ^(b)	16.4	7.56	7.93	7.4	8.9	30.0	32.0
R-OS	10.9 ^(b)	16.5	7.56	7.94	7.4	8.9	30.0	32.0
R-BF	11.0 ^(b)	16.5	7.53	7.93	7.3	8.8	30.0	32.0
R-AM	11.0 ^(b)	16.5	7.52	7.93	7.4	8.8	30.0	32.0
C-SB	10.9 ^(b)	16.5	7.51	7.90	7.3	8.8	30.0	32.0

(a) NA Not applicable.

(b) Data point out of range.

APPENDIX E

BIOASSAY RESULTS FOR 48-HOUR SUSPENDED-PARTICULATE-PHASE TEST
AND 48-HOUR REFERENCE TOXICANT TEST
FOR *MYTILUS galloprovincialis*

TABLE E.1. Test Results for 48-Hour Larval *M. galloprovincialis* Suspended-Particulate-Phase Test, Older Bay Mud Study

Sediment Treatment	Concentration (Percent SPP)	Replicate	Mean Stocking Density		Number Larvae Surviving ^(a)	Proportion Surviving	Mean Proportion Surviving		Standard Deviation	Number Normal D-cell Larvae		Proportion Normal	Mean Proportion Normal		Standard Deviation
January 1993															
OBM COMP	0	1	256	286		1.00 ^(b)				272	1.00 ^(b)				
OBM COMP	0	2	256	313		1.00 ^(b)				303	1.00 ^(b)				
OBM COMP	0	3	256	259		1.00 ^(b)	1.00		0.00	250	0.98	0.99	0.01		
OBM COMP	10	1	256	271		1.00 ^(b)				264	1.00 ^(b)				
OBM COMP	10	2	256	252		0.98				245	0.96				
OBM COMP	10	3	256	252		0.98	0.99		0.01	248	0.97	0.98	0.02		
OBM COMP	50	1	256	234		0.91				226	0.88				
OBM COMP	50	2	256	270		1.00 ^(b)				261	1.00 ^(b)				
OBM COMP	50	3	256	268		1.00 ^(b)	0.97		0.05	259	1.00 ^(b)	0.96	0.07		
OBM COMP	100	1	256	268		1.00 ^(b)				259	1.00 ^(b)				
OBM COMP	100	2	256	288		1.00 ^(b)				282	1.00 ^(b)				
OBM COMP	100	3	256	263		1.00 ^(b)	1.00		0.00	254	0.99	1.00	0.01		
January 1994															
OBM COMP	0	1	238	277		1.00 ^(b)				269	1.00 ^(b)				
OBM COMP	0	2	238	256		1.00 ^(b)				251	1.00 ^(b)				
OBM COMP	0	3	238	247		1.00 ^(b)				242	1.00 ^(b)				
OBM COMP	0	4	238	254		1.00 ^(b)				251	1.00 ^(b)				
OBM COMP	0	5	238	217		0.91	0.98		0.04	214	0.90	0.98	0.05		
OBM COMP	10	1	238	224		0.94				222	0.93				
OBM COMP	10	2	238	229		0.96				227	0.95				
OBM COMP	10	3	238	223		0.94				220	0.92				
OBM COMP	10	4	238	250		1.00 ^(b)				236	0.99				
OBM COMP	10	5	238	227		0.95	0.96		0.03	225	0.95	0.95	0.03		

TABLE E.1. (contd)

Sediment Treatment	Concentration (Percent SPP)	Replicate	Mean Stocking Density	Number Larvae Surviving ^(a)	Mean			Number			Mean		
					Proportion Surviving	Proportion Surviving	Proportion Surviving	Standard Deviation	D-cell Larvae	Proportion Normal	Proportion Normal	Standard Deviation	Standard Deviation
OBM COMP	50	1	238	196	0.82				194	0.82			
OBM COMP	50	2	238	232	0.97				230	0.97			
OBM COMP	50	3	238	214	0.90				212	0.89			
OBM COMP	50	4	238	204	0.86				203	0.85			
OBM COMP	50	5	238	260	1.00 ^(b)	0.91		0.08	253	1.00 ^(b)	0.91		0.08
OBM COMP	100	1	238	233	0.98				227	0.95			
OBM COMP	100	2	238	228	0.96				220	0.92			
OBM COMP	100	3	238	248	1.00 ^(b)				239	1.00			
OBM COMP	100	4	238	244	1.00 ^(b)				240	1.00 ^(b)			
OBM COMP	100	5	238	226	0.95	0.98		0.02	221	0.93	0.96		0.04

(a) Sum total of normal D-cell, abnormal D-cell, and developmentally delayed larvae.

(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 calculation and statistical analysis.

TABLE E.2. Water Quality Summary for 48-Hour Larval *M. galloprovincialis* Suspended-Particulate-Phase Test
Older Bay Mud Study

Sediment Treatment	Concentration (Percent SPP)	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)		Ammonia (mg/L)
		Min	Max	Min	Max	Min	Max	Min	Max	
Acceptable Range		14.0	18.0	7.30	8.30	4.0	NA ^(a)	28.0	32.0	NE ^(c)
<u>January 1993</u>										
OBM COMP	0	16.1	16.4	7.87	8.09	7.4	8.4	30.0	30.0	NM ^(d)
OBM COMP	10	16.2	16.5	7.91	8.12	7.6	8.2	30.0	30.0	NM
OBM COMP	50	16.1	16.6	8.00	8.11	7.6	8.1	30.0	30.0	NM
OBM COMP	100	16.2	16.5	8.09	8.12	7.6	8.0	30.0	30.0	NM
<u>January 1994</u>										
OBM COMP	0	15.4	15.6	7.74	7.99	7.2	8.2	31.5	32.0	1.23
OBM COMP	10	15.3	15.7	7.77	8.03	7.2	8.3	31.0	32.0	NM
OBM COMP	50	15.4	15.6	7.88	8.01	7.3	8.2	30.5	31.0	NM
OBM COMP	100	15.4	15.6	7.88	8.09	7.2	8.4	30.0	30.0	1.95

(a) Average of ammonia measurements taken during testing.

(b) NA Not applicable.

(c) NE Not established.

(d) NM Not measured.

TABLE E.3. Test Results for 48-Hour Larval *M. galloprovincialis* Copper Reference Toxicant Test, Older Bay Mud Study

Copper Concentration (µg/L)	Replicate	Mean Stocking Density	Number Larvae Surviving	Proportion Surviving	Mean Proportion Surviving	Standard Deviation	Number Normal		Mean Proportion Normal	Standard Deviation
							D-cell Larvae	Proportion Normal		
January 1993										
Brine	1	279	270	0.97			249	0.89		
	2	279	284	1.00 ^(a)			268	0.96		
	3	279	287	1.00 ^(a)	0.99	0.02	271	0.97	0.94	0.04
0	1	279	322	1.00 ^(a)			309	1.00 ^(a)		
0	2	279	279	1.00 ^(a)			265	0.95		
0	3	279	279	1.00 ^(a)	1.00 ^(a)	0.00	268	0.96	0.97	0.03
1	1	279	301	1.00 ^(a)			288	1.00 ^(a)		
1	2	279	278	1.00 ^(a)			269	0.96		
1	3	279	274	0.98	0.99 ^(a)	0.01	264	0.95	0.97	0.03
4	1	279	280	1.00 ^(a)			270	0.97		
4	2	279	282	1.00 ^(a)			269	0.96		
4	3	279	269	0.96	0.99 ^(a)	0.02	255	0.91	0.95	0.03
16	1	279	116	0.42			2	0.01		
16	2	279	125	0.45			4	0.01		
16	3	279	180	0.65	0.51	0.12	0	0.00	0.01	0.01
64	1	279	2	0.01			0	0.00		
64	2	279	1	0.00			0	0.00		
64	3	279	0	0.00	0.00	0.01	0	0.00	0.00	0.00
256	1	279	0	0.00			0	0.00		
256	2	279	0	0.00			0	0.00		
256	3	279	0	0.00	0.00	0.00	0	0.00	0.00	0.00

TABLE E.3. (contd)

Copper Concentration (µg/L)	Replicate	Mean Stocking Density	Number Larvae Surviving	Proportion Surviving	Mean Proportion Surviving	Standard Deviation	Number Normal D-cell Larvae	Proportion Normal	Mean Proportion Normal	Standard Deviation
January 1994										
Site water	1	238	269	1.00 ^(a)			266	1.00 ^(a)		
Site water	2	238	287	1.00 ^(a)			283	1.00 ^(a)		
Site water	3	238	285	1.00 ^(a)	1.00	0.00	279	1.00 ^(a)	1.00	0.00
Brine	1	238	303	1.00 ^(a)			297	1.00 ^(a)		
Brine	2	238	235	0.99			231	0.97		
Brine	3	238	263	1.00 ^(a)	1.00	0.01	259	1.00 ^(a)	0.99	0.02
0.00	1	238	238	1.00			232	0.97		
0.00	2	238	242	1.00 ^(a)			241	1.00 ^(a)		
0.00	3	238	280	1.00 ^(a)	1.00	0.00	277	1.00 ^(a)	0.99	0.01
1.00	1	238	244	1.00 ^(a)			240	1.00 ^(a)		
1.00	2	238	321	1.00 ^(a)			318	1.00 ^(a)		
1.00	3	238	272	1.00 ^(a)	1.00	0.00	271	1.00 ^(a)	1.00	0.00
4.00	1	238	241	1.00 ^(a)			236	0.99		
4.00	2	238	268	1.00 ^(a)			265	1.00 ^(a)		
4.00	3	238	257	1.00 ^(a)	1.00	0.00	247	1.00 ^(a)	1.00	0.00
16.0	1	238	88	0.37			0	0.00		
16.0	2	238	69	0.29			0	0.00		
16.0	3	238	54	0.23	0.30	0.07	0	0.00	0.00	0.00
64.0	1	238	6	0.03			0	0.00		
64.0	2	238	12	0.05			0	0.00		
64.0	3	238	15	0.06	0.05	0.02	0	0.00	0.00	0.00

(a) 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.

TABLE E.4. Water Quality Summary for 48-Hour *M. galloprovincialis* Reference Toxicant Test, Older Bay Mud Study

Copper Concentration ($\mu\text{g/L}$)	Temperature ($^{\circ}\text{C}$)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)	
	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	14.0	18.0	7.30	8.30	≥ 4.0	NA ^(a)	28.0	32.0
<u>January 1993</u>								
Brine	16.2	16.5	7.53	8.08	6.4	8.0	30.0	30.5
0	16.2	16.5	7.83	8.13	7.6	8.4	30.0	30.0
1	16.2	16.5	7.87	8.12	7.5	8.5	30.0	30.0
4	16.2	16.5	7.89	8.13	7.5	8.2	30.0	30.0
16	16.2	16.6	7.88	8.14	7.4	8.5	30.0	30.0
64	16.2	16.5	7.87	8.12	7.4	8.4	30.0	30.0
256	16.2	16.6	7.87	8.11	7.4	8.3	30.0	30.0
<u>January 1994</u>								
Site water	15.4	15.5	7.75	7.83	7.2	7.9	30.0	30.0
Brine	15.4	15.7	7.75	7.79	6.9	7.5	30.0	31.5
0	15.4	15.5	7.72	7.89	6.9	7.5	31.5	32.0
1	15.4	15.5	7.72	7.94	7.0	7.6	31.5	32.0
4	15.4	15.5	7.71	7.95	7.1	7.6	31.5	32.0
16	15.4	15.5	7.71	7.96	7.0	7.5	31.5	32.0
64	15.4	15.5	7.70	7.97	7.0	7.5	31.5	32.0

(a) NA Not applicable.

TABLE E.5. Test Results for 48-Hour Larval *M. galloprovincialis* Ammonia Reference Toxicant Test, Older Bay Mud Study

Ammonia Concentration (mg/L)	Replicate	Mean Stocking Density	Number Larvae Surviving	Proportion Surviving	Mean Proportion Surviving	Standard Deviation	Number Normal D-cell Larvae	Proportion Normal	Mean Proportion Normal	Standard Deviation
January 1994										
Site water	1	238	269	1.00 ^(a)			266	1.00 ^(a)		
	2	238	287	1.00 ^(a)			283	1.00 ^(a)		
	3	238	285	1.00 ^(a)	1.00	0.00	279	1.00 ^(a)	1.00	0.00
brine	1	238	303	1.00 ^(a)			297	1.00 ^(a)		
	2	238	235	0.99			231	0.97		
	3	238	263	1.00 ^(a)	1.00	0.01	259	1.00 ^(a)	0.99	0.02
0.0	1	238	238	1.00			232	0.97		
0.0	2	238	242	1.00 ^(a)			241	1.00 ^(a)		
0.0	3	238	280	1.00 ^(a)	1.00	0.00	277	1.00 ^(a)	0.99	0.01
1.05	1	238	251	1.00 ^(a)			246	1.00 ^(a)		
1.05	2	238	282	1.00 ^(a)			280	1.00 ^(a)		
1.05	3	238	224	0.94	0.98	0.03	220	0.92	0.97	0.04
7.48	1	238	253	1.00 ^(a)			1	0.00		
7.48	2	238	211	0.89			0	0.00		
7.48	3	238	255	1.00 ^(a)	0.96	0.07	0	0.00	0.00	0.00
16.64	1	238	296	1.00 ^(a)			0	0.00		
16.64	2	238	251	1.00 ^(a)			0	0.00		
16.64	3	238	264	1.00 ^(a)	1.00	0.00	0	0.00	0.00	0.00
60.56	1	238	87	0.37			0	0.00		
60.56	2	238	113	0.47			0	0.00		
60.56	3	238	46	0.19	0.34	0.14	0	0.00	0.00	0.00
105.4	1	238	9	0.04			0	0.00		
105.4	2	238	0	0.00			0	0.00		
105.4	3	238	0	0.00	0.01	0.02	0	0.00	0.00	0.00

(a) 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.

TABLE E.6. Water Quality Summary for 48-Hour Larval *M. galloprovincialis* Ammonia Toxicant Test, Older Bay Mud Study

Ammonia Concentration (mg/L)	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)		Ammonia (mg/L)	
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	14.0	18.0	7.30	8.30	4.0	NA ^(a)	28.0	32.0	NE ^(b)	NE
<u>January 1994</u>										
Site water	15.4	15.5	7.75	7.83	7.2	7.9	30.0	30.0	NM ^(c)	NM
Brine	15.4	15.8	7.83	7.97	6.9	7.9	30.0	32.0	NM	NM
0.0	15.5	15.8	7.70	7.86	6.8	7.9	31.0	32.0	NM	NM
1.05	15.4	15.5	7.68	7.84	7.2	8.0	31.5	32.0	0.80	1.90
7.48	15.4	15.4	7.64	7.85	7.2	8.0	31.0	32.0	6.6	9.7
16.64	15.3	15.5	7.65	7.86	7.2	7.9	30.0	31.0	14	21
60.56	15.4	15.5	7.57	7.85	7.2	8.0	28.5	30.0	46	81
105.4	15.4	15.5	7.48	7.78	7.2	8.0	28.0	28.0	89	123

(a) NA Not applicable.

(b) NE Not established.

(c) NM Not measured.

APPENDIX F

BIOASSAY RESULTS FOR 96-HOUR SUSPENDED-PARTICULATE-PHASE TEST
AND 96-HOUR REFERENCE TOXICANT TEST FOR *CITHARICHTHYS stigmaeus*

TABLE F.1. Test Results for 96-Hour *C. stigmaeus* Suspended-Particulate-Phase Test, Older Bay Mud Study

Sediment Treatment	Concentration (Percent SPP)	Replicate	<i>C. stigmaeus</i>		Proportion Surviving	Mean	Standard Deviation
			Live ^(a)	Dead or Missing		Proportion Surviving	
<u>January 1994</u>							
OBM COMP	0	1	10	0	1.00		
OBM COMP	0	2	10	0	1.00		
OBM COMP	0	3	10	0	1.00		
OBM COMP	0	4	10	0	1.00		
OBM COMP	0	5	10	0	1.00	1.00	0.00
OBM COMP	10	1	10	0	1.00		
OBM COMP	10	2	10	0	1.00		
OBM COMP	10	3	10	0	1.00		
OBM COMP	10	4	10	0	1.00		
OBM COMP	10	5	10	0	1.00	1.00	0.00
OBM COMP	50	1	10	0	1.00		
OBM COMP	50	2	10	0	1.00		
OBM COMP	50	3	10	0	1.00		
OBM COMP	50	4	9	1	0.90		
OBM COMP	50	5	10	0	1.00	0.98	0.04
OBM COMP	100	1	10	0	1.00		
OBM COMP	100	2	10	0	1.00		
OBM COMP	100	3	10	0	1.00		
OBM COMP	100	4	10	0	1.00		
OBM COMP	100	5	10	0	1.00	1.00	0.00

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

TABLE F.2. Summary of Mean Proportion Surviving 96-Hour *C. stigmaeus*
Suspended-Particulate-Phase Test, Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Concentration (Percent SPP)</u>	<u>Mean Proportion Surviving</u>
<u>January 1994</u>		
OBM COMP	0	1.00
OBM COMP	10	1.00
OBM COMP	50	0.98
OBM COMP	100	1.00

TABLE F.3. Water Quality Summary for 96-Hour *C. stigmaeus* Suspended-Particulate-Phase Test, Older Bay Mud Study

Sediment Treatment	Concentration (Percent SPP)	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)		Ammonia (mg/L)
		Min	Max	Min	Max	Min	Max	Min	Max	Average ^(a)
Acceptable Range		13.0	17.0	7.30	8.30	6.0	NA ^(b)	28.0	32.0	NE ^(c)
<u>January 1994</u>										
OBM COMP	0	13.9	14.9	7.68	7.96	7.8	8.6	31.0	31.5	NM ^(d)
OBM COMP	10	13.9	14.8	7.73	7.98	7.9	8.6	30.0	31.5	NM
OBM COMP	50	14.3	14.8	7.78	7.99	7.7	8.6	30.0	31.0	NM
OBM COMP	100	14.5	15.1	7.81	8.07	7.7	8.7	29.5	30.0	1.00

(a) Average of Day 4 measurements.

(b) NA Not applicable.

(c) NE Not established.

(d) NM Not measured.

TABLE F.4. Test Results for 96-Hour *C. stigmaeus* Copper Reference Toxicant Test, Older Bay Mud Study

Copper Concentration (mg/L)	Replicate	<i>C. stigmaeus</i>		Proportion Surviving	Mean Proportion Survival	Standard Deviation
		Live ^(a)	Dead or Missing			
<u>January 1994</u>						
0.00	1	10	0	1.00	1.00	0.00
0.00	2	10	0	1.00		
0.00	3	10	0	1.00		
0.50	1	10	0	1.00	0.97	0.06
0.50	2	10	0	1.00		
0.50	3	9	1	0.90		
1.00	1	0	10	0.00	0.37	0.55
1.00	2	1	9	0.10		
1.00	3	10	0	1.00		
1.50	1	0	10	0.00	0.13	0.23
1.50	2	0	10	0.00		
1.50	3	4	6	0.40		
2.00	1	0	10	0.00	0.03	0.06
2.00	2	1	9	0.10		
2.00	3	0	10	0.00		

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

TABLE F.5. Water Quality Summary for 96-Hour *C. stigmaeus* Copper Reference Toxicant Test, Older Bay Mud Study

Copper Concentration (mg/L)	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)	
	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	6.0	NA ^(a)	28.0	32.0
<u>January 1994</u>								
0.00	14.2	15.4	7.72	7.90	7.7	8.4	30.5	32.0
0.50	14.0	15.0	7.78	7.93	8.1	8.5	30.5	31.5
1.00	14.0	15.4	7.68	7.95	8.0	8.4	30.5	32.0
1.50	14.1	15.3	7.67	7.96	8.2	8.5	30.5	31.5
2.00	14.1	15.4	7.63	7.91	8.0	8.4	30.5	32.0

(a) NA Not applicable.

TABLE F.6. Test Results for 96-Hour *C. stigmaeus* Ammonia Reference Toxicant Test, Older Bay Mud Study

Ammonia Concentration (mg/L)	Replicate	<i>C. stigmaeus</i>		Proportion Surviving	Mean	Standard Deviation
		Live ^(a)	Dead or Missing		Proportion Survival	
<u>January 1994</u>						
0.350	1	10	0	1.00		
0.350	2	10	0	1.00		
0.350	3	10	0	1.00	1.00	0.00
12.9	1	10	0	1.00		
12.9	2	10	0	1.00		
12.9	3	10	0	1.00	1.00	0.00
17.1	1	10	0	1.00		
17.1	2	10	0	1.00		
17.1	3	10	0	1.00	1.00	0.00
21.7	1	9	1	0.90		
21.7	2	7	3	0.70		
21.7	3	8	2	0.80	0.80	0.10
25.8	1	2	8	0.20		
25.8	2	1	9	0.10		
25.8	3	5	5	0.50	0.27	0.21

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

TABLE F.7. Water Quality Summary for 96-Hour *C. stigmaeus* Ammonia Reference Toxicant Test, Older Bay Mud Study

Ammonia Concentration (mg/L)	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)		Ammonia (mg/L)	
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	6.0	NA ^(a)	28.0	32.0	NE ^(b)	NE
<u>January 1994</u>										
0.350	14.1	14.7	7.68	7.93	7.8	8.3	31.0	32.0	0.234	0.548
12.9	14.0	14.4	7.70	7.88	8.0	8.4	31.0	32.0	11.2	17.5
17.1	14.1	14.3	7.78	7.89	8.0	8.4	31.0	32.0	14.6	20.8
21.7	14.1	14.3	7.77	7.92	7.9	8.4	31.0	31.5	18.4	24.5
25.8	14.1	14.6	7.82	7.95	7.8	8.4	31.5	32.0	21.3	28.5

(a) NA Not applicable.

(b) NE Not established.

APPENDIX G

BIOASSAY RESULTS FOR 96-HOUR SUSPENDED-PARTICULATE-PHASE TEST
AND 96-HOUR REFERENCE TOXICANT TEST FOR *HOLMESIMYSIS costata*

TABLE G.1. Test Results for 96-Hour *H. costata* Suspended-Particulate-Phase Test, Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Concentration (Percent SPP)</u>	<u>Replicate</u>	<u><i>H. costata</i></u>		<u>Mean Proportion Surviving</u>	<u>Proportion Surviving</u>	<u>Standard Deviation</u>
			<u>Live^(a)</u>	<u>Dead or Missing</u>			
<u>January 1993</u>							
OBM COMP	0	1	10	0	1.00	0.93	0.06
OBM COMP	0	2	9	1	0.90		
OBM COMP	0	3	9	1	0.90		
OBM COMP	10	1	9	1	0.90	0.93	0.06
OBM COMP	10	2	10	0	1.00		
OBM COMP	10	3	9	1	0.90		
OBM COMP	50	1	9	1	0.90	0.93	0.06
OBM COMP	50	2	9	1	0.90		
OBM COMP	50	3	10	0	1.00		
OBM COMP	100	1	10	0	1.00	0.93	0.06
OBM COMP	100	2	9	1	0.90		
OBM COMP	100	3	9	1	0.90		
<u>January 1994</u>							
OBM COMP	0	1	10	0	1.00	0.98	0.04
OBM COMP	0	2	9	1	0.90		
OBM COMP	0	3	10	0	1.00		
OBM COMP	0	4	10	0	1.00		
OBM COMP	0	5	10	0	1.00		
OBM COMP	10	1	9	1	0.90	0.94	0.05
OBM COMP	10	2	10	0	1.00		
OBM COMP	10	3	9	1	0.90		
OBM COMP	10	4	10	0	1.00		
OBM COMP	10	5	9	1	0.90		
OBM COMP	50	1	10	0	1.00	0.98	0.04
OBM COMP	50	2	9	1	0.90		
OBM COMP	50	3	10	0	1.00		
OBM COMP	50	4	10	0	1.00		
OBM COMP	50	5	10	0	1.00		
OBM COMP	100	1	10	0	1.00	0.96	0.05
OBM COMP	100	2	10	0	1.00		
OBM COMP	100	3	9	1	0.90		
OBM COMP	100	4	10	0	1.00		
OBM COMP	100	5	9	1	0.90		

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

TABLE G.2. Summary of Mean Proportion Surviving 96-Hour *H. costata* Suspended-Particulate-Phase Test, Older Bay Mud Study

<u>Sediment Treatment</u>	<u>Concentration (Percent SPP)</u>	<u>Mean Proportion Surviving^(a)</u>
<u>January 1993</u>		
OBM COMP	0	0.93
OBM COMP	10	0.93
OBM COMP	50	0.93
OBM COMP	100	0.93
<u>January 1994</u>		
OBM COMP	0	0.98
OBM COMP	10	0.94
OBM COMP	50	0.98
OBM COMP	100	0.96

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

TABLE G.3. Water Quality Summary for 96-Hour *H. costata* Suspended-Particulate-Phase Test, Older Bay Mud Study

Sediment Treatment	Percent SPP	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)		Ammonia (mg/L)
		Min	Max	Min	Max	Min	Max	Min	Max	
Acceptable Range		13.0	17.0	7.30	8.30	≥4.0	NA ^(a)	28.0	32.0	NE ^(b)
<u>January 1993</u>										
OBM COMP	0	15.5	16.0	7.45	7.84	6.6	8.7	29.0	30.0	NM ^(c)
OBM COMP	10	15.6	16.0	7.63	7.89	6.7	8.7	29.0	30.0	NM
OBM COMP	50	15.6	15.9	7.60	7.99	6.6	8.6	29.0	30.0	NM
OBM COMP	100	15.6	16.0	7.51	8.11	6.6	8.6	29.0	30.0	NM
<u>January 1994</u>										
OBM COMP	0	14.9	15.9	7.69	8.05	7.5	8.3	31.0	32.0	NM
OBM COMP	10	14.9	15.9	7.62	8.07	7.7	8.3	30.5	32.0	NM
OBM COMP	50	15.0	15.8	7.62	8.12	7.7	8.3	30.0	31.5	NM
OBM COMP	100	15.0	15.9	7.66	8.17	7.7	8.4	30.0	30.5	2.22

(a) NA Not applicable.
(b) NE Not established.
(c) NM Not measured.

