Bioassay Testing of Baltimore Harbor Sediments Spiked with Cr(VI)

Final Report from the Center for Contaminant Transport, Fate, and Remediation

February 12, 2008

Report Authors:

Katherine Watlington[1,2], Andrew Graham[1], Edward Bouwer[1*], William Goodfellow[2], and John Baummer[2]

[1]Department of Geography and Environmental Engineering
Johns Hopkins University
3400 N. Charles Street
Baltimore, MD 21218

[2]EA Engineering, Science and Technology, Inc.
15 Loveton Circle
Sparks, MD 21152

[*]Corresponding Author email: bouwer@jhu.edu

Table of Contents

Page

Table of Contents (Continued)

Page

I. Objective and Executive Summary

The primary objective of this study was to determine if there is a relationship between ecotoxicity and ingestion of Baltimore Harbor sediments containing chromium. This was accomplished by spiking indigenous sediment samples with Cr(VI) and subsequently conducting amphipod bioassay toxicity tests. Whole sediment samples were used in the bioassays. *Leptocheirus plumulosus*, an indigenous amphipod, was used as the test organism. *L. plumulosus* was selected due to the organism's sensitivity to contaminants as well as its tendency to reside in and ingest sediment.

Both acute (10 day) and chronic (28 day) toxicity studies were performed using *L. plumulosus*. Survival was used as an endpoint for the acute toxicity studies, while survival, growth and reproduction were used for the chronic study endpoints. Three different concentrations of hexavalent chromium were spiked into sediments, from five locations, to determine if the addition of Cr(VI) to sediment caused toxicity to the amphipods. The lowest Cr(VI) spike concentrations were within the environmentally relevant concentration range. The middle and highest Cr(VI) spike concentrations significantly exceeded levels of measured total Cr concentrations in Baltimore Harbor sediments.

Based on the sediment chemistry in the Baltimore Harbor, it was hypothesized that Cr(VI) would be rapidly reduced to Cr(III). Research conducted through an ongoing research project within the Center for Contaminant Transport, Fate and Remediation (CTFR) at Johns Hopkins University (JHU) has demonstrated rapid reduction rates of Cr(VI) in Baltimore Harbor sediments (Graham *et al.,* unpublished data). These rapid reduction rates were also found while conducting these bioassay studies on Baltimore Harbor sediments. Results of both acute and chronic toxicity tests demonstrated that spiking sediments with environmentally relevant concentrations of Cr(VI), as well as, concentrations significantly exceeding chromium concentrations found in the Harbor did not cause additional toxicity to *L. plumulosus*.[*]

[*]Note: One sediment sample, spiked with concentrations (3210 µg/g) well above levels found in the Harbor, had 0% survival for both acute and chronic toxicity in all test beakers. This was the only sediment sample where the added Cr(VI) greatly exceeded the available acid volatile sulfides (AVS); and elevated Cr(VI) concentrations in sediments and overlying water were found..

Based on the results of this study, it can be concluded that the addition of environmentally relevant levels of Cr(VI) does not cause additional toxicity in Baltimore Harbor sediments, because Cr(VI) is rapidly reduced to nontoxic Cr(III). Moreover, since Cr(VI) addition did not result in increased mortality to the bioassay test organism, the chromium currently present in Baltimore Harbor sediments is unlikely to elicit a toxicological response from the ingestion pathway.

II. Background

Introduction

In the past two decades, an awareness of sediment toxicity has emerged, raising concern regarding the potential impacts to both ecological and human health. Contaminated sediments threaten aquatic life through bioconcentration, ingestion and bioaccumulation in the food chain. Sediments serve as both a sink and a reservoir for metals and hydrophobic organic contaminants (Long *et al.,* 1998). Evaluations of sediment quality have identified widespread toxicity across a number of water bodies. National studies by the US Environmental Protection Agency (EPA) have estimated that over 10% of the sediment in waterways across the country should be classified as being contaminated (USEPA 1997).

Problems with sediment quality have become more visible in the face of improving surface water quality. At a number of locations where water quality criteria (WQC) are being met, sediment toxicity is still present. Establishing standards for sediment quality has proven more difficult than establishing water criteria due to the complex nature of sediments and exposure routes. The presence of metals and organics in sediment (normally expressed as dry weight concentration) does not necessarily translate into a toxic response.

Physical and chemical properties of the sediment affect the bioavailability of pollutants, and thus the toxicity of sediment at a given site. Total organic carbon, sediment grain size, and the concentration of acid volatile sulfides (AVS) all influence bioavailability and thus toxicity. Biological exposure routes also vary between organisms and sites. Benthic organisms can be exposed to pollutants in the sediment through dissolved phase transport across organisms' membranes (e.g., gills, skin) and/or ingestion of contaminated sediment (USEPA 2001, USEPA 2004a).