TABLE G.4. Test Results for 96-Hour *H. costata* Copper Reference Toxicant Test, Older Bay Mud Study

Copper Concentration ($\mu\text{g/L}$)	Replicate	<i>H. costata</i>		Proportion Surviving	Mean Proportion Surviving	Standard Deviation
		Live ^(a)	Dead or Missing			
January 1993						
0	1	9	1	0.90	0.93	0.06
0	2	10	0	1.00		
0	3	9	1	0.90		
50	1	9	1	0.90	0.83	0.06
50	2	8	2	0.80		
50	3	8	2	0.80		
100	1	5	5	0.50	0.57	0.12
100	2	7	3	0.70		
100	3	5	5	0.50		
200	1	0	10	0.00	0.00	0.00
200	2	0	10	0.00		
200	3	0	10	0.00		
400	1	0	10	0.00	0.00	0.00
400	2	0	10	0.00		
400	3	0	10	0.00		
January 1994						
0	1	10	0	1.00	0.93	0.06
0	2	9	1	0.90		
0	3	9	1	0.90		
50	1	9	1	0.90	0.87	0.06
50	2	8	2	0.80		
50	3	9	1	0.90		
100	1	1	9	0.10	0.07	0.06
100	2	1	9	0.10		
100	3	0	10	0.00		
150	1	0	10	0.00	0.00	0.00
150	2	0	10	0.00		
150	3	0	10	0.00		
200	1	0	10	0.00	0.00	0.00
200	2	0	10	0.00		
200	3	0	10	0.00		

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

TABLE G.5. Water Quality Summary for 96-Hour *H. costata* Copper Reference Toxicant Test, Older Bay Mud Study

Copper Concentration ($\mu\text{g/L}$)	Temperature ($^{\circ}\text{C}$)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)	
	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	≥ 4.0	NA ^(a)	28.0	32.0
<u>January 1993</u>								
0	16.1	16.5	7.44	7.82	6.2	8.2	30.5	31.0
50	16.0	16.4	7.54	7.83	6.6	8.3	30.5	31.0
100	16.1	16.5	7.66	7.95	7.1	8.2	30.5	31.0
200	16.2	16.6	7.77	7.96	7.2	8.3	30.0	31.0
400	16.3	16.6	7.80	7.89	7.8	8.2	30.0	31.0
<u>January 1994</u>								
0	14.7	15.7	7.62	7.91	7.8	8.7	30.5	32.0
50	14.8	15.8	7.69	7.88	7.7	8.4	30.5	32.0
100	14.7	15.9	7.72	8.01	7.8	8.5	30.5	32.0
150 ^(b)	15.1	15.5	7.75	7.77	7.8	8.5	30.0	31.5
200 ^(b)	15.1	15.9	7.75	7.79	8.0	8.1	30.5	31.5

(a) NA Not applicable.

(b) There was 100% mortality of *H. costata* after 24 hours.

TABLE G.6. Test Results for 96-Hour *H. costata* Ammonia Reference Toxicant Test, Older Bay Mud Study

Ammonia Concentration (mg/L)	Replicate	<i>H. costata</i>		Proportion Surviving	Mean Proportion Surviving	Standard Deviation
		Live ^(a)	Dead or Missing			
<u>January 1994</u>						
<u>Test 1</u>						
0.68	1	9	1	0.90	0.97	0.06
0.68	2	10	0	1.00		
0.68	3	10	0	1.00		
0.71	1	8	2	0.80	0.90	0.10
0.71	2	10	0	1.00		
0.71	3	9	1	0.90		
0.79	1	9	1	0.90	0.93	0.06
0.79	2	9	1	0.90		
0.79	3	10	0	1.00		
0.87	1	9	1	0.90	0.90	0.00
0.87	2	9	1	0.90		
0.87	3	9	1	0.90		
1.32	1	9	1	0.90	0.90	0.10
1.32	2	10	0	1.00		
1.32	3	8	2	0.80		
<u>Test 2</u>						
1.64	1	9	1	0.90	0.93	0.06
1.64	2	10	0	1.00		
1.64	3	9	1	0.90		
3.03	1	10	0	1.00	0.90	0.10
3.03	2	8	2	0.80		
3.03	3	9	1	0.90		
6.66	1	9	1	0.90	0.87	0.06
6.66	2	9	1	0.90		
6.66	3	8	2	0.80		
14.8	1	8	2	0.80	0.80	0.00
14.8	2	8	2	0.80		
14.8	3	8	2	0.80		
29.96	1	9	1	0.90	0.83	0.12
29.96	2	9	1	0.90		
29.96	3	7	3	0.70		

TABLE G.6. (contd)

Ammonia Concentration (mg/L)	Replicate	<i>H. costata</i>		Proportion Surviving	Mean Proportion Surviving	Standard Deviation
		Live ^(a)	Dead or Missing			
<u>Test 3</u>						
2.37	1	10	0	1.00	0.97	0.06
2.37	2	10	0	1.00		
2.37	3	9	1	0.90		
57.99	1	3	7	0.30	0.47	0.15
57.99	2	5	5	0.50		
57.99	3	6	4	0.60		
79.11	1	0	10	0.00	0.00	0.00
79.11	2	0	10	0.00		
79.11	3	0	10	0.00		
96.8	1	0	10	0.00	0.00	0.00
96.8	2	0	10	0.00		
96.8	3	0	10	0.00		

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

**TABLE G.7. Water Quality Summary for 96-Hour *H. costata* Ammonia Reference
Toxicant Test, Older Bay Mud Study**

Ammonia Concentration (mg/L)	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)		Ammonia (mg/L)	
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	4.0	NA ^(a)	28.0	32.0	NE ^(b)	NE
<u>January 1994</u>										
<u>Test 1</u>										
0.68	14.6	15.1	7.73	7.94	7.7	8.4	31.5	32.0	0.212	1.04
0.71	14.7	15.3	7.78	7.93	7.7	8.5	31.5	32.0	0.246	1.20
0.79	14.7	15.3	7.80	7.96	8.0	8.5	31.0	32.0	0.289	1.27
0.87	14.6	15.3	7.78	7.99	7.8	8.5	31.0	32.5 ^(c)	0.406	1.31
1.32	14.7	15.4	7.76	8.01	7.9	8.4	31.0	32.0	0.785	1.87
<u>Test 2</u>										
1.64	14.7	14.9	7.60	7.98	7.0	8.3	30.5	31.5	0.286	4.18
3.03	14.7	14.8	7.59	7.98	7.4	8.4	30.5	31.5	1.73	5.25
6.66	14.7	14.9	7.69	7.97	7.7	8.4	30.5	31.5	4.79	7.81
14.8	14.7	14.8	7.68	7.95	7.6	8.5	31.0	31.5	11.9	17.6
29.96	14.7	14.9	7.69	7.90	7.8	8.4	30.5	31.5	18.7	37.7
<u>Test 3</u>										
2.37	14.8	15.6	7.46	7.84	7.4	8.3	30.5	31.0	0.556	3.93
57.99	14.7	15.5	7.65	7.78	7.7	8.2	30.5	31.0	51.2	68.9
79.11	14.6	15.6	7.54	7.84	7.1	8.2	30.5	31.0	63.7	96.4
96.8	14.6	15.4	7.48	7.88	7.6	8.3	30.5	31.0	75.4	120

(a) NA Not applicable.

(b) NE Not established.

(c) Data point out of range.

APPENDIX H

BIOASSAY RESULTS FOR 10-DAY SOLID-PHASE, FLOW-THROUGH TEST
WITH *NEPHTYS caecoides* FOR FEEDING STUDY

TABLE H.1. Test Results for 10-Day *N. caecoides* Solid-Phase, Flow-Through Preliminary Test with Different Concentrations of TOC and Food Sources, Older Bay Mud Study

Sediment Treatment	Replicate	Live ^(a)	Dead or Missing	Proportion Surviving	Mean Proportion Surviving	Standard Deviation
OBM	1	8	12	0.40		
OBM	2	6	14	0.30		
OBM	3	5	15	0.25	0.32	0.08
OBM with water	1	18	2	0.90		
OBM with water	2	13	7	0.65		
OBM with water	3	15	5	0.75	0.77	0.13
OBM-0.4% TOC (mixed with alfalfa)	1	11	9	0.55		
OBM-0.4% TOC (mixed with alfalfa)	2	18	2	0.90		
OBM-0.4% TOC (mixed with alfalfa)	3	17	3	0.85		
OBM-0.4% TOC (mixed with alfalfa)	4	17	3	0.85		
OBM-0.4% TOC (mixed with alfalfa)	5	18	2	0.90	0.81	0.15
OBM-0.8% TOC (mixed with alfalfa)	1	16	4	0.80		
OBM-0.8% TOC (mixed with alfalfa)	2	17	3	0.85		
OBM-0.8% TOC (mixed with alfalfa)	3	18	2	0.90		
OBM-0.8% TOC (mixed with alfalfa)	4	18	3	0.90		
OBM-0.8% TOC (mixed with alfalfa)	5	18	2	0.90	0.87	0.04
OBM-0.8% TOC (sprinkled with alfalfa)	1	15	5	0.75		
OBM-0.8% TOC (sprinkled with alfalfa)	2	19	1	0.95		
OBM-0.8% TOC (sprinkled with alfalfa)	3	16	4	0.80		
OBM-0.8% TOC (sprinkled with alfalfa)	4	19	1	0.95		
OBM-0.8% TOC (sprinkled with alfalfa)	5	18	2	0.90	0.87	0.09
OBM-0.8% TOC (mixed with <i>Enteromorpha</i>)	1	18	2	0.90		
OBM-0.8% TOC (mixed with <i>Enteromorpha</i>)	2	17	3	0.85		
OBM-0.8% TOC (mixed with <i>Enteromorpha</i>)	3	18	2	0.90		
OBM-0.8% TOC (mixed with <i>Enteromorpha</i>)	4	18	2	0.90		
OBM-0.8% TOC (mixed with <i>Enteromorpha</i>)	5	16	4	0.80	0.87	0.04
OBM-0.8% TOC (sprinkled with <i>Enteromorpha</i>)	1	18	2	0.90		
OBM-0.8% TOC (sprinkled with <i>Enteromorpha</i>)	2	19	1	0.95		
OBM-0.8% TOC (sprinkled with <i>Enteromorpha</i>)	3	20	0	1.00		
OBM-0.8% TOC (sprinkled with <i>Enteromorpha</i>)	4	14	6	0.70		
OBM-0.8% TOC (sprinkled with <i>Enteromorpha</i>)	5	14	6	0.70	0.85	0.14
OBM-0.8% TOC (mixed with tetramin)	1	18	2	0.90		
OBM-0.8% TOC (mixed with tetramin)	2	17	3	0.85		
OBM-0.8% TOC (mixed with tetramin)	3	16	4	0.80		
OBM-0.8% TOC (mixed with tetramin)	4	18	2	0.90		
OBM-0.8% TOC (mixed with tetramin)	5	16	4	0.80	0.85	0.05

TABLE H.1. (contd)

Sediment Treatment	Replicate	Live ^(a)	Dead or Missing	Proportion Surviving	Mean Proportion Surviving	Standard Deviation
OBM-0.8% TOC (sprinkled with tetramin)	1	14	6	0.70		
OBM-0.8% TOC (sprinkled with tetramin)	2	9	11	0.45		
OBM-0.8% TOC (sprinkled with tetramin)	3	10	10	0.50		
OBM-0.8% TOC (sprinkled with tetramin)	4	16	4	0.80		
OBM-0.8% TOC (sprinkled with tetramin)	5	14	6	0.70	0.63	0.15
OBM-1.2% TOC (mixed with alfalfa)	1	17	3	0.85		
OBM-1.2% TOC (mixed with alfalfa)	2	13	7	0.65		
OBM-1.2% TOC (mixed with alfalfa)	3	9	11	0.45		
OBM-1.2% TOC (mixed with alfalfa)	4	11	9	0.55		
OBM-1.2% TOC (mixed with alfalfa)	5	17	3	0.85	0.67	0.18
C-NE	1	20	0	1.00		
C-NE	2	20	0	1.00		
C-NE	3	19	1	0.95		
C-NE	4	20	0	1.00		
C-NE	5	20	0	1.00	0.99	0.02

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

TABLE H.2. Water Quality Summary for 10-Day *N. caecoides* Solid-Phase, Flow-Through Preliminary Test, Older Bay Mud Study

Sediment Treatment	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)	
	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	6.0	NA ^(a)	28.0	32.0
OBM	14.2	15.7	7.78	7.92	7.5	8.0	31.5	32.0
OBM with water	14.0	16.0	7.65	7.93	7.5	8.0	31.5	32.0
OBM-0.4% TOC (mixed with alfalfa)	15.0	16.0	7.70	7.89	7.3	7.8	31.5	32.0
OBM-0.8% TOC (mixed with alfalfa)	15.0	15.8	7.71	7.87	7.0	7.6	31.5	32.0
OBM-0.8% TOC (sprinkled with alfalfa)	15.1	15.8	7.73	7.93	7.4	7.8	31.5	32.0
OBM-0.8% TOC (mixed with <i>Enteromorpha</i>)	15.1	16.0	7.68	7.91	7.0	7.7	31.5	32.0
OBM-0.8% TOC (sprinkled with <i>Enteromorpha</i>)	15.0	15.8	7.72	7.88	7.3	7.8	31.5	32.0
OBM-0.8% TOC (mixed with tetramin)	15.0	15.8	7.68	7.89	7.0	7.6	31.5	32.0
OBM-0.8% TOC (sprinkled with tetramin)	15.0	15.8	7.71	7.89	7.2	7.8	31.5	32.0
OBM-1.2% TOC (mixed with alfalfa)	15.2	15.9	7.62	7.89	6.8	7.6	31.5	32.0
C-NE	15.0	15.9	7.73	7.87	7.4	7.8	31.5	32.0

(a) NA Not applicable.

TABLE H.3. Ammonia Measurements in the Overlying and Porewater from Day 10 of the *N. caecoides*, Preliminary Test, Older Bay Mud Study

Sediment Treatment	Overlying Day 10 ^(a)				
	Ammonia Concentration (mg/L)	Temperature (°C)	pH	Dissolved Oxygen (mg/L)	Salinity (o/oo)
OBM	0.082	15.1	7.85	7.6	32.0
OBM with water	0.034	15.2	7.83	7.6	32.0
OBM-0.4% TOC (mixed with alfalfa)	0.081	15.2	7.85	7.6	31.5
OBM-0.8% TOC (mixed with alfalfa)	0.057	15.2	7.79	7.3	32.0
OBM-0.8% TOC (sprinkled with alfalfa)	0.050	15.3	7.79	7.4	32.0
OBM-0.8% TOC (mixed with <i>Enteromorpha</i>)	0.054	15.2	7.80	7.5	31.5
OBM-0.8% TOC (sprinkled with <i>Enteromorpha</i>)	0.071	15.3	7.83	7.5	32.0
OBM-0.8% TOC (mixed with tetramin)	0.119	15.2	7.78	7.3	32.0
OBM-0.8% TOC (sprinkled with tetramin)	0.084	15.2	7.87	7.5	32.0
OBM-1.2% TOC (mixed with alfalfa)	0.045	15.2	7.82	7.4	32.0
C-NE	0.176	15.1	7.85	7.8	32.0

Sediment Treatment	Porewater Day 10 ^(a)				
	Ammonia Concentration (mg/L)	Temperature (°C)	pH	Dissolved Oxygen (mg/L)	Salinity (o/oo)
OBM	0.601	15.4	8.09	6.0	32.0
OBM with water	0.463	15.4	8.12	7.1	32.0
OBM-0.4% TOC (mixed with alfalfa)	1.65	15.3	7.67	5.9	31.5
OBM-0.8% TOC (mixed with alfalfa)	1.98	15.4	7.40	6.1	32.0
OBM-0.8% TOC (sprinkled with alfalfa)	0.287	15.5	7.72	5.4	32.5
OBM-0.8% TOC (mixed with <i>Enteromorpha</i>)	1.03	15.4	7.48	5.5	32.0
OBM-0.8% TOC (sprinkled with <i>Enteromorpha</i>)	0.701	15.5	7.82	6.1	32.5
OBM-0.8% TOC (mixed with tetramin)	5.90	15.4	7.32	4.1	32.5
OBM-0.8% TOC (sprinkled with tetramin)	2.63	15.5	7.88	6.2	32.0
OBM-1.2% TOC (mixed with alfalfa)	3.84	15.4	7.07	4.2	33.5
C-NE	0.401	15.4	7.78	6.2	32.0

(a) The temperature, pH, dissolved oxygen, and salinity values are an average of the five replicates.

TABLE H.4. Test Results for 10-day *N. caecoides* Solid-Phase, Flow-Through Definitive with Different Concentrations of TOC and *Enteromorpha*, Older Bay Mud Study

Sediment Treatment ^(a)	Replicate	Live ^(b)	Dead or Missing	Proportion Surviving	Mean Proportion Surviving	Standard Deviation
OBM	1	2	18	0.10	0.20	0.10
OBM	2	6	14	0.30		
OBM	3	4	16	0.20		
OBM	4	NA ^(c)	NA	NA		
OBM	5	NA	NA	NA		
OBM with water	1	13	7	0.65	0.65	0.00
OBM with water	2	13	7	0.65		
OBM with water	3	13	7	0.65		
OBM with water	4	NA	NA	NA		
OBM with water	5	NA	NA	NA		
OBM with 0.24% TOC	1	18	2	0.90	0.89	0.06
OBM with 0.24% TOC	2	16	4	0.80		
OBM with 0.24% TOC	3	19	1	0.95		
OBM with 0.24% TOC	4	18	2	0.90		
OBM with 0.24% TOC	5	18	2	0.90		
OBM with 0.27% TOC	1	13	7	0.65	0.80	0.14
OBM with 0.27% TOC	2	17	3	0.85		
OBM with 0.27% TOC	3	20	0	1.00		
OBM with 0.27% TOC	4	16	4	0.80		
OBM with 0.27% TOC	5	14	6	0.70		
OBM with 0.39% TOC	1	18	2	0.90	0.87	0.03
OBM with 0.39% TOC	2	17	3	0.85		
OBM with 0.39% TOC	3	17	3	0.85		
OBM with 0.39% TOC	4	18	2	0.90		
OBM with 0.39% TOC	5	17	3	0.85		
OBM with 0.44% TOC	1	17	3	0.85	0.78	0.13
OBM with 0.44% TOC	2	14	6	0.70		
OBM with 0.44% TOC	3	17	3	0.85		
OBM with 0.44% TOC	4	12	8	0.60		
OBM with 0.44% TOC	5	18	2	0.90		

TABLE H.4. (contd)

Sediment Treatment	Replicate	Live	Dead or Missing	Proportion Surviving	Mean Proportion Surviving	Standard Deviation
C-NE	1	20	0	1.00		
C-NE	2	20	0	1.00		
C-NE	3	20	0	1.00		
C-NE	4	20	0	1.00		
C-NE	5	20	0	1.00	1.00	0.00

(a) The percentages of TOC are actual measurements determined by analysis of sediment at Global Geochemistry.

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

(c) NA Not applicable.

TABLE H.5. Water Quality Summary for 10-day *N. caecoides* Solid-Phase, Flow-Through Definitive Test with Different Concentrations of TOC and *Enteromorpha*, Older Bay Mud Study

Sediment Treatment ^(a)	Temperature (°C)		pH		Dissolved Oxygen (mg/L)		Salinity (o/oo)	
	Min	Max	Min	Max	Min	Max	Min	Max
Acceptable Range	13.0	17.0	7.30	8.30	6.0	NA ^(b)	28.0	32.0
OBM	14.7	15.7	7.78	7.87	6.5	8.0	30.5	32.0
OBM with water	14.7	15.8	7.76	7.90	6.6	8.0	30.5	32.0
OBM with 0.24% TOC	14.7	15.8	7.79	7.89	6.3	8.0	30.5	32.0
OBM with 0.27% TOC	14.7	15.8	7.76	7.89	6.2	7.9	30.5	32.0
OBM with 0.39% TOC	14.7	15.8	7.71	7.86	6.0	7.8	30.5	32.0
OBM with 0.44% TOC	14.6	15.7	7.66	7.87	6.0	7.8	30.5	32.0
C-NE	14.7	15.8	7.78	8.00	6.3	8.0	30.5	32.0

(a) The percentages of TOC are actual measurements determined by analysis of sediment at Global Chemistry.

(b) NA Not applicable.

TABLE H.6. Ammonia Measurements in the Overlying and Porewater from Day 10 of *N. caecoides* Definitive Test, Older Bay Mud Study

Sediment Treatment ^(a)	Overlying Day 10				
	Ammonia Concentration (mg/L)	Temperature (°C)	pH	Dissolved Oxygen (mg/L)	Salinity (o/oo)
OBM	0.031	15.6	7.85	8.0	32.0
OBM with water	0.073	15.8	7.86	7.9	32.0
OBM with 0.24% TOC	0.062	15.7	7.83	7.8	32.0
OBM with 0.27% TOC	0.179	15.7	7.86	7.9	32.0
OBM with 0.39% TOC	0.153	15.8	7.81	7.6	32.0
OBM with 0.44% TOC	0.073	15.7	7.77	7.6	32.0
C-NE	0.027	15.8	7.83	7.9	32.0

Sediment Treatment	Porewater Day 10				
	Ammonia Concentration (mg/L)	Temperature (°C)	pH	Dissolved Oxygen (mg/L)	Salinity (o/oo)
OBM	3.24	14.8	7.87	7.0	32.0
OBM with water	1.28	14.7	8.13	6.6	32.0
OBM with 0.24% TOC	0.507	14.9	7.73	6.0	32.0
OBM with 0.27% TOC	2.76	14.9	7.67	6.3	32.5
OBM with 0.39% TOC	1.96	14.8	7.45	5.3	32.0
OBM with 0.44% TOC	3.35	14.9	7.29	5.3	32.0
C-NE	0.86	14.8	7.80	6.3	32.0

(a) The percentages of TOC are actual measurements determined by analysis of sediment at Global Geochemistry.

APPENDIX I

TISSUE CHEMISTRY AND QUALITY ASSURANCE DATA FOR *MACOMA nasuta*

QA/QC SUMMARY

PROGRAM
LABORATORY:
MATRIX:
PARAMETER:

Older Bay Mud Study
Battelle Marine Sciences Laboratory, Sequim, Washington
M. nasuta Tissue
Polynuclear Aromatic Hydrocarbons (PAH)

METHOD

Tissue samples were homogenized using a Tekmar tissuemizer. Approximately 5 grams of homogenized tissue were extracted with methylene chloride using a roller under ambient conditions following methods used by the National Oceanic and Atmospheric Administration for their 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 quantified using gas chromatography/mass spectrometry (GC/MS) in the selected ion mode (SIM) following EPA Method 8270 (EPA 1986).

HOLDING TIMES

Samples were received on 3/18/93 in good condition. Samples were placed into Battelle's log-in system and stored at approximately -20°C until extraction. Samples were extracted in one batch on 3/23/93 and analyzed by GC/MS on 3/29/93, which is within the established holding time of 40 days.

DETECTION LIMITS

Target detection limits of 20 µg/kg wet weight were met for all PAH compounds.

METHOD BLANKS

Four method blanks were analyzed with the sediment samples. Nine PAHs were detected in the sediment samples at values less than five times those in the method blank and have been flagged with a "B" to indicate possible blank contamination.

MATRIX SPIKES

One sample, OBM COMP, was spiked in duplicate with all PAH compounds. Matrix spike recoveries ranged from 92% to 108% which is within the laboratory QA/QC recovery limit (40%-120%). Relative percent differences between MS and MSD recoveries were within the ±30% QA/QC limit ranging from 0% to 6% which indicates good precision. Three samples, *M. nasuta* background, QC sample, and QC sample, were spiked with PAHs. Percent recoveries ranged from 69% to 125%; three recoveries were outside the acceptable QA/QC range (40% to 120%).

REPLICATES

One sample, OBM COMP, was extracted in duplicate. Precision was measured by calculating the relative percent difference (RPD) between the replicate results. No PAHs were detected in either replicate above the target detection limits of 20 µg/kg; therefore, no calculations were performed.

Three samples, *M. nasuta* background, QC sample, and R-OS, were analyzed in triplicate. Precision was measured by calculating the relative standard deviation (RSD) among replicate results. The RSDs ranged from 1% to 36% with only one value outside of the ±30% QA/QC limit.

QA/QC SUMMARY PAHs (contd)

SRMs

One SRM was analyzed, NIST 1974, a marine mussel tissue obtained by the National Institute for Standards and Technology. Eight of the sixteen PAH compounds analyzed are certified. Of these, only two are certified at levels above the target detection limit of 20 µg/kg. One compound, pyrene, was detected within the 30% target for accuracy. The other compound, fluoranthene, slightly exceeded the 30% limit.

SURROGATES

Up to six isotopically labelled compounds, d8-Naphthalene, d8-Acenaphthalene, d10-Acenaphthene, d10-Pyrene, d12-Benzo(a)pyrene, d12-Chrysene, and d14-dibenzo(a,h,i)anthracene, were added to the sediment samples prior to extraction to assess the efficiency of the method. Recoveries of all surrogates ranged from 62% to 95% which is within the quality control limits of 40% to 120%.

REFERENCES

EPA (U.S. Environmental Protection Agency). 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SWP-846 Method 8270. EPA-955-001-00000, U.S. Government Printing Office, Washington D.C.

Krahn, M. M., C. A. Wigren, R. W. Pearch, L. K. Moore, R. G. Bogar, W. D. MacLeod, Jr., S. L. Chan, and D. W. Brown. 1988. "A Rapid High-Performance Liquid Chromatographic Method for Isolating Organic Contaminants from Tissue and Sediment Extracts." NOAA Technical Memorandum NMFS F/NWC-153, Silver Spring, Maryland.

QA/QC SUMMARY

PROGRAM	Older Bay Mud Study
LABORATORY:	Battelle Marine Sciences Laboratory, Sequim, Washington
MATRIX:	<i>M. nasuta</i> Tissue
PARAMETER:	Chlorinated Pesticides
METHOD	Tissue samples were homogenized using a Tekmar tissuemizer. Approximately 5 grams of homogenized tissue were extracted with methylene chloride using a roller under ambient conditions following methods used by the National Oceanic and Atmospheric Administration for their Status and Trends Program (Krahn et al, 1988). Samples were then cleaned using silica/alumina (5% deactivated) chromatography followed by HPLC cleanup. Extracts were analyzed using Gas Chromatography/Electron Capture Detection (GC/ECD). Extracts were exchanged to methyl-t-butylether (MTBE) and analyzed by capillary column gas chromatography/electron capture detection (GC/ECD) following EPA method 8080 (1986). A second, confirmatory column (DB1701) was used to confirm the presence of pesticide compounds. Values between primary and confirmatory columns must be within a factor of two of one another to qualify as a confirmed detection. The primary column used was a J&W DB-5 capillary column (30m x 0.25mm I.D.). Samples were analyzed for a total of 22 chlorinated pesticide compounds.
HOLDING TIMES	Samples were received on 3/18/93 in good condition. Samples were placed into Battelle's log-in system and stored at approximately -20°C until extraction. Samples were extracted in one batch on 3/23/93. Extracts were analyzed by GC/ECD 4/1/93, within the required holding time of 40 days. For the January 1994 study, samples were received on 3/21/94 in good condition. Samples were logged in and stored at approximately -20°C until extraction. Extracts were analyzed by GC/ECD in three batches from 5/10/94 to 6/6/94.
DETECTION LIMITS	Target detection limits of 2 µg/kg wet weight were met for all samples. Toxaphene and chlordane detection limits of 30 µg/kg were also met. Detection limits reported are based on an MDL study involving low level spikes of 7 replicate extractions. The MDLs, defined as 3 times the standard deviation of the mean recovery, were all below 2 µg/kg.
METHOD BLANKS	Four method blanks were analyzed with the sediment samples. One pesticide, tech-chlordane, was detected in Blank 1 at 13.0 µg/kg. None of the sediment samples had detectable concentrations of tech-chlordane above the target detection limit of 30 µg/kg.

QA/QC SUMMARY PESTICIDES (contd)

MATRIX SPIKES

One sample, OBM COMP, was spiked in duplicate with a subset of six pesticides. Matrix spike recoveries ranged from 103% to 119%, which is within the laboratory QA/QC recovery limit (40% to 120%). Precision was measured by comparing the relative percent difference (RPD) between spike recoveries. The RPDs ranged from 0% to 9%, which were below the precision goal of $\leq 30\%$, indicating acceptable precision.

Three samples, *M. nasuta* background, QC sample, and QC sample, were spiked with a subset of six pesticides. Matrix spike recoveries ranged from 50% to 108%, which is within the laboratory QA/QC recovery limit (40% to 120%).

REPLICATES

One sample, OBM COMP, was analyzed in duplicate. Precision was measured by calculating the relative percent difference (RPD) between the replicate results. Four compounds were detected in both replicates and RPD values ranged from 0% to 20% which is below the precision goal of $\leq 30\%$, indicating acceptable precision.

Three samples, *M. nasuta* background, QC sample, and R-OS, were analyzed in triplicate. Precision was measured by calculating the relative standard deviation (RSD) among the triplicate results. Two compounds, aldrin and 4,4'-DDT, were detected in all replicates and produced RSD values of 12% and 16%, respectively. These values were below the precision goal of $\leq 30\%$, indicating acceptable precision.

SRMs

One SRM, NIST 1974, a marine mussel tissue obtained from the National Institute for Standards and Technology, was analyzed for pesticides. Although this sample is not certified for pesticides, it does present consensus values for 5 pesticides. Results for 4,4'-DDD, were within 30% of the consensus mean. Results for α -chlordane, dieldrin, 4,4'-DDE, and 4,4'-DDT exceeded the upper range of the 30% difference, however, since these values are only "consensus," no further action was taken. Historically, higher values have been obtained for 4,4'-DDT. This appears to be a result of matrix interference from the sample. All other QA/QC results for 4,4'-DDT were acceptable.

SURROGATES

Up to four compounds [PCB 103, PCB 198, tetrachlorometaxylene (TCMX), and octachloronaphthalene (OCN)] were added to all sediment samples prior to extraction to assess the efficiency of the analysis. Recoveries of PCB 103, PCB 198, and TCMX ranged from 61% to 116%, which is within the QA/QC guidelines of 40% - 120%. Recoveries of OCN ranged from 117% to 162% with all but one falling outside of the QA/QC range. Since all of the other surrogates were acceptable, no corrective action was taken.

QA/QC SUMMARY PESTICIDES (contd)

REFERENCES

EPA (U.S. Environmental Protection Agency). 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SWP-846 Method 8270. EPA-955-001-00000, U.S. Government Printing Office, Washington D.C.

Krahn, M. M., C. A. Wigren, R. W. Pearch, L. K. Moore, R. G. Bogar, W. D. MacLeod, Jr., S. L. Chan, and D. W. Brown. 1988. "A Rapid High-Performance Liquid Chromatographic Method for Isolating Organic Contaminants from Tissue and Sediment Extracts." NOAA Technical Memorandum NMFS F/NWC-153, Silver Spring, Maryland.

QA/QC SUMMARY

PROGRAM
LABORATORY:
MATRIX:
PARAMETER:

Older Bay Mud Study
Battelle Marine Sciences Laboratory, Sequim, Washington
***M. nasuta* Tissue**
Polychlorinated Biphenyls (PCBs)

METHOD

Tissue samples were homogenized using a Tekmar tissuemizer. Approximately 5 grams of homogenized tissue were extracted with methylene chloride using a roller under ambient conditions following methods used by the National Oceanic and Atmospheric Administration for their Status and Trends Program (Krahn et al, 1988). Samples were then cleaned using silica/alumina (5% deactivated) chromatography followed by HPLC cleanup. Extracts were analyzed using Gas Chromatography/Electron Capture Detection (GC/ECD). Extracts were exchanged to methyl-t-butylether (MTBE) and analyzed by capillary column gas chromatography/electron capture detection (GC/ECD) following EPA method 8080 (1986). The primary column used was a J&W DB-5 capillary column (30m x 0.25mm I.D.). Samples were analyzed for four Aroclor mixtures: 1242, 1248, 1254 and 1260.

HOLDING TIMES

Samples were received on 3/18/93 in good condition. Samples were logged into Battelle log-in system and stored at approximately -20°C until extraction. Samples were extracted in one batch on 3/23/93. Extracts were analyzed by GC/ECD 4/1/93, within the required holding time of 40 days.

DETECTION LIMITS

Target detection limits of 20 µg/kg wet weight were met for all samples. Detection limits reported are based on an MDL study involving low level spikes of 7 replicate extractions. The MDLs, defined as 3 times the standard deviation of the mean recovery were below 20 µg/kg.

METHOD BLANKS

Four method blanks were analyzed with the sediment samples. No PCB Aroclors were detected in the blank.

MATRIX SPIKES

One sample, OBM COMP, was spiked in duplicate with Aroclor 1254. Matrix spike recoveries ranged from 115% to 124%. The recovery for the matrix spike duplicate slightly exceeded the laboratory QA/QC recovery limit (40% - 120%). Precision was measured by comparing the Relative Percent Difference (RPD) between spike recoveries. The RPD was 7%, which is below the precision goal of ≤30%.

Three samples, *M. nasuta* background, QC sample, and QC sample, were spiked with Aroclor 1254. Matrix spike percent recoveries ranged from 72% to 106% which is within the QA/QC recovery limits of 40% to 120%.

QA/QC SUMMARY PCBs (contd)

REPLICATES

One sample, OBM COMP, was analyzed in duplicate. Precision was measured by calculating the RPD between the replicate results. No Aroclors were detected in either replicate, RPDs were not calculated.

Three samples, *M. nasuta* background, QC sample, and R-OS, were analyzed in triplicate. Precision was measured by calculating the RSD among the replicate results. The RSD's could not be calculated because Aroclors were not detected in any samples.

SRMs

Not applicable.

SURROGATES

Up to four compounds [PCB 103, PCB 198, tetrachlorometaxylene (TCMX), and octachloronaphthalene (OCN)] were added to all sediment samples prior to extraction to assess the efficiency of the analysis. Recoveries of PCB 103, PCB 198, and TCMX ranged from 61% to 116%, which is within the QA/QC guidelines of 40% - 120%. Recoveries of OCN ranged from 117% to 162% with all but one falling outside of the QA/QC range. Since all of the other surrogates were acceptable, no corrective action was taken.

REFERENCES

EPA (U.S. Environmental Protection Agency). 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SWP-846 Method 8270. EPA-955-001-00000, U.S. Government Printing Office, Washington D.C.

Krahn, M. M., C. A. Wigren, R. W. Pearch, L. K. Moore, R. G. Bogar, W. D. MacLeod, Jr., S. L. Chan, and D. W. Brown. 1988. "A Rapid High-Performance Liquid Chromatographic Method for Isolating Organic Contaminants from Tissue and Sediment Extracts." NOAA Technical Memorandum NMFS F/NWC-153, Silver Spring, Maryland.

QA/QC SUMMARY

PROGRAM
LABORATORY:
MATRIX:
PARAMETER:

Older Bay Mud Study
Battelle Marine Sciences Laboratory, Sequim, Washington
***M. nasuta* Tissue**
Metals

METHOD

A total of 10 metals was analyzed for: silver (Ag), arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), nickel (Ni), lead (Pb), selenium (Se) and zinc (Zn). Five metals, As, Cu, Ni, Se and Zn were analyzed by energy diffusive x-ray fluorescence (XRF) and four metals, Ag, Cd, Cr and Pb were analyzed using Zeeman Graphite Furnace Atomic Absorption (GFAA) spectrometry following EPA Method 200.9 for the January 1993 study. Mercury was analyzed using cold-vapor atomic absorption spectroscopy (CVAA) according to the method of Bloom and Crecelius (1983). For the 1994 study, Ag, As, Cd, Cr, Cu, Ni, Pb, and Zn were analyzed by ICP/Mass Spectrometry, Se was analyzed by GFAA, and Hg was analyzed by CVAA.

To prepare tissue 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. The XRF analysis was performed on a 0.5-g aliquot of dried, ground material pressed into a pellet with a diameter of 2 cm. For GFAA, ICP/MS and CVAA analyses, 0.2- to 0.5-g aliquots of dried homogenous sample were digested using a mixture of nitric/perchloric acids.

HOLDING TIMES

Samples were received on 3/18/93 in good condition. Samples were placed into Battelle log-in system, frozen to -80°C and subsequently freeze dried within approximately 7 days of sample receipt. Samples were all analyzed within 180 days of collection. Mercury was analyzed within the 28 day holding time.

DETECTION LIMITS

Target detection limits were met for all metals.

METHOD BLANKS

Method blanks were analyzed for five metals (Ag, Cd, Cr, Hg, and Pb) during January 1993 study and for ten metals in January 1994 study. Method blanks are not analyzed by XRF. Arsenic was detected in the blanks, however, these levels were well below the detection limits; therefore, the data was not flagged to indicate blank contamination. All other metals were undetected in the blanks.

MATRIX SPIKES

OBM COMP was spiked with four metals (Ag, Cd, Cr, Hg, and Pb) during the January 1993 study. QC sample, QC sample, and R-AM, were spiked with all ten metals during the January 1994 study. Matrix spike recoveries ranged from 74% to 117%, with all but one percent recovery within the QA/QC limits of 75% to 125%. Samples for XRF are analyzed whole and cannot be spiked.

REPLICATES

One sample, OBM COMP, was analyzed in duplicate. Precision was measured by calculating the relative percent difference (RPD) between the replicate results. The RPDs ranged from 1% to 36%. All metals were within the QA/QC limits of $\pm 20\%$ indicating acceptable precision, with the exception of Ni which had an RPD of 36%.

OLDER BAY MUD

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QA/QC SUMMARY METALS (contd)

Three samples, QC sample, QC sample, and R-BF, were analyzed in triplicate. Precision was measured by calculating the relative standard deviation. The RSDs ranged from 0% to 11%, indicating acceptable precision.

SRMs

The SRM, 1566a (Oyster tissue from the National Institute of Standards and Technology, NIST), was analyzed for each batch of metals. Results for all metals except one were within $\pm 30\%$ of mean certified value, indicating acceptable accuracy.

REFERENCES

Bloom, N.S., and E.A. Crecelius. 1983. "Determination of Mercury in Seawater at Sub-Nanogram per Liter Levels." *Marine Chemistry* 21:337-390.

EPA (U.S. Environmental Protection Agency). 1991. *Methods for the Determination of Metals in Environmental Samples*. EPA-600/4-91-010 Methods 200.8, 200.9, Environmental Services Division, Monitoring Management Branch, Cincinnati, Ohio.

QA/QC SUMMARY

PROGRAM	Older Bay Mud Study
LABORATORY:	Battelle Marine Sciences Laboratory, Sequim, Washington
MATRIX:	<i>M. nasuta</i> Tissue
PARAMETER:	Butyltins
METHOD	Butyltin analyses were performed following the method of Unger et al. (1986).
HOLDING TIMES	Samples were received on 3/18/93 in good condition. Samples were placed into Battelle's log-in system and stored at approximately -20°C until extraction. Samples were extracted in one batch on 3/23/93. Extracts were analyzed by GC/FPD on 3/25/93, which is within the established holding time of 40 days. For the January 1994 study, samples were received on 3/21/94 in good condition. Samples were logged in and stored at approximately -20°C until extraction. Extracts were analyzed in two batches from 5/12/94 to 5/13/94.
DETECTION LIMITS	Target detection limits of 1.0 µg/kg on a wet weight basis were not met for all tissues. The actual detection limits ranged from undetected at 0.48 µg/kg to undetected at 1.39 µg/kg. Detection limits reported are defined as Limits of Quantitation (LOQ) which are determined as 10 times the standard deviation of results from 7 replicate low level matrix spikes. Values detected between the LOQ and the MDL (defined as 3 times the standard deviation) are flagged with a "J" flag.
METHOD BLANKS	One method blank was analyzed with each batch of sediment samples. Tributyltins were not detected in the blank. Dibutyltin was detected in Blank 1 at 3.9 µg/kg, which is below the LOQ; therefore, no associated data were flagged.
MATRIX SPIKES	One sample, OBM COMP, was spiked in duplicate with di- and tributyltin. Matrix spike recoveries ranged from 101% to 105% for the tri- and dibutyltins, which is within the QA/QC limits of 40% to 120%. Relative percent differences between MS and MSD recoveries ranged from 1% to 10% which is within the ±30% QA/QC limit for tri- and dibutyltins, indicating acceptable precision. Three samples, QC sample, OBM Comp, and C-SB, were spiked with di- and tri-butyltin compounds. Matrix spike recoveries ranged from 93% to 108%.
REPLICATES	One sample, OBM COMP, was analyzed in duplicate. Precision was measured by calculating the RPD between the replicate results. The RPD for tributyltin was 5% indicating acceptable precision. The RPD for dibutyltin was 33%, exceeding the precision goal of 30%. Three samples, QC sample, R-OS, and QC sample, were analyzed in triplicate. Precision was measured by calculating the RSD. The RSD ranged from 3% to 6%, indicating acceptable precision.

QA/QC SUMMARY BUTYLTINS (contd)

SRMs

Not applicable.

SURROGATES

One compound, triphenyltin chloride, is added to the sediment samples prior to extraction to assess the efficiency of the method. This compound also is used as an internal standard since all data are corrected for the recovery of the compound. Recoveries ranged from 61% to 107% for samples, which is within the QA/QC limits of 40% to 120%.

REFERENCES

Unger, M.A., W.G. Macintyre, J. Reaves and R.J. Huggett. 1986. "GC Determination of Butyltins in Natural Waters by Flame Photometric Detection of Hexyl Derivatives with Mass Spectrometric Confirmation." *Chemosphere*. 15(4):461-470.

TABLE I.1. Total Detected Polynuclear Aromatic Hydrocarbons (PAHs), Wet Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> PAHs (µg/kg wet weight)		
			Total Low Molecular Weight PAHs	Total High Molecular Weight PAHs	Total PAHs
<u>January 1993</u>					
OBM COMP	1	1	0	0	0
OBM COMP	2	1	0	0	0
OBM COMP	3	1	0	0	0
OBM COMP	4	1	0	0	0
OBM COMP, Replicate 1	5	1	0	0	0
OBM COMP, Replicate 2	5	1	0	0	0
<u>January 1994</u>					
R-OS	1	4	2	11	13
R-OS, Replicate 1	2	4	2	15	17
R-OS, Replicate 2	2	4	3	11	14
R-OS, Replicate 3	2	4	2	14	16
R-OS	3	4	3	10	13
R-OS	4	4	3	12	15
R-OS	5	4	2	10	12
R-BF	1	3	4	65	69
R-BF	2	2	7	66	73
R-BF	3	3	11	48	59
R-BF	4	3	4	49	53
R-BF	5	3	2	45	47
R-AM	1	2	46	524	570
R-AM	2	3	29	286	315
R-AM	3	3	50	492	542
R-AM	4	2	40	351	391
R-AM	5	3	55	403	458
C-SB	1	4	3	18	21
C-SB	2	4	3	39	42
C-SB	3	4	4	19	23
C-SB	4	4	3	19	22
C-SB	5	4	3	18	21
<i>M. nasuta</i> Background	1	2	7	36	43
<i>M. nasuta</i> Background	2	2	8	36	44
<i>M. nasuta</i> Background, Replicate 1	3	2	10	39	49
<i>M. nasuta</i> Background, Replicate 2	3	2	11	49	60
<i>M. nasuta</i> Background, Replicate 3	3	2	7	49	56
<i>M. nasuta</i> Background	4	2	8	40	48
<i>M. nasuta</i> Background	5	2	8	25	33

TABLE I.2. Low Molecular Weight Polynuclear Aromatic Hydrocarbons (LPAHs), Wet Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> LPAHs (µg/kg wet weight)					
			Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
Target DL ^(a)			20	20	20	20	20	20
<u>January 1993</u>								
OBM COMP	1	1	20 U ^(b)	20 U	20 U	20 U	20 U	20 U
OBM COMP	2	1	20 U	20 U	20 U	20 U	20 U	20 U
OBM COMP	3	1	20 U	20 U	20 U	20 U	20 U	20 U
OBM COMP	4	1	20 U	20 U	20 U	20 U	20 U	20 U
OBM COMP, Replicate 1	5	1	20 U	20 U	20 U	20 U	20 U	20 U
OBM COMP, Replicate 2	5	1	20 U	20 U	20 U	20 U	20 U	20 U
<u>January 1994</u>								
R-OS	1	4	0.83	0.81 U	0.77 U	1.35 U	0.74 ^(c)	2.18 U
R-OS, Replicate 1	2	4	2.06	1.61 U	1.53 U	2.70 U	1.27 U	4.37 U
R-OS, Replicate 2	2	4	2.56 ^(c)	1.59 U	1.52 U	2.67 U	1.26 U	4.32 U
R-OS, Replicate 3	2	4	2.00	1.61 U	1.53 U	2.70 U	1.27 U	4.37 U
R-OS	3	4	1.16	0.81 U	0.77 U	1.48 ^(c)	0.85	2.18 U
R-OS	4	4	1.29	0.82 U	0.88	1.38 U	0.88	2.23 U
R-OS	5	4	0.80	0.82 U	0.78 U	1.38 U	0.95	2.23 U
R-BF	1	3	2.72 B ^(d)	1.61 U	1.53 U	2.70 U	1.65	4.37 U
R-BF	2	2	0.85 B	0.82 U	0.92 ^(c)	1.38 U	2.26 B	2.86 ^(c)
R-BF	3	3	3.42 B	1.64 U	1.56 U	2.88	2.13 ^(c)	2.42 ^(c)
R-BF	4	3	2.05 B ^(c)	1.53 U	1.46 U	2.56 U	1.63 ^(c)	4.15 U
R-BF	5	3	1.98 B	1.63 U	1.65 U	2.73 U	1.28 U	4.41 U
R-AM	1	2	1.49 B	2.02	1.33	2.39	24.1	14.9
R-AM	2	3	1.92 B	1.64 U	1.56 U	3.46 ^(c)	14.6	9.23
R-AM	3	3	2.33 B	2.26 ^(c)	1.56 U	3.52 ^(c)	25.6	16.1
R-AM	4	2	1.66 B	2.14	1.35	2.95	20.3	11.6
R-AM	5	3	2.98 B	1.92 ^(c)	1.91	4.72	26.7	16.7
C-SB	1	4	1.92	0.81 U	0.77 U	1.35 U	1.45	2.18 U
C-SB	2	4	1.47	0.81 U	1.01 ^(c)	1.35 U	0.92 ^(c)	2.18 U
C-SB	3	4	0.80 ^(c)	0.82 U	0.78 U	1.48 ^(c)	1.40 ^(c)	2.23 U
C-SB	4	4	1.56 ^(c)	0.81 U	0.77 U	1.35 U	1.42 ^(c)	2.18 U
C-SB	5	4	1.69 ^(c)	0.82 U	0.78 U	1.38 U	1.28 ^(c)	2.23 U
<i>M. nasuta</i> Background	1	2	1.13 B	0.82 U	0.89 ^(c)	1.38 U	2.86 B	2.32 ^(c)
<i>M. nasuta</i> Background	2	2	2.16 B ^(c)	1.61 U	1.89	2.70 U	3.45 B	4.37 U
<i>M. nasuta</i> Background, Replicate 1	3	2	1.76 B ^(c)	1.59 U	1.52 U	2.67 U	3.63 B	4.39 ^(c)
<i>M. nasuta</i> Background, Replicate 2	3	2	1.55 B ^(c)	1.55 U	1.61 ^(c)	2.59 U	3.75 B	4.42
<i>M. nasuta</i> Background, Replicate 3	3	2	1.32 B ^(c)	1.58 U	1.72 ^(c)	2.64 U	3.68 B	4.28 U
<i>M. nasuta</i> Background	4	2	0.88 B ^(c)	0.81 U	1.19	1.35 U	3.68 B	2.34 ^(c)
<i>M. nasuta</i> Background	5	2	0.83 B ^(c)	0.97 ^(c)	0.86 ^(c)	1.38 U	2.68 B	2.38 ^(c)

(a) DL Detection limit.

(b) U Undetected at or above detection limit.

(c) Ratio of confirmation ion to primary ion is outside of the theoretical ratio of 20% established for EPA-CLP programs.

(d) B Analyte detected in sample at less than five times the value in associated method blank.

TABLE I.3. High Molecular Weight Polynuclear Aromatic Hydrocarbons (HPAHs), Wet Weight, in Tissue of *M. nasuta*,
Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> HPAHs (µg/kg wet weight)									
			Fluor- anthrene	Pyrene	Benzo(a)- anthra- cene	Chrysene	Benzo(b)- fluor- anthrene	Benzo(k)- fluor- anthrene	Benzo(a)- pyrene	Indeno- (1,2,3- cd) pyrene	Dibenzo- (a,h) anthra- cene	Benzo- (g,h,i) perylene
Target DL ^(a)			20	20	20	20	20	20	20	20	20	20
January 1993												
OBM COMP	1	1	20 U ^(b)	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U
OBM COMP	2	1	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U
OBM COMP	3	1	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U
OBM COMP	4	1	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U
OBM COMP, Replicate 1	5	1	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U
OBM COMP, Replicate 2	5	1	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U
January 1994												
R-OS	1	4	3.47 ^(c)	1.16 U	1.35 B ^(d)	0.97 ^(c)	1.74 ^(c)	1.36 ^(c)	1.85 ^(c)	2.66 U	2.87 U	2.37 U
R-OS, Replicate 1	2	4	4.58 ^(c)	2.31 U	2.49 B ^(d)	1.28 U	0.54 U	1.62 U	2.23 ^(c)	5.32 U	5.73 U	5.75 ^(c)
R-OS, Replicate 2	2	4	5.04 ^(c)	2.29 U	2.22 B ^(d)	1.84 ^(c)	0.54 U	1.60 U	2.34 ^(c)	5.26 U	5.67 U	4.69 U
R-OS, Replicate 3	2	4	4.47 ^(c)	2.31 U	2.09 B ^(d)	1.28 U	0.54 U	1.62 U	2.20 ^(c)	5.32 U	5.73 U	5.58 ^(c)
R-OS	3	4	3.55 ^(c)	1.16 U	1.29 B ^(d)	1.42	2.00 ^(c)	0.81 U	1.81 ^(c)	2.66 U	2.87 U	2.37 U
R-OS	4	4	4.17 ^(c)	1.18 U	1.50 B	1.72 ^(c)	2.11 ^(c)	0.82 U	2.40 ^(c)	2.71 U	2.92 U	2.42 U
R-OS	5	4	3.25 ^(c)	1.18 U	1.43 B ^(d)	1.06 ^(c)	1.81 ^(c)	0.82 U	1.91 ^(c)	2.71 U	2.92 U	2.42 U
R-BF	1	3	12.1 ^(c)	2.31 U	4.56 B	4.82	11.8	5.28	8.35 ^(c)	6.17	5.73 U	11.6
R-BF	2	2	12.8 ^(c)	14.7 B ^(c)	3.34 B	5.45 B	10.6 B	5.05 B	1.90 B ^(c)	4.30	2.92 U	7.63 B
R-BF	3	3	4.46 U	2.36 U	5.94 B	5.88	12.6	5.51 ^(c)	8.98 ^(c)	5.43 U	5.85 U	8.87
R-BF	4	3	11.0 ^(c)	2.19 U	4.30 B	4.49	9.83	4.31	6.81 ^(c)	5.05 U	5.44 U	8.55
R-BF	5	3	9.78 ^(c)	2.34 U	3.56 B	3.75 ^(c)	9.32	4.45 ^(c)	7.01 ^(c)	5.37 U	5.79 U	6.85 ^(c)
R-AM	1	2	111	121	51.6	75.2	53.0	20.3 B	48.7	17.5	4.00	22.1 B
R-AM	2	3	63.1	57.1 ^(c)	29.2	42.7	27.3	12.4	26.0	11.9	5.85 U	16.6
R-AM	3	3	105 ^(c)	116	52.9	69.2	49.5	19.8	44.9	14.7	5.85 U	20.2
R-AM	4	2	69.8	83.8	30.9	51.5	36.0	14.4 B	32.1	12.9	3.11	16.0 B
R-AM	5	3	83.7 ^(c)	93.1 ^(c)	36.8	55.8	40.9	16.0	36.9	18.0	5.56 U	21.7
C-SB	1	4	6.20 ^(c)	1.16 U	1.74 B	1.98	3.40	1.80 ^(c)	2.53 ^(c)	2.66 U	2.87 U	2.37 U
C-SB	2	4	5.94 ^(c)	1.16 U	1.55 B	1.75	3.08	1.62 ^(c)	2.83 ^(c)	2.66 U	2.87 U	21.8
C-SB	3	4	8.26 ^(c)	1.18 U	1.68 B	2.18	3.43	1.34 ^(c)	2.30 ^(c)	2.71 U	2.92 U	2.42 U
C-SB	4	4	6.71 ^(c)	1.16 U	1.82 B ^(c)	2.38	3.45	1.93 ^(c)	2.41 ^(c)	2.66 U	2.87 U	2.37 U
C-SB	5	4	5.86 ^(c)	1.18 U	1.66 B	2.09	3.51 ^(c)	1.79 ^(c)	2.74 ^(c)	2.71 U	2.92 U	2.42 U

TABLE I.3. (contd)

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> HPAHs (ug/kg wet weight)									
			Fluor-anthene	Pyrene	Benzo(a)-anthracene	Chrysene	Benzo(b)-fluor-anthene	Benzo(k)-fluor-anthene	Benzo(a)-pyrene	Indeno-(1,2,3-cd)pyrene	Dibenzo-(a,h)anthracene	Benzo-(g,h,i)perylene
<i>M. nasuta</i> Background	1	2	9.25 (c)	6.50 B ^(c)	1.49 B ^(c)	2.77 B	3.03 B ^(c)	2.58 B ^(c)	3.42 B ^(c)	3.75	2.92 U	3.65 B ^(c)
<i>M. nasuta</i> Background	2	2	9.88 (c)	5.80 B ^(c)	2.36 B	3.59 B	5.38 B ^(c)	4.73 B ^(c)	4.30 B ^(c)	5.32 U	5.73 U	4.74 U
<i>M. nasuta</i> Background, Replicate 1	3	2	12.4 (c)	5.47 B ^(c)	2.55 B ^(c)	3.95 B	5.59 B	4.64 B ^(c)	4.42 B ^(c)	5.26 U	5.67 U	4.69 U
<i>M. nasuta</i> Background, Replicate 2	3	2	12.2 (c)	5.24 B ^(c)	2.55 B	3.67 B	5.06 B ^(c)	4.56 B ^(c)	4.13 B ^(c)	5.39	5.50 U	6.70 B ^(c)
<i>M. nasuta</i> Background, Replicate 3	3	2	12.2 (c)	9.50 B ^(c)	2.44 B ^(c)	3.78 B	5.55 B ^(c)	4.88 B ^(c)	4.32 B ^(c)	5.21 U	5.61 U	6.25 B ^(c)
<i>M. nasuta</i> Background	4	2	10.2 (c)	7.18 B ^(c)	1.42 B	3.02 B	3.12 B ^(c)	2.67 B ^(c)	3.71 B ^(c)	5.72	2.87 U	3.21 B ^(c)
<i>M. nasuta</i> Background	5	2	8.29 (c)	6.05 B ^(c)	1.27 B ^(c)	2.50 B ^(c)	2.98 B ^(c)	2.36 B ^(c)	1.88 B ^(c)	2.71 U	2.92 U	2.42 U

(a) DL Detection limit.

(b) U Undetected at or above detection limit.

(c) Ratio of confirmation ion to primary ion is outside of the theoretical ratio of 20% established for EPA-CLP programs.

(d) B Analyte detected in sample at less than five times the value in associated method blank.

TABLE I.4. Total Detected Polynuclear Aromatic Hydrocarbons (PAHs), Dry Weight,
in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	<i>M. nasuta</i> PAHs (µg/kg dry weight)		
				Total Low Molecular Weight PAHs	Total High Molecular Weight PAHs	Total PAHs
<u>January 1993</u>						
OBM COMP	1	1	13.6	0	0	0
OBM COMP	2	1	13.2	0	0	0
OBM COMP	3	1	12.3	0	0	0
OBM COMP	4	1	12.3	0	0	0
OBM COMP, Replicate 1	5	1	11.4	0	0	0
OBM COMP, Replicate 2	5	1	12.0	0	0	0
<u>January 1994</u>						
R-OS	1	4	12.7	12	85	97
R-OS, Replicate 1	2	4	12.6	16	119	135
R-OS, Replicate 2	2	4	12.6	20	91	111
R-OS, Replicate 3	2	4	12.6	16	114	130
R-OS	3	4	13.8	25	73	98
R-OS	4	4	12.7	24	94	118
R-OS	5	4	13.4	13	71	84
R-BF	1	3	15.3	29	423	452
R-BF	2	2	14.6	47	449	496
R-BF	3	3	14.8	73	323	396
R-BF	4	3	15.9	23	310	333
R-BF	5	3	14.9	13	300	313
R-AM	1	2	15.8	293	3321	3614
R-AM	2	3	13.8	212	2072	2284
R-AM	3	3	15.4	323	3188	3511
R-AM	4	2	14.5	276	2414	2690
R-AM	5	3	14.7	375	2739	3114
C-SB	1	4	14.4	23	122	146
C-SB	2	4	13.3	26	292	317
C-SB	3	4	14.0	26	137	163
C-SB	4	4	13.6	22	138	160
C-SB	5	4	13.6	22	130	152
<i>M. nasuta</i> Background	1	2	13.5	53	271	324
<i>M. nasuta</i> Background	2	2	12.0	63	300	363
<i>M. nasuta</i> Background, Replicate 1	3	2	13.1	75	299	374
<i>M. nasuta</i> Background, Replicate 2	3	2	13.1	87	379	466
<i>M. nasuta</i> Background, Replicate 3	3	2	13.1	52	375	427
<i>M. nasuta</i> Background	4	2	14.2	57	283	340
<i>M. nasuta</i> Background	5	2	17.7	44	144	187

TABLE I.5. Low Molecular Weight Polynuclear Aromatic Hydrocarbons (LPAHs), Dry Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	<i>M. nasuta</i> LPAHs (µg/kg dry weight)					
				Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
January 1993									
OBM COMP	1	1	13.6	147 U ^(a)	147 U	147 U	147 U	147 U	147 U
OBM COMP	2	1	13.2	152 U	152 U	152 U	152 U	152 U	152 U
OBM COMP	3	1	12.3	163 U	163 U	163 U	163 U	163 U	163 U
OBM COMP	4	1	12.3	163 U	163 U	163 U	163 U	163 U	163 U
OBM COMP, Replicate 1	5	1	11.4	175 U	175 U	175 U	175 U	175 U	175 U
OBM COMP, Replicate 2	5	1	12.0	167 U	167 U	167 U	167 U	167 U	167 U
January 1994									
R-OS	1	4	12.7	6.53	6.37 U	6.06 U	10.6 U	5.82 ^(b)	17.2 U
R-OS, Replicate 1	2	4	12.6	16.3	12.8 U	12.1 U	21.4 U	10.1 U	34.6 U
R-OS, Replicate 2	2	4	12.6	20.3 ^(b)	12.6 U	12.0 U	21.2 U	10.0 U	34.2 U
R-OS, Replicate 3	2	4	12.6	15.8	12.8 U	12.1 U	21.4 U	10.1 U	34.6 U
R-OS	3	4	13.8	8.38	5.85 U	5.56 U	10.7 ^(b)	6.14	15.8 U
R-OS	4	4	12.7	10.2	6.46 U	6.93	10.9 U	6.93	17.6 U
R-OS	5	4	13.4	5.97	6.11 U	5.82 U	10.3 U	7.08	16.6 U
R-BF	1	3	15.3	17.8 B ^(c)	10.5 U	10.0 U	17.7 U	10.8	28.6 U
R-BF	2	2	14.6	5.81 B	5.60 U	6.28 ^(b)	9.43 U	15.4 B	19.5 ^(b)
R-BF	3	3	14.8	23.1 B	11.1 U	10.6 U	19.5	14.4 ^(b)	16.4 ^(b)
R-BF	4	3	15.9	12.9 B ^(b)	9.62 U	9.18 U	16.1 U	10.2 ^(b)	26.1 U
R-BF	5	3	14.9	13.3 B	10.9 U	11.1 U	18.3 U	8.58 U	29.6 U
R-AM	1	2	15.8	9.44 B	12.8	8.42	15.1	153	94.4
R-AM	2	3	13.8	13.9 B	11.9 U	11.3 U	25.1 ^(b)	106	66.8
R-AM	3	3	15.4	15.1 B	14.6 ^(b)	10.1 U	22.8 ^(b)	166	104
R-AM	4	2	14.5	11.4 B	14.7	9.30	20.3	140	79.9
R-AM	5	3	14.7	20.3 B	13.1 ^(b)	13.0	32.1	182	114

TABLE I.5. (contd)

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	<i>M. nasuta</i> LPAHs (µg/kg dry weight)					
				Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
C-SB	1	4	14.4	13.3	5.61 U	5.34 U	9.36 U	10.0	15.1 U
C-SB	2	4	13.3	11.1	6.11 U	7.62 (b)	10.2 U	6.94 (b)	16.5 U
C-SB	3	4	14.0	5.70 (b)	5.84 U	5.56 U	10.5 (b)	10.0 (b)	15.9 U
C-SB	4	4	13.6	11.5 (b)	5.97 U	5.67 U	9.95 U	10.5 (b)	16.1 U
C-SB	5	4	13.6	12.4 (b)	6.03 U	5.74 U	10.1 U	9.41 (b)	16.4 U
<i>M. nasuta</i> Background	1	2	13.5	8.40 B	6.09 U	6.61 (b)	10.3 U	21.2 B	17.2 (b)
<i>M. nasuta</i> Background	2	2	12.0	18.0 B (b)	13.4 U	15.8	22.5 U	28.8 B	36.4 U
<i>M. nasuta</i> Background, Replicate 1	3	2	13.1	13.5 B (b)	12.2 U	11.6 U	20.5 U	27.8 B	33.6 (b)
<i>M. nasuta</i> Background, Replicate 2	3	2	13.1	11.9 B (b)	11.9 U	12.3 (b)	19.8 U	28.7 B	33.9
<i>M. nasuta</i> Background, Replicate 3	3	2	13.1	10.1 B (b)	12.1 U	13.2 (b)	20.2 U	28.2 B	32.8 U
<i>M. nasuta</i> Background	4	2	14.2	6.19 B (b)	5.70 U	8.37	9.50 U	25.9 B	16.5 (b)
<i>M. nasuta</i> Background	5	2	17.7	4.70 B (b)	5.50 (b)	4.87 (b)	7.82 U	15.2 B	13.5 (b)

(a) U Undetected at or above detection limit.

(b) Ratio of confirmation ion to primary ion is outside of the theoretical ratio of 20% established for EPA-CLP programs.

(c) B Analyte detected in sample at less than five times value in associated method blank.

TABLE I.6. High Molecular Weight Polynuclear Aromatic Hydrocarbons (HPAHs), Dry Weight, in Tissue of *M. nasuta*,
Older Bay Mud Study

M. nasuta HPAHs (µg/kg dry weight)															
Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	M. nasuta HPAHs (µg/kg dry weight)											
				Fluor- anthene	Pyrene	Benzo(a)- anthra- cene	Chrysene	Benzo(b)- fluor- anthene	Benzo(k)- fluor- anthene	Benzo(a)- pyrene	Indeno- (1,2,3- cd) pyrene	Dibenzo- (a,h) anthra- cene	Benzo- (g,h,i) perylene		
January 1993															
OBM COMP	1	1	13.6	147	U ^(a)	147	U	147	U	147	U	147	U	147	U
	2	1	13.2	152	U	152	U	152	U	152	U	152	U	152	U
	3	1	12.3	163	U	163	U	163	U	163	U	163	U	163	U
	4	1	12.3	163	U	163	U	163	U	163	U	163	U	163	U
	5	1	11.4	175	U	175	U	175	U	175	U	175	U	175	U
OBM COMP, Replicate 2	5	1	12.0	167	U	167	U	167	U	167	U	167	U	167	U
January 1994															
R-OS	1	4	12.7	27.3 ^(b)	9.13 U	10.6 B ^(c)	7.63 ^(b)	13.7 ^(b)	10.7 ^(b)	14.6 ^(b)	20.9 U	22.6 U	18.6 U	U	
R-OS, Replicate 1	2	4	12.6	36.3 ^(b)	18.3 U	19.7 B ^(b)	10.1 U	4.28 U	12.8 U	17.7 ^(b)	42.2 U	45.4 U	45.6 ^(b)	U	
R-OS, Replicate 2	2	4	12.6	39.9 ^(b)	18.1 U	17.6 B ^(b)	14.6 ^(b)	4.28 U	12.7 U	18.5 ^(b)	41.7 U	44.9 U	37.2 U	U	
R-OS, Replicate 3	2	4	12.6	35.4 ^(b)	18.3 U	16.6 B ^(b)	10.1 U	4.28 U	12.8 U	17.4 ^(b)	42.2 U	45.4 U	44.2 ^(b)	U	
R-OS	3	4	13.8	25.7 ^(b)	8.38 U	9.32 B ^(b)	10.3	14.5 ^(b)	5.85 U	13.1 ^(b)	19.2 U	20.7 U	17.1 U	U	
R-OS	4	4	12.7	32.9 ^(b)	9.30 U	11.8 B	13.6 ^(b)	16.6 ^(b)	6.46 U	18.9 ^(b)	21.4 U	23.0 U	19.1 U	U	
R-OS	5	4	13.4	24.2 ^(b)	8.80 U	10.7 B ^(b)	7.90 ^(b)	13.5 ^(b)	6.11 U	14.2 ^(b)	20.2 U	21.8 U	18.0 U	U	
R-BF	1	3	15.3	79.2 ^(b)	15.1 U	29.8 B	31.5	77.2	34.6	54.6 ^(b)	40.4	37.5 U	75.9	U	
R-BF	2	2	14.6	87.4 ^(b)	100 B ^(b)	22.8 B	37.2 B	72.4 B	34.5 B	13.0 B ^(b)	29.4	19.9 U	52.1 B	B	
R-BF	3	3	14.8	30.2 U	16.0 U	40.2 B	39.8	85.3	37.3 ^(b)	60.8 ^(b)	36.7 U	39.6 U	60.0	U	
R-BF	4	3	15.9	69.1 ^(b)	13.8 U	27.0 B	28.2	61.8	27.1	42.8 ^(b)	31.7 U	34.2 U	53.7	U	
R-BF	5	3	14.9	65.5 ^(b)	15.7 U	23.9 B	25.1 ^(b)	62.5	29.8 ^(b)	47.0 ^(b)	36.0 U	38.8 U	45.9 ^(b)	U	
R-AM	1	2	15.8	703	766	327	476	336	129 B	308	111	25.3	140 B	B	
R-AM	2	3	13.8	457	413 ^(b)	211	309	198	89.8	188	86.2	42.4 U	120	U	
R-AM	3	3	15.4	680	751	343	448	321	128	291	95.2	37.9 U	131	U	
R-AM	4	2	14.5	481	577	213	355	248	99.2 B	221	88.8	21.4	110 B	B	
R-AM	5	3	14.7	569	633 ^(b)	250	379	278	109	251	122	37.8 U	148	U	

TABLE I.6. (contd)

<i>M. nasuta</i> HPAHs (µg/kg dry weight)																								
Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight		Fluor-anthene		Benzo(a)-anthracene		Pyrene	Benzo(a)-anthracene		Chrysene	Benzo(b)-fluor-anthene		Benzo(k)-fluor-anthene		Benzo(a)-pyrene		Indeno-(1,2,3-cd)pyrene	Dibenzo-(a,h)anthracene	Benzo-(g,h,i)perylene			
C-SB	1	4	14.4	43.0	(b)	8.04	U	12.1	B	13.7		23.6		12.5	(b)	17.5	(b)	18.4	U	19.9	U	16.4	U	
C-SB	2	4	13.3	44.8	(b)	8.75	U	11.7	B	13.2		23.2		12.2	(b)	21.4	(b)	20.1	U	21.7	U	165		
C-SB	3	4	14.0	58.9	(b)	8.41	U	12.0	B	15.5		24.4		9.6	(b)	16.4	(b)	19.3	U	20.8	U	17.2	U	
C-SB	4	4	13.6	49.4	(b)	8.55	U	13.4	B(b)	17.5		25.4		14.2	(b)	17.8	(b)	19.6	U	21.1	U	17.5	U	
C-SB	5	4	13.6	43.1	(b)	8.68	U	12.2	B	15.4		25.8	(b)	13.2	(b)	20.1	(b)	19.9	U	21.5	U	17.8	U	
<i>M. nasuta</i> Background	1	2	13.5	68.7	(b)	48.3	B(b)	11.1	B(b)	20.6	B		22.5	B(b)	19.2	B(b)	25.4	B(b)	27.9	21.7	U	27.1	B(b)	
<i>M. nasuta</i> Background	2	2	12.0	82.3	(b)	48.3	B(b)	19.7	B	29.9	B		44.8	B(b)	39.4	B(b)	35.8	B(b)	44.3	U	47.8	U	39.5	U
<i>M. nasuta</i> Background, Replicate 1	3	2	13.1	95.0	(b)	41.9	B(b)	19.5	B(b)	30.3	B		42.8	B(b)	35.6	B(b)	33.9	B(b)	40.3	U	43.4	U	35.9	U
<i>M. nasuta</i> Background, Replicate 2	3	2	13.1	93.5	(b)	40.2	B(b)	19.5	B	28.1	B		38.8	B(b)	34.9	B(b)	31.6	B(b)	41.3	42.1	U	51.3	B(b)	
<i>M. nasuta</i> Background, Replicate 3	3	2	13.1	93.5	(b)	72.8	B(b)	18.7	B(b)	29.0	B		42.5	B(b)	37.4	B(b)	33.1	B(b)	39.9	U	43.0	U	47.9	B(b)
<i>M. nasuta</i> Background	4	2	14.2	71.8	(b)	50.5	B(b)	10.0	B	21.3	B		22.0	B(b)	18.8	B(b)	26.1	B(b)	40.3	20.2	U	22.6	B(b)	
<i>M. nasuta</i> Background	5	2	17.7	47.0	(b)	34.3	B(b)	7.20	B(b)	14.2	B(b)		16.9	B(b)	13.4	B(b)	10.7	B(b)	15.4	U	16.5	U	13.7	U

(a) U Undetected at or above detection limit.

(b) Ratio of confirmation ion to primary ion is outside of the theoretical ratio of 20% established for EPA-CLP programs.

(c) B Analyte detected in sample at less than five times value in associated method blank.

TABLE I.7. Quality Control Data for Low Molecular Weight Polynuclear Aromatic Hydrocarbons (LPAHs), Wet Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> LPAHs ($\mu\text{g/kg}$ wet weight)				
			Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene
Matrix Blank							
Blank		1	20.00 U ^(a)	20.00 U	20.00 U	20.00 U	20.00 U
Blank		2	1.25 ^(b)	1.73 U	1.64 U	2.89 U	1.62 ^(b)
Blank		3	1.79 ^(b)	1.79 U	1.71 U	3.00 U	1.41 U
Blank		4	0.60 U	0.97 U	0.92 U	1.62 U	0.76 U
Matrix Spike							
OBM COMP	3	1	20.00 U	20.00 U	20.00 U	20.00 U	20.00 U
OBM COMP MS	3		167.00	174.30	171.60	173.20	183.70
Concentration Recovered			167.00	174.30	171.60	173.20	183.70
Amount Spiked			181.16	181.16	181.16	181.16	181.16
Percent Recovery			92%	96%	95%	96%	101%
OBM COMP	3	1	20.00 U	20.00 U	20.00 U	20.00 U	20.00 U
OBM COMP MSD	3		167.70	171.90	175.00	175.20	180.20
Concentration Recovered			167.70	171.90	175.00	175.20	180.20
Amount Spiked			181.16	181.16	181.16	181.16	181.16
Percent Recovery			93%	95%	97%	97%	99%
RPD			0%	1%	2%	1%	1%
I-Stat			0.00	0.01	0.01	0.01	0.01
<i>M. nasuta</i> Background							
<i>M. nasuta</i> Background, MS	2	2	2.16 ^(b)	1.61 U	1.89	2.70 U	3.45 B
Concentration Recovered	2		44.7	42.8	45.5	42.9	46.5
Amount Spiked			42.5	42.8	43.6	42.9	46.4
Percent Recovery			47.6	47.6	47.6	47.6	47.6
			89%	90%	92%	90%	97%
QC Sample	5	3	5.56 B	3.29 U	3.13 U	5.51 U	2.59 U
QC Sample, MS	5	3	90.4	85.2	89.7	86.1	83.5
Concentration Recovered			84.8	85.2	89.7	86.1	89.0
Amount Spiked			91.4	91.4	91.4	91.4	89.0
Percent Recovery			93%	93%	98%	94%	91%
QC Sample	4	4	2.44	1.61 U	1.53 U	2.70 U	1.33 ^(b)
QC Sample MS	4	4	50.5	48.3	50.7	48.8	49.1
Concentration Recovered			48.1	48.3	50.7	48.8	56.6
Amount Spiked			50.0	50.0	50.0	50.0	56.6
Percent Recovery			96%	97%	101%	98%	50.0
							113%

TABLE I.7. (contd)

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> LPAHs ($\mu\text{g/kg}$ wet weight)					
			Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
Standard Reference Material								
Certified Value NIST 1974			NC ^(d)	NC	NC	NC	5.6	0.75
			NA ^(e)	NA	NA	NA	±1.4	±0.21
NIST 1974	1		NA	NA	NA	NA	20.00 U	20.00 U
Analytical Replicates								
OBM COMP, Replicate 1	5	1	20.00 U	20.00 U	20.00 U	20.00 U	20.00 U	20.00 U
OBM COMP, Replicate 2	5	1	20.00 U	20.00 U	20.00 U	20.00 U	20.00 U	20.00 U
RPD			NA	NA	NA	NA	NA	NA
I-Stat			NA	NA	NA	NA	NA	NA
<i>M. nasuta</i> Background, Replicate 1	3	2	1.76 ^(b) B	1.59 U	1.52 U	2.67 U	3.63 B	4.39 ^(b)
<i>M. nasuta</i> Background, Replicate 2	3	2	1.55 ^(b) B	1.55 U	1.61 ^(b)	2.59 U	3.75 B	4.42
<i>M. nasuta</i> Background, Replicate 3	3	2	1.32 ^(b) B	1.58 U	1.72 ^(b)	2.64 U	3.68 B	4.28 U
RSD			14%	NA	NA	NA	2%	NA
QC Sample, Replicate 1	2	3	3.58 ^(b) B	3.02 U	2.88 U	5.07 U	2.38 U	8.20 U
QC Sample, Replicate 2	2	3	4.80 B	2.96 U	2.82 U	4.96 U	2.40 ^(b)	8.02 U
QC Sample, Replicate 3	2	3	3.15 B	2.89 U	2.75 U	4.85 U	2.28 U	7.84 U
RSD			22%	NA	NA	NA	NA	NA
R-OS, Replicate 1	2	4	2.06	1.61 U	1.53 U	2.70 U	1.27 U	4.37 U
R-OS, Replicate 2	2	4	2.56 ^(b)	1.59 U	1.52 U	2.67 U	1.26 U	4.32 U
R-OS, Replicate 3	2	4	2.00	1.61 U	1.53 U	2.70 U	1.27 U	4.37 U
RSD			14%	NA	NA	NA	NA	NA

- (a) U Undetected at or above detection limit.
 (b) Ratio of confirmation ion to primary ion is outside of the theoretical ratio of 20% established for EPA-CLP programs.
 (c) B Analyte detected in sample at less than five times value in associated method blank.
 (d) NC Not certified.
 (e) NA Not applicable.

TABLE I.8. Quality Control Data for High Molecular Weight Polynuclear Aromatic Hydrocarbons (HPAHs), Wet Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

M. nasuta HPAHs (µg/kg wet weight)											
Sediment Treatment	Replicate	Analytical Batch	Fluor-anthene	Pyrene	Benzo(a)-anthra-cene	Chrysene	Benzo(b)-fluor-anthene	Benzo(k)-fluor-anthene	Benzo(a)-pyrene	Indeno-(1,2,3-cd)pyrene	Dibenzo-(a,h)-anthra-cene
Method Blanks											
Blank	1	1	20.0 U ^(a)	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U
Blank	2	2	3.45 U	6.24 ^(b)	2.30	2.34 ^(b)	5.66 ^(b)	5.42 ^(b)	2.95 ^(b)	5.70 U	6.14 U
Blank	3	3	3.59 U	2.57 U	2.15 ^(b)	1.42 U	0.61 U	1.8 U	1.72 U	5.91 U	6.37 U
Blank	4	4	1.94 U	1.39 U	1.11 ^(b)	0.77 U	0.33 U	0.97 U	0.93 U	3.20 U	3.45 U
Matrix Spikes											
OBM COMP	3	1	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U
OBM COMP, MS	3	1	182	182	185	174	193	181	186	182	181
Concentration Recovered			182	182	185	174	193	181	186	182	181
Amount Spiked			181	181	181	181	181	181	181	181	181
Percent Recovery			100%	100%	102%	96%	106%	100%	103%	101%	100%
OBM COMP	3	1	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U
OBM COMP, MSD	3	1	185	186	191	177	196	183	189	193	186
Concentration Recovered			185	186	191	177	196	183	189	193	186
Amount Spiked			181	181	181	181	181	181	181	181	181
Percent Recovery			102%	103%	106%	98%	108%	101%	101%	101%	103%
RPD			2%	3%	4%	2%	2%	1%	1%	6%	3%
t-Stat			0.01	0.01	0.02	0.01	0.01	0.01	0.01	0.03	0.01
M. nasuta Background											
M. nasuta Background, MS	2	2	9.88 ^(b)	5.80 ^(b)	2.36 B	3.59 B	5.38 B ^(b)	4.73 B ^(b)	4.30 B ^(b)	5.32 U	5.73 U
Concentration Recovered			52.8	45.3	40.9	42.6	50.2	50.7	50.5	54.4	50.0
Amount Spiked			42.9	39.5	38.5	39.0	44.8	46.0	46.2	54.4	59.7
Percent Recovery			90%	83%	81%	82%	94%	97%	97%	47.6	47.6
QC Sample											
QC Sample, MS	5	3	10.2 ^(b)	4.72 U	5.14 B ^(b)	2.61 U	11.6 ^(b)	6.48 ^(b)	7.62 ^(b)	10.9 U	11.7 U
Concentration Recovered			96.2	63.5	88.7	80.1	100	96.6	94.8	89.1	90.6
Amount Spiked			86.0	63.5	83.6	80.1	88.4	90.1	87.2	89.1	90.6
Percent Recovery			94%	69%	91%	88%	97%	99%	91.4	91.4	91.4
QC Sample											
QC Sample, MS	4	4	8.63 ^(b)	4.22	5.72	4.04 ^(b)	38.7	14.9	21.4	6.63	5.73 U
Concentration Recovered			58.2	57.8	53.8	51.7	100	75.4	78.2	54.2	53.2
Amount Spiked			49.6	53.6	48.1	47.7	61.3	60.5	56.8	47.6	53.2
Percent Recovery			99%	107%	96%	95%	123% ^(d)	121% ^(d)	114%	95%	106%

TABLE I.8. (cont'd)

M. nasuta HPAHs (µg/kg wet weight)												
Sediment Treatment	Replicate	Analytical Batch	Fluor-anthene	Pyrene	Benzo(a)-anthracene	Chrysene	Benzo(b)-fluor-anthene	Benzo(k)-fluor-anthene	Benzo(a)-pyrene	Indeno-(1,2,3-cd)pyrene	Dibenzo-(a,h)-anthracene	Benzo-(g,h,i)perylene
Standard Reference Material												
Certified Value NIST 1974			33.6 ±5.8	34.1 ±3.7	NC ^(a) NA ^(b)	NC NA	6.5 ±1.2	NC NA	2.29 ±0.47	1.80 ±0.33	NC NA	2.47 ±0.28
	1		45.1 ^(a)	39.8	NA	NA	20.0 U	NA	20.0 U	20.0 U	NA	20.0 U
Analytical Replicates												
OBM COMP, Replicate 1	5	1	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U
OBM COMP, Replicate 2	5	1	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U	20.0 U
RPD			NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
I-Stat			NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
M. nasuta Background, Replicate 1	3	2	12.4 ^(a)	5.47 B ^(a)	2.55 B ^(a)	3.95 B	5.59 B	4.64 B ^(a)	4.42 B ^(a)	5.26 U	5.67 U	4.69 U
M. nasuta Background, Replicate 2	3	2	12.2 ^(a)	5.24 B ^(a)	2.55 B	3.67 B	5.06 B ^(a)	4.56 B ^(a)	4.13 B ^(a)	5.39	5.50 U	6.70 B ^(a)
M. nasuta Background, Replicate 3	3	2	12.2 ^(a)	9.50 B ^(a)	2.44 B ^(a)	3.78 B	5.55 B ^(a)	4.88 B ^(a)	4.32 B ^(a)	5.21 U	5.61 U	6.25 B ^(a)
RSD			1%	36% ^(a)	3%	4%	5%	4%	3%	NA	NA	NA
QC Sample, Replicate 1	2	3	10.7 ^(a)	4.34 U	5.04 B ^(a)	4.08 ^(a)	10.7	6.20 ^(a)	6.94 ^(a)	9.98 U	10.8 U	8.90 U
QC Sample, Replicate 2	2	3	11.0 ^(a)	4.25 U	4.95 B ^(a)	3.51 ^(a)	10.1 ^(a)	5.68 ^(a)	6.96 ^(a)	9.77 U	10.5 U	8.71 U
QC Sample, Replicate 3	2	3	10.5 ^(a)	4.15 U	4.47 B ^(a)	2.30 U	9.82 ^(a)	5.51 ^(a)	6.77 ^(a)	9.55 U	10.3 U	8.96 ^(a)
RRSD			2%	NA	6%	NA	4%	6%	2%	NA	NA	NA
R-OS, Replicate 1	2	4	4.58 ^(a)	2.31 U	2.49 B ^(a)	1.28 U	0.54 U	1.62 U	2.23 ^(a)	5.32 U	5.73 U	5.75 ^(a)
R-OS, Replicate 2	2	4	5.04 ^(a)	2.29 U	2.22 B ^(a)	1.84 ^(a)	0.54 U	1.60 U	2.34 ^(a)	5.26 U	5.67 U	4.69 U
R-OS, Replicate 3	2	4	4.47 ^(a)	2.31 U	2.09 B ^(a)	1.28 U	0.54 U	1.62 U	2.20 ^(a)	5.32 U	5.73 U	5.58 ^(a)
RSD			6%	NA	9%	NA	NA	NA	3%	NA	NA	NA

- (a) U Undetected at or above detection limit.
 (b) Ratio of confirmation ion to primary ion is outside of the theoretical ratio of 20% established for EPA-CLP programs.
 (c) B Analyte detected in sample at less than five times value in associated method blank.
 (d) Outside quality control criteria (40-120%) for matrix spike recoveries.
 (e) NC Not certified.
 (f) NA Not applicable.
 (g) Outside quality control criteria (±30%) for SRMs.
 (h) Value exceeds relative precision goal of ≤30%.

TABLE I.9. Surrogate Percent Recoveries for Polynuclear Aromatic Hydrocarbons (PAHs), Including Quality Control Data, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	Surrogate Percent Recoveries							Dibenzo (a,h,i) anthracene d14	
			Naphthalene d8	Acenaphthene d8	Acenaphthene d10	Pyrene d10	Benzo[a]pyrene d12	Chrysene d12			
January 1993											
OBM COMP	1	1	NA ^(a)	75	NA	77	87	NA	NA		
	2	1	NA	80	NA	81	95	NA	NA		
	3	1	NA	75	NA	73	84	NA	NA		
	4	1	NA	78	NA	79	91	NA	NA		
	5	1	NA	71	NA	72	84	NA	NA		
	5	1	NA	71	NA	72	84	NA	NA		
January 1994											
R-OS	1	4	77	NA	82	NA	NA	86	91		
R-OS, Replicate 1	2	4	69	NA	74	NA	NA	78	82		
R-OS, Replicate 2	2	4	73	NA	77	NA	NA	80	87		
R-OS, Replicate 3	2	4	71	NA	78	NA	NA	83	83		
R-OS	3	4	72	NA	77	NA	NA	81	86		
R-OS	4	4	78	NA	81	NA	NA	79	85		
R-OS	5	4	73	NA	77	NA	NA	79	87		
R-BF	1	3	65	NA	72	NA	NA	74	84		
R-BF	2	2	74	NA	79	NA	NA	82	81		
R-BF	3	3	62	NA	65	NA	NA	66	74		
R-BF	4	3	71	NA	75	NA	NA	84	77		
R-BF	5	3	78	NA	79	NA	NA	83	90		
R-AM	1	2	73	NA	78	NA	NA	82	87		
R-AM	2	3	75	NA	78	NA	NA	79	85		
R-AM	3	3	70	NA	73	NA	NA	79	84		
R-AM	4	2	72	NA	74	NA	NA	78	79		
R-AM	5	3	76	NA	81	NA	NA	88	87		
C-SB	1	4	70	NA	82	NA	NA	78	80		
C-SB	2	4	74	NA	80	NA	NA	84	89		
C-SB	3	4	72	NA	79	NA	NA	74	80		
C-SB	4	4	59	NA	67	NA	NA	69	66		
C-SB	5	4	64	NA	70	NA	NA	70	71		

TABLE I.9. (contd)

Sediment Treatment	Replicate	Analytical Batch	Surrogate Percent Recoveries						
			Naphthalene d8	Acenaphthene d8	Acenaphthene d10	Pyrene d10	Benz[a]pyrene d12	Chrysene d12	Dibenzo (a,h,i) anthracene d14
<i>M. nasuta</i> Background	1	2	69	NA	73	NA	NA	77	68
<i>M. nasuta</i> Background	2	2	65	NA	71	NA	NA	72	69
<i>M. nasuta</i> Background, Replicate 1	3	2	70	NA	77	NA	NA	81	77
<i>M. nasuta</i> Background, Replicate 2	3	2	62	NA	67	NA	NA	72	69
<i>M. nasuta</i> Background, Replicate 3	3	2	70	NA	72	NA	NA	73	68
<i>M. nasuta</i> Background	4	2	76	NA	81	NA	NA	84	75
<i>M. nasuta</i> Background	5	2	77	NA	79	NA	NA	82	81
<u>Quality Control Data</u>									
<u>Method Blanks</u>									
Blank		1	NA	84	NA	91	99	NA	NA
Blank		2	82	NA	77	NA	NA	77	47
Blank		3	76	NA	73	NA	NA	71	69
Blank		4	58	NA	57	NA	NA	73	78
<u>Matrix Spikes</u>									
OBM COMP MS	3	1	NA	80	NA	79	93	NA	NA
OBM COMP MSD	3	1	NA	77	NA	79	93	NA	NA
<i>M. nasuta</i> Background	2	2	65	NA	71	NA	NA	72	69
<i>M. nasuta</i> Background, MS	2	2	67	NA	72	NA	NA	76	73
QC Sample	5	3	73	NA	76	NA	NA	75	85
QC Sample, MS	5	3	76	NA	78	NA	NA	81	86
QC Sample	4	4	71	NA	77	NA	NA	91	75
QC Sample, MS	4	4	76	NA	80	NA	NA	85	90
<u>Standard Reference Material</u>									
SRM NIST 1974	1	1	NA	87	NA	87	104	NA	NA

TABLE I.9. (contd)

Sediment Treatment	Surrogate Percent Recoveries								Dibenzo (a,h,i) anthracene d14
	Replicate	Analytical Batch	Naphthalene d8	Acenaphthene d8	Acenaphthene d10	Pyrene d10	Benzo[a]pyrene d12	Chrysene d12	
Analytical Replicates									
OBM COMP, Replicate 1	5	1	NA	71	NA	72	84	NA	NA
OBM COMP, Replicate 2	5	1	NA	71	NA	72	84	NA	NA
<i>M. nasuta</i> Background, Replicate 1	3	2	70	NA	77	NA	NA	81	77
<i>M. nasuta</i> Background, Replicate 2	3	2	62	NA	67	NA	NA	72	69
<i>M. nasuta</i> Background, Replicate 3	3	2	70	NA	72	NA	NA	73	68
QC Sample, Replicate 1	2	3	66	NA	71	NA	NA	83	75
QC Sample, Replicate 2	2	3	63	NA	66	NA	NA	68	77
QC Sample, Replicate 3	2	3	71	NA	77	NA	NA	81	85
R-OS, Replicate 1	2	4	69	NA	74	NA	NA	78	82
R-OS, Replicate 2	2	4	73	NA	77	NA	NA	80	87
R-OS, Replicate 3	2	4	71	NA	78	NA	NA	83	83

(a) NA Not applicable.

TABLE I.10. Chlorinated Pesticides (alphabetical, Aldrin - 4,4'-DDT), Wet Weight, in Tissue of *M. nasuta*,
Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> Pesticides (µg/kg wet weight)										
			Aldrin	Alpha BHC	Beta BHC	Delta BHC	Gamma BHC	Alpha Chlordane	Gamma Chlordane	Tech-Chlordane	4,4'-DDD	4,4'-DDE	4,4'-DDT
Target DL ^(a)			2	2	2	2	2	2	2	30	2	2	2
January 1993													
OBM COMP	1	1	0.36 U ^(b)	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0 U	12.1	0.28 U	2.15
OBM COMP	2	1	0.36 U	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0 U	25.2	0.28 U	0.85
OBM COMP	3	1	0.36 U	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0 U	31.1	0.28 U	1.07
OBM COMP	4	1	0.36 U	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0 U	34.7	0.28 U	0.71 U
OBM COMP, Replicate 1	5	1	0.36 U	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0 U	40.1	2.04	0.71 U
OBM COMP, Replicate 2	5	1	0.36 U	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0 U	41.3	1.67	0.71 U
January 1994													
OBM COMP	1	3	0.18 U	0.50 U	0.50 U	0.50 U	0.24 U	NA ^(c)	NA	30 U	0.46 U	0.14 U	0.35 U
OBM COMP	2	3	0.18 U	0.50 U	0.50 U	0.50 U	0.24 U	NA	NA	30 U	0.46 U	0.14 U	0.35 U
OBM COMP	3	3	0.17 U	0.48 U	0.48 U	0.48 U	0.23 U	NA	NA	30 U	0.45 U	0.13 U	0.34 U
OBM COMP	4	3	0.18 U	0.50 U	0.50 U	0.50 U	0.24 U	NA	NA	30 U	0.47 U	0.14 U	0.36 U
OBM COMP	5	3	0.18 U	0.50 U	0.50 U	0.50 U	0.24 U	NA	NA	30 U	0.47 U	0.14 U	0.36 U
R-OS	1	4	0.09 U	0.25 U	0.25 U	0.25 U	0.13	NA	NA	30 U	0.23 U	0.07 U	0.17 U
R-OS, Replicate 1	2	4	0.18 U	0.49 U	0.49 U	0.49 U	0.24 U	NA	NA	30 U	0.46 U	0.14 U	0.35 U
R-OS, Replicate 2	2	4	0.18 U	0.49 U	0.49 U	0.49 U	0.23 U	NA	NA	30 U	0.46 U	0.14 U	0.35 U
R-OS, Replicate 3	2	4	0.18 U	0.49 U	0.49 U	0.49 U	0.24 U	NA	NA	30 U	0.46 U	0.14 U	0.35 U
R-OS	3	4	0.09 U	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	0.07 U	0.17 U
R-OS	4	4	0.09 U	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	0.07 U	0.18 U
R-OS	5	4	0.09 U	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	0.07 U	0.18 U
R-BF	1	3	0.17 U	0.48 U	0.48 U	0.48 U	0.23 U	NA	NA	30 U	0.45 U	0.13 U	0.34 U
R-BF	2	2	0.09 U	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	1.14	1.81
R-BF	3	3	0.18 U	0.50 U	0.50 U	0.50 U	0.24 U	NA	NA	30 U	0.47 U	0.14 U	0.36 U
R-BF	4	3	0.17 U	0.47 U	0.47 U	0.47 U	0.22 U	NA	NA	30 U	0.44 U	0.13 U	0.33 U
R-BF	5	3	0.18 U	0.50 U	0.50 U	0.50 U	0.24 U	NA	NA	30 U	0.46 U	0.14 U	0.35 U
R-AM	1	2	0.09 U	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	0.07 U	2.91
R-AM	2	3	0.18 U	0.50 U	0.50 U	0.50 U	0.24 U	NA	NA	30 U	0.46 U	0.14 U	0.35 U
R-AM	3	3	0.18 U	0.50 U	0.50 U	0.50 U	0.24 U	NA	NA	30 U	0.47 U	0.14 U	0.36 U
R-AM	4	2	0.09 U	0.24 U	0.24 U	0.24 U	0.12 U	NA	NA	30 U	0.23 U	1.03	0.17 U
R-AM	5	3	0.17 U	0.48 U	0.48 U	0.48 U	0.23 U	NA	NA	30 U	0.45 U	0.13 U	0.34 U

TABLE I.10. (contd)

Sediment Treatment	Replicate	<i>M. nasuta</i> Pesticides (µg/kg wet weight)												
		Analytical Batch		Aldrin	Alpha BHC	Beta BHC	Delta BHC	Gamma BHC	Alpha Chlordane	Gamma Chlordane	Tech-Chlordane	4,4'-DDD	4,4'-DDE	4,4'-DDT
C-SB	1	4	4	0.09 U	0.25 U	0.25 U	0.25 U	0.15	NA	NA	30 U	0.23 U	0.07 U	0.17 U
C-SB	2	4	4	0.09 U	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	0.07 U	0.17 U
C-SB	3	4	4	0.09 U	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	0.07 U	0.17 U
C-SB	4	4	4	0.01 U	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	0.07 U	0.17 U
C-SB	5	4	4	0.09 U	0.25 U	0.25 U	0.25 U	0.14	NA	NA	30 U	0.23 U	0.07 U	0.18 U
<i>M. nasuta</i> Background	1	2	2	1.93	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	0.54	2.06
<i>M. nasuta</i> Background	2	2	2	0.18 U	0.49 U	0.49 U	0.49 U	0.24 U	NA	NA	30 U	0.46 U	0.38	4.06
<i>M. nasuta</i> Background, Replicate 1	3	2	2	2.35	0.49 U	0.49 U	0.49 U	0.23 U	NA	NA	30 U	0.46 U	0.14 U	2.88
<i>M. nasuta</i> Background, Replicate 2	3	2	2	2.79	0.47 U	0.47 U	0.47 U	0.23 U	NA	NA	30 U	0.44 U	0.53	3.46
<i>M. nasuta</i> Background, Replicate 3	3	2	2	2.95	0.48 U	0.48 U	0.48 U	0.23 U	NA	NA	30 U	0.45 U	0.13 U	2.55
<i>M. nasuta</i> Background	4	2	2	0.09 U	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	0.31	1.17
<i>M. nasuta</i> Background	5	2	2	1.38	0.25 U	0.25 U	0.25 U	0.12 U	NA	NA	30 U	0.23 U	0.55	1.28

(a) DL Detection limit.

(b) U Undetected at or above detection limit.

(c) NA Not applicable.

**TABLE I.11. Chlorinated Pesticides (alphabetical, Dieldrin - Toxaphene), Wet Weight, in Tissue of *M. nasuta*,
Older Bay Mud Study**

Sediment Treatment	Replicate	Analytical Batch	Dieldrin	<i>M. nasuta</i> Pesticides ($\mu\text{g/kg}$ wet weight)							Toxa- phene
				Endo- Sulfan I	Endo- Sulfan II	Endo- Sulfan- Sulfate	Endrin	Endrin Aldehyde	Hepta- chlor	Hepta- chlor epoxide	
Target DL ^(a)			2	2	2	2	2	2	2	2	30
January 1993											
OBM COMP	1	1	1.0 U ^(b)	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U	16.0 U
OBM COMP	2	1	2.57	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U	16.0 U
OBM COMP	3	1	1.94	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U	16.0 U
OBM COMP	4	1	2.64	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U	16.0 U
OBM COMP, Replicate 1	5	1	3.21	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U	16.0 U
OBM COMP, Replicate 2	5	1	3.38	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U	16.0 U
January 1994											
OBM COMP	1	3	0.31 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.15 U	0.48 U	30 U
OBM COMP	2	3	0.31 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.15 U	0.48 U	30 U
OBM COMP	3	3	0.31 U	0.48 U	0.48 U	0.48 U	0.48 U	0.48 U	0.14 U	0.47 U	30 U
OBM COMP	4	3	0.32 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.15 U	0.49 U	30 U
OBM COMP	5	3	0.32 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.15 U	0.49 U	30 U
R-OS	1	4	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.07 U	0.24 U	30 U
R-OS, Replicate 1	2	4	0.31 U	0.49 U	0.49 U	0.49 U	0.49 U	0.49 U	0.15 U	0.48 U	30 U
R-OS, Replicate 2	2	4	0.31 U	0.49 U	0.49 U	0.49 U	0.49 U	0.49 U	0.15 U	0.47 U	30 U
R-OS, Replicate 3	2	4	0.31 U	0.49 U	0.49 U	0.49 U	0.49 U	0.49 U	0.15 U	0.48 U	30 U
R-OS	3	4	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.07 U	0.24 U	30 U
R-OS	4	4	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.08 U	0.24 U	30 U
R-OS	5	4	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.08 U	0.24 U	30 U
R-BF	1	3	0.31 U	0.48 U	0.48 U	0.48 U	0.48 U	0.48 U	0.14 U	0.47 U	30 U
R-BF	2	2	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.08 U	0.24 U	30 U
R-BF	3	3	0.32 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.15 U	0.49 U	30 U
R-BF	4	3	0.30 U	0.47 U	0.47 U	0.47 U	0.47 U	0.47 U	0.14 U	0.46 U	30 U
R-BF	5	3	0.31 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.15 U	0.48 U	30 U
R-AM	1	2	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.07 U	0.24 U	30 U
R-AM	2	3	0.31 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.15 U	0.48 U	30 U
R-AM	3	3	0.32 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.15 U	0.49 U	30 U
R-AM	4	2	0.15 U	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U	0.07 U	0.24 U	30 U
R-AM	5	3	0.30 U	0.48 U	0.48 U	0.48 U	0.48 U	0.48 U	0.14 U	0.47 U	30 U

TABLE I.11. (contd)

Sediment Treatment	<i>M. nasuta</i> Pesticides (µg/kg wet weight)										
	Replicate	Analytical Batch	Dieldrin	Endo-Sulfan I	Endo-Sulfan II	Endo-Sulfan-Sulfate	Endrin	Endrin Aldehyde	Hepta-chlor	Hepta-chlor epoxide	Toxa- phone
C-SB	1	4	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.07 U	0.24 U	30 U
C-SB	2	4	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.07 U	0.24 U	30 U
C-SB	3	4	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.07 U	0.24 U	30 U
C-SB	4	4	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.01 U	0.24 U	30 U
C-SB	5	4	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.08 U	0.24 U	30 U
<i>M. nasuta</i> Background	1	2	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.08 U	0.24 U	30 U
<i>M. nasuta</i> Background	2	2	0.31 U	0.49 U	0.49 U	0.49 U	0.49 U	0.49 U	0.15 U	0.48 U	30 U
<i>M. nasuta</i> Background, Replicate 1	3	2	0.31 U	0.49 U	0.49 U	0.49 U	0.49 U	0.49 U	0.15 U	0.47 U	30 U
<i>M. nasuta</i> Background, Replicate 2	3	2	0.30 U	0.47 U	0.47 U	0.47 U	0.47 U	0.47 U	0.14 U	0.46 U	30 U
<i>M. nasuta</i> Background, Replicate 3	3	2	0.31 U	0.48 U	0.48 U	0.48 U	0.48 U	0.48 U	0.14 U	0.47 U	30 U
<i>M. nasuta</i> Background	4	2	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.07 U	0.24 U	30 U
<i>M. nasuta</i> Background	5	2	0.16 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.08 U	0.24 U	30 U

(a) DL Detection limit.

(b) U Undetected at or above detection limit.

TABLE I.12. Chlorinated Pesticides (alphabetical, Aldrin - 4,4'-DDT), Dry Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight		M. nasuta Pesticides (µg/kg dry weight)											
			Aldrin	Alpha BHC	Beta BHC	Delta BHC	Gamma BHC	Alpha-Chlor-dane		Gamma-Chlor-dane	Tech-Chlor-dane	4,4'-DDD	4,4'-DDE	4,4'-DDT		
January 1993																
OBM COMP	1	1	13.6	2.6 U ^(a)	7.4 U	7.4 U	7.4 U	4.0 U	7.4 U	7.4 U	95.6 U	89.0	2.1 U	15.8		
OBM COMP	2	1	13.2	2.7 U	7.6 U	7.6 U	7.6 U	3.6 U	7.6 U	7.6 U	98.5 U	191	2.1 U	6.4		
OBM COMP	3	1	12.3	2.9 U	8.1 U	8.1 U	8.1 U	3.9 U	8.1 U	8.1 U	106 U	253	2.3 U	8.7		
OBM COMP	4	1	11.4	3.2 U	8.8 U	8.8 U	8.8 U	4.2 U	8.8 U	8.8 U	114 U	304	2.5 U	6.2 U		
OBM COMP, Replicate 1	5	1	12.0	3.0 U	8.3 U	8.3 U	8.3 U	4.0 U	8.3 U	8.3 U	108 U	334	17.0	5.9 U		
OBM COMP, Replicate 2	5	1	12.0	3.0 U	8.3 U	8.3 U	8.3 U	4.0 U	8.3 U	8.3 U	108 U	344	13.9	5.9 U		
January 1994																
OBM COMP	1	3	14.0	1.28 U	3.6 U	3.6 U	3.6 U	1.7 U	NA ^(b)	NA	210 U	3.3 U	1.00 U	2.5 U		
OBM COMP	2	3	13.2	1.37 U	3.8 U	3.8 U	3.8 U	1.8 U	NA	NA	230 U	3.5 U	1.06 U	2.7 U		
OBM COMP	3	3	12.2	1.39 U	3.9 U	3.9 U	3.9 U	1.9 U	NA	NA	250 U	3.7 U	1.06 U	2.8 U		
OBM COMP	4	3	14.3	1.26 U	3.5 U	3.5 U	3.5 U	1.7 U	NA	NA	210 U	3.3 U	0.98 U	2.5 U		
OBM COMP	5	3	14.5	1.24 U	3.4 U	3.4 U	3.4 U	1.7 U	NA	NA	210 U	3.2 U	0.96 U	2.5 U		
R-OS	1	4	12.7	0.71 U	2.0 U	2.0 U	2.0 U	1.0	NA	NA	240 U	1.8 U	0.6 U	1.3 U		
R-OS, Replicate 1	2	4	12.6	1.43 U	3.9 U	3.9 U	3.9 U	1.9 U	NA	NA	240 U	3.6 U	1.11 U	2.8 U		
R-OS, Replicate 2	2	4	12.6	1.43 U	3.9 U	3.9 U	3.9 U	1.8 U	NA	NA	240 U	3.6 U	1.11 U	2.8 U		
R-OS, Replicate 3	2	4	12.6	1.43 U	3.9 U	3.9 U	3.9 U	1.9 U	NA	NA	240 U	3.6 U	1.11 U	2.8 U		
R-OS	3	4	13.8	0.65 U	1.8 U	1.8 U	1.8 U	0.9 U	NA	NA	220 U	1.7 U	0.5 U	1.2 U		
R-OS	4	4	12.7	0.71 U	2.0 U	2.0 U	2.0 U	0.9 U	NA	NA	240 U	1.8 U	0.6 U	1.4 U		
R-OS	5	4	13.4	0.67 U	1.9 U	1.9 U	1.9 U	0.9 U	NA	NA	220 U	1.7 U	0.5 U	1.3 U		
R-BF	1	3	15.3	1.11 U	3.1 U	3.1 U	3.1 U	1.5 U	NA	NA	200 U	2.9 U	0.85 U	2.2 U		
R-BF	2	2	14.6	0.61 U	1.7 U	1.7 U	1.7 U	0.8 U	NA	NA	210 U	1.6 U	7.79	12.4		
R-BF	3	3	14.8	1.22 U	3.4 U	3.4 U	3.4 U	1.6 U	NA	NA	200 U	3.2 U	0.95 U	2.4 U		
R-BF	4	3	15.9	1.07 U	3.0 U	3.0 U	3.0 U	1.4 U	NA	NA	190 U	2.8 U	0.82 U	2.1 U		
R-BF	5	3	14.9	1.21 U	3.4 U	3.4 U	3.4 U	1.6 U	NA	NA	200 U	3.1 U	0.94 U	2.3 U		
R-AM	1	2	15.8	0.6 U	1.6 U	1.6 U	1.6 U	0.8 U	NA	NA	190 U	1.5 U	0.4 U	18.4		
R-AM	2	3	13.8	1.30 U	3.6 U	3.6 U	3.6 U	1.7 U	NA	NA	220 U	3.3 U	1.01 U	2.5 U		
R-AM	3	3	15.4	1.17 U	3.2 U	3.2 U	3.2 U	1.6 U	NA	NA	190 U	3.0 U	0.91 U	2.3 U		
R-AM	4	2	14.5	0.62 U	1.7 U	1.7 U	1.7 U	0.8 U	NA	NA	210 U	1.6 U	7.09	1.2 U		
R-AM	5	3	14.7	1.16 U	3.3 U	3.3 U	3.3 U	1.6 U	NA	NA	200 U	3.1 U	0.88 U	2.3 U		

TABLE I.12. (contd)

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	M. nasuta Pesticides (µg/kg dry weight)									
				Alpha-Chlor-dane					Gamma-Chlor-dane				
				Alpha BHC	Beta BHC	Delta BHC	Gamma BHC	Alpha-Chlor-dane	Gamma-Chlor-dane	Tech-Chlor-dane	4,4'-DDD	4,4'-DDE	4,4'-DDT
C-SB	1	4	14.4	1.7 U	1.7 U	1.7 U	1.0	NA	NA	210 U	1.6 U	0.5 U	1.2 U
C-SB	2	4	13.3	1.9 U	1.9 U	1.9 U	0.9 U	NA	NA	230 U	1.7 U	0.5 U	1.3 U
C-SB	3	4	14.0	1.8 U	1.8 U	1.8 U	0.9 U	NA	NA	210 U	1.6 U	0.5 U	1.2 U
C-SB	4	4	13.6	1.8 U	1.8 U	1.8 U	0.9 U	NA	NA	220 U	1.7 U	0.5 U	1.3 U
C-SB	5	4	13.6	1.8 U	1.8 U	1.8 U	1.0	NA	NA	220 U	1.7 U	0.5 U	1.3 U
M. nasuta Background	1	2	13.5	1.9 U	1.9 U	1.9 U	0.9 U	NA	NA	220 U	1.7 U	4.01	15.3
M. nasuta Background	2	2	12.0	4.1 U	4.1 U	4.1 U	2.0 U	NA	NA	250 U	3.8 U	3.17	33.8
M. nasuta Background, Replicate 1	3	2	13.1	3.8 U	3.8 U	3.8 U	1.8 U	NA	NA	230 U	3.5 U	1.07 U	22.1
M. nasuta Background, Replicate 2	3	2	13.1	3.6 U	3.6 U	3.6 U	1.8 U	NA	NA	230 U	3.4 U	4.06	26.5
M. nasuta Background, Replicate 3	3	2	13.1	3.7 U	3.7 U	3.7 U	1.8 U	NA	NA	230 U	3.4 U	1.00 U	19.5
M. nasuta Background	4	2	14.2	1.8 U	1.8 U	1.8 U	0.8 U	NA	NA	210 U	1.6 U	2.18	8.23
M. nasuta Background	5	2	17.7	1.4 U	1.4 U	1.4 U	0.7 U	NA	NA	170 U	1.3 U	3.12	7.25

(a) U Undetected at or above detection limit.

(b) NA Not applicable.

TABLE I.13. Chlorinated Pesticides (alphabetical Dieldrin - Toxaphene), Dry Weight, in Tissue of *M. nasuta*,
Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	M. nasuta Pesticides (µg/kg dry weight)									
				Dieldrin	Endo-Sulfan I	Endo-Sulfan II	Endo-Sulfate	Endrin	Aldehyde	Hepta-chlor	Hepta-chlor epoxide	Toxa-phene	
January 1993													
OBM COMP	1	1	13.6	7.4 U ^(a)	7.4 U	7.4 U	7.4 U	7.4 U	7.4 U	7.4 U	2.2 U	7.2 U	118 U
OBM COMP	2	1	13.2	19.5	7.6 U	7.6 U	7.6 U	7.6 U	7.6 U	7.6 U	2.3 U	7.4 U	121 U
OBM COMP	3	1	12.3	15.8	8.1 U	8.1 U	8.1 U	8.1 U	8.1 U	8.1 U	2.4 U	8.0 U	130 U
OBM COMP	4	1	11.4	23.2	8.8 U	8.8 U	8.8 U	8.8 U	8.8 U	8.8 U	2.6 U	8.6 U	140 U
OBM COMP, Replicate 1	5	1	12.0	26.8	8.3 U	8.3 U	8.3 U	8.3 U	8.3 U	8.3 U	2.5 U	8.2 U	133 U
OBM COMP, Replicate 2	5	1	12.0	28.2	8.3 U	8.3 U	8.3 U	8.3 U	8.3 U	8.3 U	2.5 U	8.2 U	133 U
January 1994													
OBM COMP	1	3	14.0	2.2 U	3.6 U	3.6 U	3.6 U	3.6 U	3.6 U	3.6 U	1.07 U	3.4 U	210 U
OBM COMP	2	3	13.2	2.4 U	3.8 U	3.8 U	3.8 U	3.8 U	3.8 U	3.8 U	1.14 U	3.6 U	230 U
OBM COMP	3	3	12.2	2.5 U	3.9 U	3.9 U	3.9 U	3.9 U	3.9 U	3.9 U	1.15 U	3.8 U	250 U
OBM COMP	4	3	14.3	2.2 U	3.5 U	3.5 U	3.5 U	3.5 U	3.5 U	3.5 U	1.05 U	3.4 U	210 U
OBM COMP	5	3	14.5	2.2 U	3.4 U	3.4 U	3.4 U	3.4 U	3.4 U	3.4 U	1.03 U	3.4 U	210 U
R-OS	1	4	12.7	1.3 U	2.0 U	2.0 U	2.0 U	2.0 U	2.0 U	2.0 U	0.6 U	1.9 U	240 U
R-OS, Replicate 1	2	4	12.6	2.5 U	3.9 U	3.9 U	3.9 U	3.9 U	3.9 U	3.9 U	1.19 U	3.8 U	240 U
R-OS, Replicate 2	2	4	12.6	2.5 U	3.9 U	3.9 U	3.9 U	3.9 U	3.9 U	3.9 U	1.19 U	3.7 U	240 U
R-OS, Replicate 3	2	4	12.6	2.5 U	3.9 U	3.9 U	3.9 U	3.9 U	3.9 U	3.9 U	1.19 U	3.8 U	240 U
R-OS	3	4	13.8	1.2 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	0.5 U	1.7 U	220 U
R-OS	4	4	12.7	1.3 U	2.0 U	2.0 U	2.0 U	2.0 U	2.0 U	2.0 U	0.6 U	1.9 U	240 U
R-OS	5	4	13.4	1.2 U	1.9 U	1.9 U	1.9 U	1.9 U	1.9 U	1.9 U	0.6 U	1.8 U	220 U
R-BF	1	3	15.3	2.0 U	3.1 U	3.1 U	3.1 U	3.1 U	3.1 U	3.1 U	0.92 U	3.1 U	200 U
R-BF	2	2	14.6	1.1 U	1.7 U	1.7 U	1.7 U	1.7 U	1.7 U	1.7 U	0.5 U	1.6 U	210 U
R-BF	3	3	14.8	2.2 U	3.4 U	3.4 U	3.4 U	3.4 U	3.4 U	3.4 U	1.01 U	3.3 U	200 U
R-BF	4	3	15.9	1.9 U	3.0 U	3.0 U	3.0 U	3.0 U	3.0 U	3.0 U	0.88 U	2.9 U	190 U
R-BF	5	3	14.9	2.1 U	3.4 U	3.4 U	3.4 U	3.4 U	3.4 U	3.4 U	1.01 U	3.2 U	200 U
R-AM	1	2	15.8	1.0 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	1.6 U	0.4 U	1.5 U	190 U
R-AM	2	3	13.8	2.2 U	3.6 U	3.6 U	3.6 U	3.6 U	3.6 U	3.6 U	1.09 U	3.5 U	220 U
R-AM	3	3	15.4	2.1 U	3.2 U	3.2 U	3.2 U	3.2 U	3.2 U	3.2 U	0.97 U	3.2 U	190 U
R-AM	4	2	14.5	1.0 U	1.7 U	1.7 U	1.7 U	1.7 U	1.7 U	1.7 U	0.5 U	1.7 U	210 U
R-AM	5	3	14.7	2.0 U	3.3 U	3.3 U	3.3 U	3.3 U	3.3 U	3.3 U	0.95 U	3.2 U	200 U

TABLE I.13. (contd)

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	<i>M. nasuta</i> Pesticides (µg/kg dry weight)								
				Dieldrin	Endo-Sulfan I	Endo-Sulfan II	Endo-Sulfate	Endrin	Aldehyde	Hepta-chlor	Hepta-chlor epoxide	Toxa-phene
C-SB	1	4	14.4	1.1 U	1.7 U	1.7 U	1.7 U	1.7 U	1.7 U	0.5 U	1.7 U	210 U
C-SB	2	4	13.3	1.2 U	1.9 U	1.9 U	1.9 U	1.9 U	1.9 U	0.5 U	1.8 U	230 U
C-SB	3	4	14.0	1.1 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	0.5 U	1.7 U	210 U
C-SB	4	4	13.6	1.2 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	0.1 U	1.8 U	220 U
C-SB	5	4	13.6	1.2 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	0.6 U	1.8 U	220 U
<i>M. nasuta</i> Background	1	2	13.5	1.2 U	1.9 U	1.9 U	1.9 U	1.9 U	1.9 U	0.6 U	1.8 U	220 U
<i>M. nasuta</i> Background	2	2	12.0	2.6 U	4.1 U	4.1 U	4.1 U	4.1 U	4.1 U	1.25 U	4.0 U	250 U
<i>M. nasuta</i> Background, Replicate 1	3	2	13.1	2.4 U	3.8 U	3.8 U	3.8 U	3.8 U	3.8 U	1.15 U	3.6 U	230 U
<i>M. nasuta</i> Background, Replicate 2	3	2	13.1	2.3 U	3.6 U	3.6 U	3.6 U	3.6 U	3.6 U	1.07 U	3.5 U	230 U
<i>M. nasuta</i> Background, Replicate 3	3	2	13.1	2.4 U	3.7 U	3.7 U	3.7 U	3.7 U	3.7 U	1.07 U	3.6 U	230 U
<i>M. nasuta</i> Background	4	2	14.2	1.1 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	0.5 U	1.7 U	210 U
<i>M. nasuta</i> Background	5	2	17.7	0.9 U	1.4 U	1.4 U	1.4 U	1.4 U	1.4 U	0.5 U	1.4 U	170 U

(a) U Undetected at or above detection limit.

TABLE I.14. Quality Control Data for Chlorinated Pesticides (alphabetical, Aldrin - 4,4'-DDT), Wet Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	<i>M. nasuta</i> Pesticides (µg/kg wet weight)										
	Replicate	Analytical Batch	Aldrin	Alpha BHC	Beta BHC	Delta BHC	Gamma BHC	Alpha-Chlor-dane	Gamma-Chlor-dane	Tech-Chlor-dane	
Method Blanks											
Blank	1		0.36 U ^(a)	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0	0.94 U
Blank	2		0.19 U	0.53 U	0.53 U	0.53 U	0.25 U	NA ^(b)	NA	30 U	0.15 U
Blank	3		0.20 U	0.55 U	0.55 U	0.55 U	0.26 U	NA	NA	30 U	0.15 U
Blank	4		0.11 U	0.30 U	0.30 U	0.30 U	0.14 U	NA	NA	30 U	0.08 U
Matrix Spikes											
OBM COMP	3	1	0.36 U	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0	0.28 U
OBM COMP, MS	3	1	9.94	NA	NA	NA	9.24	NA	NA	NA	NA
Concentration Recovered			9.94	NA	NA	NA	9.24	NA	NA	NA	NA
Amount Spiked			9.00	NS ^(c)	NS	NS	9.00	NS	NS	NS	NS
Percent Recovery			110%	NA	NA	NA	103%	NA	NA	NA	NA
OBM COMP	3	1	0.36 U	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0	0.28 U
OBM COMP, MSD	3	1	10.40	NA	NA	NA	9.48	NA	NA	NA	NA
Concentration Recovered			10.40	NA	NA	NA	9.48	NA	NA	NA	NA
Amount Spiked			9.00	NS	NS	NS	9.00	NS	NS	NS	NS
Percent Recovery			116%	NA	NA	NA	105%	NA	NA	NA	NA
RPD			5%	NA	NA	NA	3%	NA	NA	NA	5%
I-Stat			0.02	NA	NA	NA	0.01	NA	NA	NA	0.02
<i>M. nasuta</i> Background											
<i>M. nasuta</i> Background, MS	2	2	0.18 U	NA	NA	NA	0.24 U	NA	NA	NA	NA
Concentration Recovered	2	2	3.75	NA	NA	NA	4.08	NA	NA	NA	NA
Amount Spiked			4.75	NS	NS	NS	4.75	NA	NA	NS	NA
Percent Recovery			79%	NA	NA	NA	86%	NA	NA	NA	80%
QC Sample	5	3	0.36 U	NA	NA	NA	0.47 U	NA	NA	NA	0.70 U
QC Sample, MS	5	3	6.35	NA	NA	NA	9.90	NA	NA	NA	NA
Concentration Recovered			6.35	NA	NA	NA	9.90	NA	NA	NA	NA
Amount Spiked			9.15	NS	NS	NS	9.15	NA	NA	NS	NA
Percent Recovery			69%	NA	NA	NA	108%	NA	NA	NA	93%
QC Sample	4	4	0.18 U	NA	NA	NA	0.24 U	NA	NA	NA	0.35 U
QC Sample, MS	4	4	3.66	NA	NA	NA	2.49	NA	NA	NA	NA
Concentration Recovered			3.66	NA	NA	NA	2.49	NA	NA	NA	NA
Amount Spiked			5.00	NS	NS	NS	5.00	NA	NA	NS	NA
Percent Recovery			73%	NA	NA	NA	50%	NA	NA	NA	78%

TABLE I.14. (contd)

Sediment Treatment	M. nasuta Pesticides (µg/kg wet weight)												
	Replicate	Analytical Batch	Aldrin	Alpha BHC	Beta BHC	Delta BHC	Gamma BHC	Alpha-Chlor-dane	Gamma-Chlor-dane	Tech-Chlor-dane	4,4'-DDD	4,4'-DDE	4,4'-DDT
Standard Reference Material													
Non-certified value NIST 1974			NC ^(d)	NC	NC	NC	NC	3.2 ±0.2	NC	NC	8.4 ±0.4	5.9 ±0.2	0.3 ±0.3
NIST 1974	1		NA	NA	NA	NA	NA	0.99	NA	NA	6.67	7.72 ^(e)	9.09 ^(e)
Analytical Replicates													
OBM COMP, Replicate 1	5	1	0.36 U	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0	40.1	2.04	0.71 U
OBM COMP, Replicate 2	5	1	0.36 U	1.0 U	1.0 U	1.0 U	0.48 U	1.0 U	1.0 U	13.0	41.3	1.67	0.71 U
RPD			NA	NA	NA	NA	NA	NA	NA	0%	3%	20%	NA
I-Stat			NA	NA	NA	NA	NA	NA	NA	0.00	0.01	0.10	NA
M. nasuta Background, Replicate 1	3	2	2.35	0.49 U	0.49 U	0.49 U	0.23 U	NA	NA	30 U	0.46 U	0.14 U	2.88
M. nasuta Background, Replicate 2	3	2	2.79	0.47 U	0.47 U	0.47 U	0.23 U	NA	NA	30 U	0.44 U	0.53	3.46
M. nasuta Background, Replicate 3	3	2	2.95	0.48 U	0.48 U	0.48 U	0.23 U	NA	NA	30 U	0.45 U	0.13 U	2.55
RSD			12%	NA	NA	NA	NA	NA	NA	NA	NA	NA	16%
QC Sample, Replicate 1	2	3	0.34 U	0.92 U	0.92 U	0.92 U	0.44 U	NA	NA	30 U	0.86 U	0.26 U	0.65 U
QC Sample, Replicate 2	2	3	0.33 U	0.90 U	0.90 U	0.90 U	0.43 U	NA	NA	30 U	0.85 U	0.25 U	0.64 U
QC Sample, Replicate 3	2	3	0.32 U	0.88 U	0.88 U	0.88 U	0.42 U	NA	NA	30 U	0.83 U	0.25 U	0.63 U
RSD			NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
R-OS, Replicate 1	2	4	0.18 U	0.49 U	0.49 U	0.49 U	0.24 U	NA	NA	30 U	0.46 U	0.14 U	0.35 U
R-OS, Replicate 2	2	4	0.18 U	0.49 U	0.49 U	0.49 U	0.23 U	NA	NA	30 U	0.46 U	0.14 U	0.35 U
R-OS, Replicate 3	2	4	0.18 U	0.49 U	0.49 U	0.49 U	0.24 U	NA	NA	30 U	0.46 U	0.14 U	0.35 U
RSD			NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA

(a) U Undetected at or above detection limit.

(b) NA Not applicable.

(c) NS Not spiked.

(d) NC Not certified.

(e) Outside quality control criteria (±30%) for SRMs.

TABLE I.15. Quality Control Data for Chlorinated Pesticides (alphabetical, Dieldrin - Toxaphene), Wet Weight, In Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> Pesticides (µg/kg wet weight)								
			Dieldrin	Endo-Sulfan I	Endo-Sulfan II	Endo-Sulfan-Sulfate	Endrin	Aldehyde	Hepta-chlor	Hepta-chlor epoxide	Toxa-phene
Method Blank											
Blank		1	0.64 U ^(a)	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U	16 U
Blank		2	0.33 U	0.53 U	0.53 U	0.53 U	0.53 U	0.53 U	0.16 U	0.51 U	30 U
Blank		3	0.35 U	0.55 U	0.55 U	0.55 U	0.55 U	0.55 U	0.16 U	0.53 U	30 U
Blank		4	0.19 U	0.30 U	0.30 U	0.30 U	0.30 U	0.30 U	0.09 U	0.29 U	30 U
Matrix Spikes											
OBM COMP	3	1	1.94	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U	16 U
OBM COMP, MS	3	1	41.1	NA ^(b)	NA	NA	NA	38.1	9.65	NA	NA
Concentration Recovered			39.2	NA	NA	NA	NA	38.1	9.65	NA	NA
Amount Spiked			36.0	NS ^(c)	NS	NS	NS	36.0	9.00	NS	NS
Percent Recovery			109%	NA	NA	NA	NA	106%	107%	NA	NA
OBM COMP	3	1	1.94	1.0 U	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U	16 U
OBM COMP, MSD	3	1	44.6	NA	NA	NA	NA	40.0	9.64	NA	NA
Concentration Recovered			42.7	NA	NA	NA	NA	40.0	9.64	NA	NA
Amount Spiked			36.0	NS	NS	NS	NS	36.0	9.00	NS	NS
Percent Recovery			119%	NA	NA	NA	NA	111%	107%	NA	NA
<i>M. nasuta</i> Background											
<i>M. nasuta</i> Background, MS	2	2	0.31 U	NA	NA	NA	NA	0.49 U	0.15 U	NA	NA
Concentration Recovered			16.3	NA	NA	NA	NA	15.9	3.79	NA	NA
Amount Spiked			16.3	NA	NA	NA	NA	15.9	3.79	NA	NA
Percent Recovery			86%	NA	NS	NS	NS	19.0	4.75	NS	NS
<i>M. nasuta</i> QC Sample											
QC Sample, MS	5	3	0.62 U	NA	NA	NA	NA	0.98 U	0.30 U	NA	NA
Concentration Recovered			30.6	NA	NA	NA	NA	24.4	4.53	NA	NA
Amount Spiked			30.6	NA	NA	NA	NA	24.4	4.53	NA	NA
Percent Recovery			84%	NA	NS	NS	NS	36.6	9.15	NS	NS
<i>M. nasuta</i> QC Sample											
QC Sample, MS	4	4	6.54	NA	NA	NA	NA	0.49 U	0.15 U	NA	NA
Concentration Recovered			24.2	NA	NA	NA	NA	12.7	3.07	NA	NA
Amount Spiked			17.7	NA	NA	NA	NA	12.7	3.07	NA	NA
Percent Recovery			88%	NA	NS	NS	NS	20.0	5.00	NS	NS
				NA	NA	NA	NA	64%	61%	NA	NA

TABLE I.15. (contd)

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> Pesticides (µg/kg wet weight)						
			Dieldrin	Endo-Sulfan I	Endo-Sulfan II	Endo-Sulfan-Sulfate	Endrin Aldehyde	Hepta-chlor epoxide	Toxa-phene
Standard Reference Material									
Non-certified value NIST 1974			1.0 ±0.5	NC (d)	NC	NC	NC	NC	NC
NIST 1974		1	2.87 (e)	NA	NA	NA	NA	NA	NA
Analytical Replicates									
OBM COMP, Replicate 1	5	1	3.21	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U
OBM COMP, Replicate 2	5	1	3.38	1.0 U	1.0 U	1.0 U	1.0 U	0.30 U	0.98 U
RPD			5%	NA	NA	NA	NA	NA	NA
I-Stat			0.03	NA	NA	NA	NA	NA	NA
<i>M. nasuta</i> Background, Replicate 1	3	2	0.31 U	0.49 U	0.49 U	0.49 U	0.49 U	0.15 U	0.47 U
<i>M. nasuta</i> Background, Replicate 2	3	2	0.30 U	0.47 U	0.47 U	0.47 U	0.47 U	0.14 U	0.46 U
<i>M. nasuta</i> Background, Replicate 3	3	2	0.31 U	0.48 U	0.48 U	0.48 U	0.48 U	0.14 U	0.47 U
RSD			NA	NA	NA	NA	NA	NA	NA
QC Sample, Replicate 1	2	3	0.59 U	0.92 U	0.92 U	0.92 U	0.92 U	0.28 U	0.90 U
QC Sample, Replicate 2	2	3	0.57 U	0.90 U	0.90 U	0.90 U	0.90 U	0.27 U	0.88 U
QC Sample, Replicate 3	2	3	0.56 U	0.88 U	0.88 U	0.88 U	0.88 U	0.27 U	0.86 U
RSD			NA	NA	NA	NA	NA	NA	NA
R-OS, Replicate 1	2	4	1.03	0.49 U	0.49 U	0.49 U	0.49 U	0.15 U	0.48 U
R-OS, Replicate 2	2	4	0.31 U	0.49 U	0.49 U	0.49 U	0.49 U	0.15 U	0.47 U
R-OS, Replicate 3	2	4	0.31 U	0.49 U	0.49 U	0.49 U	0.49 U	0.15 U	0.48 U
RSD			NA	NA	NA	NA	NA	NA	NA

(a) U Undetected at or above detection limit.

(b) NA Not applicable.

(c) NS Not spiked.

(d) NC Not certified.

(e) Outside quality control criteria (±30%) for SRMs.

TABLE I.16. Polychlorinated Biphenyls (PCBs), Wet Weight, in Tissue of *M. nasuta*,
Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> PCBs (µg/kg wet weight)			
			Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
Target DL ^(a)			20	20	20	20
<u>January 1993</u>						
OBM COMP	1	1	3.2 U ^(b)	3.2 U	3.2 U	3.2 U
OBM COMP	2	1	3.2 U	3.2 U	3.2 U	3.2 U
OBM COMP	3	1	3.2 U	3.2 U	3.2 U	3.2 U
OBM COMP	4	1	3.2 U	3.2 U	3.2 U	3.2 U
OBM COMP, Replicate 1	5	1	3.2 U	3.2 U	3.2 U	3.2 U
OBM COMP, Replicate 2	5	1	3.2 U	3.2 U	3.2 U	3.2 U
<u>January 1994</u>						
OBM COMP	1	3	20 U	20 U	20 U	20 U
OBM COMP	2	3	20 U	20 U	20 U	20 U
OBM COMP	3	3	20 U	20 U	20 U	20 U
OBM COMP	4	3	20 U	20 U	20 U	20 U
OBM COMP	5	3	20 U	20 U	20 U	20 U
R-OS	1	4	20 U	20 U	20 U	20 U
R-OS, Replicate 1	2	4	20 U	20 U	20 U	20 U
R-OS, Replicate 2	2	4	20 U	20 U	20 U	20 U
R-OS, Replicate 3	2	4	20 U	20 U	20 U	20 U
R-OS	3	4	20 U	20 U	20 U	20 U
R-OS	4	4	20 U	20 U	20 U	20 U
R-OS	5	4	20 U	20 U	20 U	20 U
R-BF	1	3	20 U	20 U	20 U	20 U
R-BF	2	2	20 U	20 U	20 U	20 U
R-BF	3	3	20 U	20 U	20 U	20 U
R-BF	4	3	20 U	20 U	20 U	20 U
R-BF	5	3	20 U	20 U	20 U	20 U
R-AM	1	2	20 U	20 U	20 U	20 U
R-AM	2	3	20 U	20 U	20 U	20 U
R-AM	3	3	20 U	20 U	20 U	20 U
R-AM	4	2	20 U	20 U	20 U	20 U
R-AM	5	3	20 U	20 U	20 U	20 U
C-SB	1	4	20 U	20 U	20 U	20 U
C-SB	2	4	20 U	20 U	20 U	20 U
C-SB	3	4	20 U	20 U	20 U	20 U
C-SB	4	4	20 U	20 U	20 U	20 U
C-SB	5	4	20 U	20 U	20 U	20 U

TABLE I.16. (contd)

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> PCBs (µg/kg wet weight)			
			Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
<i>M. nasuta</i> Background	1	2	20 U	20 U	20 U	20 U
<i>M. nasuta</i> Background	2	2	20 U	20 U	20 U	20 U
<i>M. nasuta</i> Background, Replicate 1	3	2	20 U	20 U	20 U	20 U
<i>M. nasuta</i> Background, Replicate 2	3	2	20 U	20 U	20 U	20 U
<i>M. nasuta</i> Background, Replicate 3	3	2	20 U	20 U	20 U	20 U
<i>M. nasuta</i> Background	4	2	20 U	20 U	20 U	20 U
<i>M. nasuta</i> Background	5	2	20 U	20 U	20 U	20 U

(a) DL Detection limit.

(b) Undetected at or above detection limit.

TABLE I.17. Polychlorinated Biphenyls (PCBs), Dry Weight, in Tissue of *M. nasuta*,
Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	<i>M. nasuta</i> PCBs (µg/kg dry weight)			
				Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
<u>January 1993</u>							
OBM COMP	1	1	13.6	23.5 U ^(a)	23.5 U	23.5 U	23.5 U
OBM COMP	2	1	13.2	24.2 U	24.2 U	24.2 U	24.2 U
OBM COMP	3	1	12.3	26.0 U	26.0 U	26.0 U	26.0 U
OBM COMP	4	1	11.4	28.1 U	28.1 U	28.1 U	28.1 U
OBM COMP, Replicate 1	5	1	12.0	26.7 U	26.7 U	26.7 U	26.7 U
OBM COMP, Replicate 2	5	1	12.0	26.7 U	26.7 U	26.7 U	26.7 U
<u>January 1994</u>							
OBM COMP	1	3	14.0	140 U	140 U	140 U	140 U
OBM COMP	2	3	13.2	150 U	150 U	150 U	150 U
OBM COMP	3	3	12.2	160 U	160 U	160 U	160 U
OBM COMP	4	3	14.3	140 U	140 U	140 U	140 U
OBM COMP	5	3	14.5	140 U	140 U	140 U	140 U
R-OS	1	4	12.7	160 U	160 U	160 U	160 U
R-OS, Replicate 1	2	4	12.6	160 U	160 U	160 U	160 U
R-OS, Replicate 2	2	4	12.6	160 U	160 U	160 U	160 U
R-OS, Replicate 3	2	4	12.6	160 U	160 U	160 U	160 U
R-OS	3	4	13.8	150 U	150 U	150 U	150 U
R-OS	4	4	12.7	160 U	160 U	160 U	160 U
R-OS	5	4	13.4	150 U	150 U	150 U	150 U
R-BF	1	3	15.3	130 U	130 U	130 U	130 U
R-BF	2	2	14.6	140 U	140 U	140 U	140 U
R-BF	3	3	14.8	140 U	140 U	140 U	140 U
R-BF	4	3	15.9	130 U	130 U	130 U	130 U
R-BF	5	3	14.9	130 U	130 U	130 U	130 U
R-AM	1	2	15.8	130 U	130 U	130 U	130 U
R-AM	2	3	13.8	150 U	150 U	150 U	150 U
R-AM	3	3	15.4	130 U	130 U	130 U	130 U
R-AM	4	2	14.5	140 U	140 U	140 U	140 U
R-AM	5	3	14.7	140 U	140 U	140 U	140 U
C-SB	1	4	14.4	140 U	140 U	140 U	140 U
C-SB	2	4	13.3	150 U	150 U	150 U	150 U
C-SB	3	4	14.0	140 U	140 U	140 U	140 U
C-SB	4	4	13.6	150 U	150 U	150 U	150 U
C-SB	5	4	13.6	150 U	150 U	150 U	150 U

TABLE I.17. (contd)

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	<i>M. nasuta</i> PCBs (µg/kg dry weight)			
				Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
<i>M. nasuta</i> Background	1	2	13.5	150 U	150 U	150 U	150 U
<i>M. nasuta</i> Background	2	2	12.0	170 U	170 U	170 U	170 U
<i>M. nasuta</i> Background, Replicate 1	3	2	13.1	150 U	150 U	150 U	150 U
<i>M. nasuta</i> Background, Replicate 2	3	2	13.1	150 U	150 U	150 U	150 U
<i>M. nasuta</i> Background, Replicate 3	3	2	13.1	150 U	150 U	150 U	150 U
<i>M. nasuta</i> Background	4	2	14.2	140 U	140 U	140 U	140 U
<i>M. nasuta</i> Background	5	2	17.7	110 U	110 U	110 U	110 U

(a) Undetected at or above detection limit.

TABLE I.18. Quality Control Data for Polychlorinated Biphenyls (PCBs), Wet Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	M. nasuta PCBs (µg/kg wet weight)			
			Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
<u>Method Blanks</u>						
Blank		1	3.2 U ^(a)	3.2 U	3.2 U	3.2 U
Blank		2	20 U	20 U	20 U	20 U
Blank		3	20 U	20 U	20 U	20 U
Blank		4	20 U	20 U	20 U	20 U
<u>Matrix Spikes</u>						
OBM COMP	3	1	3.2 U	3.2 U	3.2 U	3.2 U
OBM COMP, MS	3	1	NA ^(b)	NA	207.6	NA
Concentration Recovered			NA	NA	207.6	NA
Amount Spiked			NS ^(c)	NS	180.0	NS
Percent Recovery			NA	NA	115%	NA
OBM COMP	3	1	3.2 U	3.2 U	3.2 U	3.2 U
OBM COMP, MSD	3	1	NA	NA	222.3	NA
Concentration Recovered			NA	NA	222.3	NA
Amount Spiked			NS	NS	180.0	NS
Percent Recovery			NA	NA	124% ^(d)	NA
RPD			NA	NA	7%	NA
I-Stat			NA	NA	0.03	NA
M. nasuta Background	2	2	NA	NA	20 U	NA
M. nasuta Background, MS	2	2	NA	NA	101	NA
Concentration Recovered			NA	NA	101	NA
Amount Spiked			NS	NS	95	NS
Percent Recovery			NA	NA	106%	NA
QC Sample	5	3	NA	NA	20 U	NA
QC Sample, MS	5	3	NA	NA	131	NA
Concentration Recovered			NA	NA	131	NA
Amount Spiked			NS	NS	183	NS
Percent Recovery			NA	NA	72%	NA
QC Sample	4	4	NA	NA	20 U	NA
QC Sample, MS	4	4	NA	NA	101	NA
Concentration Recovered			NA	NA	101	NA
Amount Spiked			NS	NS	100	NS
Percent Recovery			NA	NA	101%	NA

Standard Reference Material

Certified SRM not available for PCBs.

TABLE I.18. (contd)

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> PCBs (µg/kg wet weight)			
			Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
<u>Analytical Replicates</u>						
OBM COMP, Replicate 1	5	1	3.2 U	3.2 U	3.2 U	3.2 U
OBM COMP, Replicate 2	5	1	3.2 U	3.2 U	3.2 U	3.2 U
RPD			NA	NA	NA	NA
I-Stat			NA	NA	NA	NA
<i>M. nasuta</i> Background, Replicate 1	3	2	20 U	20 U	20 U	20 U
<i>M. nasuta</i> Background, Replicate 2	3	2	20 U	20 U	20 U	20 U
<i>M. nasuta</i> Background, Replicate 3	3	2	20 U	20 U	20 U	20 U
RSD			NA	NA	NA	NA
QC Sample, Replicate 1	2	3	20 U	20 U	20 U	20 U
QC Sample, Replicate 2	2	3	20 U	20 U	20 U	20 U
QC Sample, Replicate 3	2	3	20 U	20 U	20 U	20 U
RSD			NA	NA	NA	NA
R-OS, Replicate 1	2	4	20 U	20 U	20 U	20 U
R-OS, Replicate 2	2	4	20 U	20 U	20 U	20 U
R-OS, Replicate 3	2	4	20 U	20 U	20 U	20 U
RSD			NA	NA	NA	NA

(a) U Undetected at or above detection limit.

(b) NA Not applicable.

(c) NS Not spiked.

(d) Exceeds quality control criteria (40% - 120%) for matrix spike recoveries.

TABLE I.19. Surrogate Percent Recoveries and Quality Control Data for Pesticides and Polychlorinated Biphenyls (PCBs), Wet Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	Surrogate Percent Recoveries			
			PCB 103	PCB 198	TCMX	OCN
<u>January 1993</u>						
OBM COMP	1	1	NA ^(a)	NA	111	135 ^(b)
OBM COMP	2	1	NA	NA	110	162 ^(b)
OBM COMP	3	1	NA	NA	114	129 ^(b)
OBM COMP	4	1	NA	NA	110	129 ^(b)
OBM COMP, Replicate 1	5	1	NA	NA	112	124 ^(b)
OBM COMP, Replicate 2	5	1	NA	NA	112	129 ^(b)
<u>January 1994</u>						
OBM COMP	1	3	66	51	NA	NA
OBM COMP	2	3	66	54	NA	NA
OBM COMP	3	3	61	53	NA	NA
OBM COMP	4	3	65	55	NA	NA
OBM COMP	5	3	72	61	NA	NA
R-OS	1	4	83	69	NA	NA
R-OS, Replicate 1	2	4	73	62	NA	NA
R-OS, Replicate 2	2	4	74	66	NA	NA
R-OS, Replicate 3	2	4	71	66	NA	NA
R-OS	3	4	78	67	NA	NA
R-OS	4	4	79	73	NA	NA
R-OS	5	4	77	69	NA	NA
R-BF	1	3	64	63	NA	NA
R-BF	2	2	69	70	NA	NA
R-BF	3	3	59	53	NA	NA
R-BF	4	3	75	62	NA	NA
R-BF	5	3	74	70	NA	NA
R-AM	1	2	72	75	NA	NA
R-AM	2	3	75	72	NA	NA
R-AM	3	3	72	66	NA	NA
R-AM	4	2	69	65	NA	NA
R-AM	5	3	76	68	NA	NA
C-SB	1	4	78	87	NA	NA
C-SB	2	4	82	77	NA	NA
C-SB	3	4	79	73	NA	NA
C-SB	4	4	74	62	NA	NA
C-SB	5	4	77	65	NA	NA

TABLE I.19. (contd)

Sediment Treatment	Replicate	Analytical Batch	Surrogate Percent Recoveries			
			PCB 103	PCB 198	TCMX	OCN
<i>M. nasuta</i> Background	1	2	67	95	NA	NA
<i>M. nasuta</i> Background	2	2	67	80	NA	NA
<i>M. nasuta</i> Background, Replicate 1	3	2	70	93	NA	NA
<i>M. nasuta</i> Background, Replicate 2	3	2	63	80	NA	NA
<i>M. nasuta</i> Background, Replicate 3	3	2	62	93	NA	NA
<i>M. nasuta</i> Background	4	2	67	116	NA	NA
<i>M. nasuta</i> Background	5	2	71	75	NA	NA
<u>Quality Control Data</u>						
<u>Method Blank</u>						
Blank		1	NA	NA	110	117
Blank		2	73	77	NA	NA
Blank		3	77	68	NA	NA
Blank		4	70	74	NA	NA
<u>Matrix Spike</u>						
OBM COMP, MS		1	NA	NA	110	130 ^(b)
OBM COMP, MSD		1	NA	NA	114	131 ^(b)
<i>M. nasuta</i> Background	2	2	67	80	NA	NA
<i>M. nasuta</i> Background, MS	2	2	69	84	NA	NA
QC Sample	5	3	71	60	NA	NA
QC Sample, MS	5	3	73	64	NA	NA
QC Sample	4	4	78	64	NA	NA
QC Sample, MS	4	4	81	68	NA	NA
<u>Analytical Replicates</u>						
OBM COMP, Replicate 1	5	1	NA	NA	112	124 ^(b)
OBM COMP, Replicate 2	5	1	NA	NA	112	129 ^(b)
<i>M. nasuta</i> Background, Replicate 1	3	2	70	93	NA	NA
<i>M. nasuta</i> Background, Replicate 2	3	2	63	80	NA	NA
<i>M. nasuta</i> Background, Replicate 3	3	2	62	93	NA	NA

TABLE I.19. (contd)

Sediment Treatment	Replicate	Analytical Batch	Surrogate Percent Recoveries			
			PCB 103	PCB 198	TCMX	OCN
QC Sample, Replicate 1	2	3	71	61	NA	NA
QC Sample, Replicate 2	2	3	66	57	NA	NA
QC Sample, Replicate 3	2	3	73	64	NA	NA
R-OS, Replicate 1	2	4	73	62	NA	NA
R-OS, Replicate 2	2	4	74	66	NA	NA
R-OS, Replicate 3	2	4	71	66	NA	NA

(a) NA Not applicable.

(b) Exceeds quality control criteria (40% - 120%) for percent recoveries.

TABLE I.20. Metals Analysis, Dry Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> Metals (mg/kg dry weight)									
			Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
Target DL ^(a)			0.1	2.0	0.1	1.0	4.3	0.02	1.0	1.0	1.0	35
January 1993												
OBM COMP	1	1	0.34	26.2	0.63	3.15	22.2	0.136	3.09	1.20	2.52	99.2
OBM COMP	2	1	0.49	29.5	0.60	2.66	24.0	0.115	5.38	1.53	1.72	93.9
OBM COMP, Replicate 1	3	1	0.55	28.8	0.51	1.95	21.8	0.133	3.31	1.33	1.80	154
OBM COMP, Replicate 2	3	1	0.59	30.4	0.54	1.77	23.3	0.135	4.74	1.39	2.11	152
OBM COMP	4	1	0.25	27.8	0.65	2.36	14.8	0.138	3.89	1.02	1.32	97.2
OBM COMP	5	1	0.36	30.8	0.62	2.85	18.7	0.123	3.23	1.43	1.48	104
January 1994												
R-OS	1	2	0.176	18.2	0.179	1.61	9.67	0.059	4.03	1.09	1.68	113
R-OS	2	2	0.242	23.7	0.193	2.29	10.9	0.073	5.14	1.35	1.96	91.1
R-OS	3	2	0.173	20.7	0.164	2.17	10.9	0.069	4.49	0.918	1.74	94.5
R-OS	4	2	0.458	31.9	0.208	3.47	19.8	0.099	6.68	1.32	2.15	102
R-OS	5	2	0.194	21.6	0.171	2.01	10.9	0.045	3.91	0.866	1.65	84.5
R-BF	1	2	0.365	25.2	0.228	1.91	16.6	0.084	5.19	2.41	2.26	154
R-BF, Replicate 1	2	2	0.303	24.6	0.202	2.43	14.2	0.110	4.86	2.08	2.07	115
R-BF, Replicate 2	2	2	0.279	24.8	0.194	2.49	14.1	0.092	4.75	2.00	1.90	117
R-BF, Replicate 3	2	2	0.281	24.3	0.195	2.70	14.2	0.093	4.75	1.99	1.85	116
R-BF	3	2	0.179	22.0	0.132 U ^(b)	2.86	13.9	0.109	4.45	2.03	1.17	81.1
R-BF	4	2	0.109 U	17.2	0.140	2.12	10.3	0.091	3.69	1.48	1.17	94.8
R-BF	5	2	0.181	22.7	0.179	2.52	12.3	0.092	3.93	1.99	1.35	67.1
R-AM	1	2	0.151	21.7	0.186	1.32	10.6	0.078	2.61	1.44	1.62	107
R-AM	2	2	0.246	19.8	0.226	1.51	12.4	0.106	3.36	1.64	1.64	110
R-AM	3	2	0.388	32.8	0.174	1.70	17.8	0.100	3.25	2.19	1.89	88.8
R-AM	4	2	0.179	19.4	0.252	1.45	10.6	0.092	3.82	1.82	1.64	96.1
R-AM	5	2	0.239	20.3	0.208	1.51	11.7	0.099	3.04	1.69	1.07	93.3
C-SB	1	2	0.147	22.3	0.177	1.40	11.3	0.060	3.05	1.25	1.72	92.0
C-SB	2	2	0.159	25.0	0.160	1.42	12.8	0.054	2.94	1.36	1.64	92.7
C-SB	3	2	0.143	22.5	0.180	1.55	10.3	0.043	2.87	1.21	1.64	83.6
C-SB	4	2	0.226	26.4	0.204	2.11	13.1	0.054	3.45	1.37	1.76	83.3
C-SB	5	2	0.238	24.7	0.167	1.21	14.5	0.043	2.79	1.52	1.51	95.2

TABLE I.20. (contd)

Sediment Treatment	Replicate	Analytical Batch	<i>M. nasuta</i> Metals (mg/kg dry weight)									
			Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Sa	Zn
<i>M. nasuta</i> Background	1	1	0.748	30.0	0.205	1.75	16.0	0.062	2.28	1.69	1.62	82.1
<i>M. nasuta</i> Background	2	2	0.399	35.8	0.243	1.13	12.0	0.059	2.90	0.970	1.87	112
<i>M. nasuta</i> Background	3	2	0.371	24.8	0.304	0.818	15.1	0.066	2.47	0.977	1.65	123
<i>M. nasuta</i> Background	4	2	0.680	33.3	0.269	0.968	21.8	0.067	2.83	2.42	1.93	108
<i>M. nasuta</i> Background	5	2	0.262	32.0	0.250	0.948	10.4	0.061	2.64	0.633	1.87	108

(a) DL Detection limit.

(b) U Undetected at or above detection limit.

TABLE I.21. Metals Analysis, Wet Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	M. nasuta Metals (mg/kg wet weight)									
				Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
January 1993													
OBM COMP	1	1	13.9	0.05	3.6	0.09	0.44	3.1	0.019	0.43	0.17	0.35	13.8
	2	1	13.5	0.07	4.0	0.08	0.36	3.2	0.016	0.73	0.21	0.23	12.7
	3	1	12.9	0.07	3.7	0.07	0.25	2.8	0.017	0.43	0.17	0.23	19.9
	4	1	11.9	0.08	3.9	0.07	0.23	3.0	0.017	0.61	0.18	0.27	19.6
	5	1	12.9	0.03	3.3	0.08	0.28	1.8	0.016	0.46	0.12	0.16	11.6
OBM COMP				0.05	4.0	0.08	0.37	2.4	0.016	0.42	0.18	0.19	13.4
January 1994													
R-OS	1	2	12.7	0.022	2.31	0.023	0.204	1.23	0.007	0.512	0.138	0.214	14.4
R-OS	2	2	12.6	0.031	2.99	0.024	0.289	1.37	0.009	0.648	0.170	0.247	11.5
R-OS	3	2	13.8	0.024	2.87	0.023	0.300	1.50	0.010	0.621	0.127	0.241	13.1
R-OS	4	2	12.7	0.058	4.05	0.026	0.440	2.51	0.013	0.847	0.167	0.273	12.9
R-OS	5	2	13.4	0.026	2.90	0.023	0.269	1.46	0.006	0.524	0.116	0.221	11.3
R-BF	1	2	15.3	0.056	3.85	0.035	0.292	2.53	0.013	0.793	0.368	0.345	23.5
R-BF, Replicate 1	2	2	14.6	0.044	3.60	0.030	0.355	2.07	0.016	0.711	0.304	0.303	16.8
R-BF, Replicate 2	2	2	14.6	0.041	3.63	0.028	0.364	2.06	0.013	0.695	0.292	0.278	17.1
R-BF, Replicate 3	2	2	14.6	0.041	3.56	0.029	0.395	2.07	0.014	0.695	0.291	0.271	17.0
R-BF	3	2	14.8	0.026	3.25	0.020	0.422	2.05	0.016	0.657	0.300	0.173	12.0
R-BF	4	2	15.9	0.017 U	2.74	0.022	0.337	1.63	0.014	0.587	0.235	0.186	15.1
R-BF	5	2	14.9	0.027	3.39	0.027	0.376	1.83	0.014	0.586	0.296	0.201	10.0
R-AM	1	2	15.8	0.024	3.43	0.029	0.208	1.67	0.012	0.412	0.227	0.256	16.9
R-AM	2	2	13.8	0.034	2.74	0.031	0.208	1.71	0.015	0.464	0.226	0.226	15.2
R-AM	3	2	15.4	0.060	5.07	0.027	0.262	2.74	0.015	0.501	0.338	0.292	13.7
R-AM	4	2	14.5	0.026	2.82	0.037	0.210	1.53	0.013	0.554	0.264	0.238	14.0
R-AM	5	2	14.7	0.035	2.99	0.031	0.222	1.72	0.015	0.447	0.248	0.157	13.7
C-SB	1	2	14.4	0.021	3.22	0.026	0.202	1.63	0.009	0.440	0.180	0.248	13.3
C-SB	2	2	13.3	0.021	3.31	0.021	0.188	1.69	0.007	0.389	0.180	0.217	12.3
C-SB	3	2	14.0	0.020	3.16	0.025	0.217	1.44	0.006	0.402	0.169	0.230	11.7
C-SB	4	2	13.6	0.031	3.58	0.028	0.286	1.77	0.007	0.468	0.186	0.239	11.3
C-SB	5	2	13.6	0.032	3.36	0.023	0.164	1.97	0.006	0.379	0.206	0.205	12.9

TABLE I.21. (contd)

Sediment Treatment	Replicate	Analytical Batch	Percent Dry Weight	<i>M. nasuta</i> Metals (mg/kg wet weight)									
				Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
<i>M. nasuta</i> Background	1	1	13.5	0.101	4.04	0.028	0.235	2.15	0.008	0.307	0.227	0.218	11.1
<i>M. nasuta</i> Background	2	2	12.0	0.048	4.30	0.029	0.135	1.44	0.007	0.348	0.116	0.224	13.4
<i>M. nasuta</i> Background	3	2	13.1	0.048	3.24	0.040	0.107	1.97	0.009	0.322	0.127	0.215	16.1
<i>M. nasuta</i> Background	4	2	14.2	0.097	4.73	0.038	0.138	3.09	0.010	0.402	0.343	0.274	15.3
<i>M. nasuta</i> Background	5	2	17.7	0.046	5.65	0.044	0.167	1.83	0.011	0.466	0.112	0.330	19.1

(a) U Undetected at or above detection limit.

TABLE I.22. Quality Control Data for Metals Analysis, Dry Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	M. nasuta Metals (mg/kg dry weight)									
			Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
Method Blanks												
Blank	1	1	0.01 U ^(a)	NA ^(b)	0.04 U	0.52 U	NA	0.001 U	NA	0.08 U	NA	NA
Blank	2	2	0.109 U	3.99	0.132 U	0.758 U	3.34 U	0.004 U	0.933 U	0.417 U	0.11 U	39.7 U
Blank	2	2	0.109 U	2.96	0.132 U	0.758 U	3.34 U	0.004 U	0.933 U	0.417 U	0.11 U	39.7 U
Blank	2	2	0.109 U	2.93 U	0.132 U	0.758 U	3.34 U	0.004 U	0.933 U	0.417 U	0.11 U	39.7 U
Matrix Spikes												
OBM COMP ^(c)	3	1	0.57	NA	0.53	1.86	NA	0.134	NA	1.36	NA	NA
OBM COMP, MS	3	1	1.64	NA	2.62	5.47	NA	1.017	NA	3.35	NA	NA
Concentration Recovered			1.07	NA	2.09	3.61	NA	0.883	NA	1.99	NA	NA
Amount Spiked			1.00	NS ^(d)	2.00	4.00	NS	1.00	NS	2.00	NS	NS
Percent Recovery			107%	NA	105%	90%	NA	88%	NA	100%	NA	NA
QC Sample	2	2	0.177	22.2	0.143	2.00	13.1	0.083	3.48	2.12	1.48	93.1
QC Sample, MS	2	2	0.936	48.8	1.96	6.87	37.3	0.509	8.55	4.31	5.38	286
Concentration Recovered			0.759	26.6	1.81	4.87	24.2	0.426	5.07	2.19	3.90	193
Amount Spiked			1.00	25.0	2.00	5.00	25.0	0.497	5.00	2.00	5.00	200
Percent Recovery			76%	106%	91%	97%	97%	86%	101%	109%	78%	96%
QC Sample	5	2	0.141	20.8	0.144	2.21	11.3	0.077	5.46	1.22	1.50	74.1
QC Sample, MS	5	2	0.878	48.7	2.03	7.04	36.3	0.610	10.4	3.30	5.72	270
Concentration Recovered			0.737	27.9	1.88	4.83	25.0	0.533	4.94	2.08	4.22	196
Amount Spiked			1.00	25.0	2.00	5.00	25.0	0.497	5.00	2.00	5.00	200
Percent Recovery			74% ^(e)	112%	94%	97%	100%	107%	99%	104%	84%	98%
R-AM	1	2	0.151	21.7	0.186	1.32	10.6	0.078	2.61	1.44	1.62	107
R-AM, MS	1	2	1.00	50.9	2.13	6.27	36.5	0.561	7.64	3.48	5.92	315
Concentration Recovered			0.852	29.2	1.94	4.95	25.9	0.483	5.03	2.04	4.30	208
Amount Spiked			1.00	25.0	2.00	5.00	25.0	0.500	5.00	2.00	5.00	200
Percent Recovery			85%	117%	97%	99%	104%	97%	101%	102%	86%	104%
Standard Reference Material												
Certified Value 1566a			1.68 +/-0.15	14.0 +/-1.2	4.15 +/-0.38	1.43 +/-0.46	66.3 +/-4.3	0.0642 +/-0.0067	2.25 +/-0.44	0.371 +/-0.014	2.21 +/-0.24	830 +/-57
SRM 1566a	1	1	1.56	15.0	4.67	1.46	65.4	0.062	2.22	0.37	2.55	877
SRM 1566a-1	2	2	1.53	15.3	4.13	1.22	64.3	0.056	1.70	0.367	2.35	896
SRM 1566a-2	2	2	1.00 ^(f)	14.2	4.05	1.13	67.2	0.066	1.83	0.390	2.56	935
SRM 1566a-3	2	2	1.48	14.2	4.14	1.22	68.0	0.059	2.32	0.372	2.12	943

TABLE 1.22. (contd)

Sediment Treatment	Replicate	Analytical Batch	M. nasuta Metals (mg/kg dry weight)									
			Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
Analytical Replicates												
OBM COMP, Replicate 1	3	1	0.55	28.8	0.51	1.95	21.8	0.133	3.31	1.33	1.81	154.1
OBM COMP, Replicate 2	3	1	0.59	30.4	0.54	1.77	23.3	0.135	4.74	1.39	2.11	152.0
RPD			7%	5%	6%	10%	7%	1%	36% ^(a)	4%	15%	1%
I-Stat			0.04	0.03	0.03	0.05	0.03	0.01	0.18	0.02	0.08	0.01
QC Sample, Replicate 1	2	2	0.121	30.9	0.208	1.43	9.30	0.099	3.44	2.00	2.06	117
QC Sample, Replicate 2	2	2	0.122	31.6	0.190	1.57	9.51	0.092	3.40	2.02	1.92	119
QC Sample, Replicate 3	2	2	0.112	31.4	0.196	1.59	9.52	0.098	3.43	2.11	1.97	117
RSD			5%	1%	5%	6%	1%	4%	1%	3%	4%	1%
QC Sample, Replicate 1	4	2	0.177	23.7	0.234	2.20	11.7	0.058	3.59	2.21	1.76	97.6
QC Sample, Replicate 2	4	2	0.171	22.2	0.213	1.90	11.2	0.058	3.61	1.98	1.57	91.0
QC Sample, Replicate 3	4	2	0.161	21.3	0.204	1.72	10.9	0.070	3.40	1.84	1.64	89.5
RSD			5%	5%	7%	13%	4%	11%	3%	9%	6%	5%
R-BF, Replicate 1	2	2	0.303	24.6	0.202	2.43	14.2	0.110	4.86	2.08	2.07	115
R-BF, Replicate 2	2	2	0.279	24.8	0.194	2.49	14.1	0.092	4.75	2.00	1.90	117
R-BF, Replicate 3	2	2	0.281	24.3	0.195	2.70	14.2	0.093	4.75	1.99	1.85	116
RSD			5%	1%	2%	6%	0%	10%	1%	2%	6%	1%

(a) U Undetected at or above detection limit.

(b) NA Not applicable.

(c) Value is a mean of replicate three.

(d) NS Not spiked.

(e) Outside quality control range (75-125%) for matrix spike recoveries.

(f) Outside quality control criteria ($\pm 30\%$) for SRMs.(g) Value exceeds relative precision goal of $\leq 30\%$.

TABLE I.23. Butyltin Results, Wet and Dry Weight, in Tissue *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	Triphenyltin % Internal Standard	(µg/kg wet weight)		Percent Dry Weight	(µg/kg dry weight)	
				Tri-Butyltin	Di-Butyltin		Tri-Butyltin	Di-Butyltin
Target DL ^(a)		NA	NA ^(b)	1.00	1.00	NA	NA	NA
<u>January 1993</u>								
OBM COMP	1	1	78	6.9	6.6	13.6	50.7	48.5
OBM COMP	2	1	83	8.4	7.6	13.2	63.6	57.6
OBM COMP, Replicate 1	3	1	94	7.2	6.0	12.3	58.5	48.8
OBM COMP, Replicate 2	3	1	83	7.6	4.3 J ^(c)	12.3	61.8	35.0 J
OBM COMP	4	1	77	6.6	7.0	11.4	57.9	61.4
OBM COMP	5	1	80	6.4	6.9	12.0	53.3	57.5
<u>January 1994</u>								
OBM COMP	1	3	99	1.61	1.39 U ^(d)	14.0	11.5	9.92 U
OBM COMP	2	3	94	2.03	1.72	13.2	15.4	13.1
OBM COMP	3	3	100	1.89	1.39 U	12.2	15.5	11.4 U
OBM COMP	4	3	94	1.89	1.53	14.3	13.2	10.7
OBM COMP	5	3	98	1.83	1.39 U	14.5	12.6	9.58 U
R-OS	1	3	106	1.66	2.02	12.7	13.1	15.9
R-OS	2	3	52	1.94	1.76	12.6	15.4	13.9
R-OS	3	3	61	1.85	1.39 U	13.8	13.4	10.0 U
R-OS	4	3	99	1.75	1.39 U	12.7	13.8	11.0 U
R-OS, Replicate 1	5	3	99	1.82	1.39 U	13.4	13.6	10.4 U
R-OS, Replicate 2	5	3	107	1.86	1.47	13.4	13.9	11.0
R-OS, Replicate 3	5	3	98	1.73	1.39 U	13.4	12.9	10.4 U
R-BF	1	3	98	2.95	1.39 U	15.3	19.3	9.10 U
R-BF	2	2	82	2.30	1.39 U	14.6	15.7	9.49 U
R-BF	3	3	102	3.11	1.57	14.8	21.0	10.6
R-BF	4	2	96	2.74	1.92	15.9	17.2	12.1
R-BF	5	3	100	2.38	1.69	14.9	16.0	11.3
R-AM	1	2	93	3.88	1.40	15.8	24.6	8.87
R-AM	2	2	85	4.87	2.10	13.8	35.3	15.2
R-AM	3	2	94	3.31	1.39 U	15.4	21.4	9.00 U
R-AM	4	2	91	3.86	1.39 U	14.5	26.6	9.57 U
R-AM	5	3	92	4.45	1.49	14.7	30.3	10.1
C-SB	1	4	95	1.56	1.39	14.4	10.8	9.63
C-SB	2	4	97	1.39	1.39 U	13.3	10.5	10.5 U
C-SB	3	4	90	1.47	1.39 U	14.0	10.5	9.91 U
C-SB	4	4	90	1.60	1.39 U	13.6	11.8	10.2 U
C-SB	5	4	90	1.41	1.39 U	13.6	10.4	10.2 U
<i>M. nasuta</i> Background	1	2	89	0.55	1.39 U	13.5	4.09	10.3 U
<i>M. nasuta</i> Background	2	2	95	0.53	1.39 U	12.0	4.42	11.6 U
<i>M. nasuta</i> Background	3	2	96	0.65	1.39 U	13.1	4.98	10.7 U
<i>M. nasuta</i> Background	4	2	66	0.48 U	1.39 U	14.2	3.38 U	9.78 U
<i>M. nasuta</i> Background	5	2	91	0.83	1.57	17.7	4.70	8.90

(a) DL Detection limit.

(b) NA Not applicable.

(c) J Analyte detected below method detection limit (MDL), but above instrument detection limit (IDL).

(d) U Undetected at or above detection limit.

TABLE I.24. Quality Control Data for Butyltin Results, Wet Weight, in Tissue of *M. nasuta*, Older Bay Mud Study

Sediment Treatment	Replicate	Analytical Batch	Triphenyltin % Internal Standard	(µg/kg wet weight)	
				Tri-Butyltin	Di-Butyltin
<u>Method Blanks</u>					
Blank		1	111	1.6 U ^(a)	3.9 J ^(b)
Blank		2	100	0.48 U	1.39 U
Blank		3	103	0.48 U	1.39 U
Blank		4	97	0.48 U	1.39 U
<u>Matrix Spikes</u>					
OBM COMP	4	1	77	6.6	7.0
OBM COMP, MS	4	1	94	221.0	228.2
Concentration Recovered				214.4	221.2
Amount Spiked				211.9	211.9
Percent Recovery				101%	104%
OBM COMP	4	1	77	6.6	7.0
OBM COMP, MSD	4	1	91	233.0	237.6
Concentration Recovered				226.4	230.6
Amount Spiked				219.3	219.3
Percent Recovery				103%	105%
RPD				2%	1%
I-Stat				0.01	0.00
QC Sample	2	2	95	0.77	1.39 U
QC Sample, MS	2	2	98	54.2	46.6
Concentration Recovered			NA ^(c)	54.2	46.6
Amount Spiked			NA	50.0	50.0
Percent Recovery			NA	108%	93%
OBM COMP	3	3	100	1.89	1.39 U
OBM COMP, MS	3	3	99	49.0	48.7
Concentration Recovered			NA	47.1	48.7
Amount Spiked			NA	49.6	49.6
Percent Recovery			NA	95%	98%
C-SB	4	4	90	1.60	1.39 U
C-SB, MS	4	4	93	52.5	49.8
Concentration Recovered			NA	50.9	49.8
Amount Spiked			NA	48.7	48.7
Percent Recovery			NA	105%	102%

TABLE I.24. (contd)

Sediment Treatment	Replicate	Analytical Batch	Triphenyltin % Internal Standard	(µg/kg wet weight)	
				Tri- Butyltin	Di- Butyltin
<u>Analytical Replicates</u>					
OBM COMP, Replicate 1	3	1	94	7.2	6.0
OBM COMP, Replicate 2	3	1	83	7.6	4.3 J
RPD				5%	33% ^(d)
I-Stat				0.03	0.17
QC Sample, Replicate 1	1	2	92	1.21	1.39 U
QC Sample, Replicate 2	1	2	96	1.26	1.39 U
QC Sample, Replicate 3	1	2	95	1.11	1.39 U
RSD			NA	6%	NA
R-OS, Replicate 1	5	3	99	1.82	1.39 U
R-OS, Replicate 2	5	3	107	1.86	1.47
R-OS, Replicate 3	5	3	98	1.73	1.39 U
RSD			NA	4%	NA
QC Sample, Replicate 1	5	4	99	2.97	1.39 U
QC Sample, Replicate 2	5	4	92	2.82	1.39 U
QC Sample, Replicate 3	5	4	95	2.97	1.39 U
RSD			NA	3%	NA

(a) U Undetected at or above detection limit.

(b) J Analyte detected below method detection limit (MDL), but above instrument detection limit (IDL).

(c) NA Not applicable.

(d) Value exceeds relative precision goal of $\leq 30\%$.