Data from past studies show that sediments collected from many locations around the Baltimore Harbor exhibit toxicity and are classified as "impaired" (Klosterhaus *et al.,* 2006, McGee *et al.,* 1999). At many sampling locations within the harbor, the WQC for measured constituents are being met; however, sediments from the sites are still toxic to estuarine species. The nature of

sediment toxicity has made it extremely difficult to establish cause-and-effect relationships with specific contaminants in the Harbor and toxicity. A recent toxicity identification evaluation (TIE) carried out by the University of Maryland failed to find specific contaminants or classes of contaminants responsible for toxicity in the Baltimore Harbor (Klosterhaus *et al.,* 2006).

Toxicity Evaluation Methods

A number of methodologies have been used to determine the relationship of contaminants with toxicity. Most recently, TIE methods have been applied to sediment samples. While TIE studies have proven useful in establishing cause and effect relationships in water samples, sediment samples have proven more difficult (Ho *et al.*, 2002). In the past, TIE studies have been performed on pore water as a means of examining sediment toxicity; however, pore water studies do not factor in exposure through ingestion which is increasingly being viewed as a significant source of contaminant exposure (Lee *et al.,* 2000). Unfortunately, experimental constraints of performing whole sediment TIE studies, namely procedures for sequestration of metals or organics, have made them largely unsuccessful.

In looking for a better means for establishing cause and effect relationships in contaminated sediment, CTFR recently reviewed the literature and prepared a literature review and annotated bibliography for the Maryland Department of the Environment (Watlington *et al*, 2007). The review focuses on sediment evaluation methods. Based on the literature, CTFR concluded that spiking studies with whole sediment bioassays provided the best method for establishing causality with respect to toxicity. By spiking sediments with a single contaminant, acute and chronic toxicity of the spiked samples can be compared with the original toxicity of the sampling site (Murdoch *et al.,* 1997).

No change in toxicity would indicate that increases in the concentration of the specific contaminant do not cause increased toxicity. In this case, it would be unlikely that current concentrations of a specific contaminant at the sampling site play a role in any observed toxicity on site (e.g., no dose-response relationship). On the other hand, increases in toxicity would strongly suggest that a specific contaminant would contribute to the toxicity of sediments at a

specific site. The nature of spiking allows this method to be used with a wide range of sediments, from those that are relatively clean, to those that exhibit a moderate level of toxicity. In addition, the use of whole sediment samples with test species that live in the sediment and ingest the sediment allows for both dissolved phase transport and ingestion to be evaluated.

Chromium and Contaminated Sediments

While chromium is not an "emerging" contaminant, health concerns and public awareness of this toxicant have risen in recent years. Due to the presence of chromium in Baltimore Harbor sediments, this contaminant has been a concern to public and private organizations concerned with the overall health of the Baltimore Harbor. In previous studies, chromium has been shown to exist at elevated levels in Harbor sediments; however, chromium bioavailability and relative toxicity has been called into question (Baker *et al.*, 1997; MDE, 2005). Both trivalent chromium (Cr(III)) and hexavalent chromium (Cr(VI)) can be found in aquatic systems; however, Cr(VI) is much more soluble and thus more mobile than Cr(III). Cr(VI) is also highly toxic, while Cr(III) is, to all intents and purposes, non-toxic at environmental concentrations.

It has been determined that chromium exists as Cr(III) in most anoxic sediments. A number of studies have shown that sediments containing acid volatile sulfides (AVS) do not typically contain Cr(VI), because any Cr(VI) entering the sediment is rapidly reduced to Cr(III). AVS, a common component of anoxic sediments, is an operationally defined variable consisting of hydrogen sulfide, polysulfides, mackinawite (FeS), greigite (Fe_3S_4), and metal sulfides (Rickard and Morse, 2005). Mackinawite is believed to be the principal solid phase AVS component in many anoxic sediments. Recent studies have shown synthetic mackinawite to be an efficient reductant of hexavalent chromium (Boursiquot *et al*., 2002; Mullet *et al.,* 2004).

Even in spiking studies with Cr(VI), reduction to Cr(III) in the presence of AVS occurs fairly quickly. Studies have also shown, however, that when AVS is not present or when the Cr(VI) concentration exceeds the AVS reducing capacity, Cr(VI) will persist (Berry *et al.*, 2004; Besser *et al*., 2004; Becker *et al*., 2006). When this situation occurs, toxicity is expected (Rifkin *et al.*, 2004). Other studies on chromium toxicity have demonstrated that Cr(III) located in sediments is non-toxic to estuarine organisms. Both amphipods and polychaetes have been used in toxicity

tests with Cr(III) spiked sediments. Neither species demonstrated an observable reaction to increased Cr(III) concentrations (Berry *et al.*, 2002; Berry *et al*., 2004; Oshida *et al.*,1981). Thus, it has been concluded that when sufficient AVS is present, nearly all of the chromium in the sediment will be found in the trivalent form, and will be nontoxic to aquatic organisms.

Unfortunately, most documented studies of chromium concentrations in the Baltimore Harbor sediments have only reported total chromium concentrations. Basing management and regulatory decisions on total chromium concentrations is not a sound scientific approach as discussed by Rifkin *et al.* (2004). The data for Baltimore Harbor show that AVS levels are significantly higher than metal concentrations with a considerable level of excess sulfides. Based on these findings, it has been concluded that the total sediment chromium concentration would almost all be present in the Cr(III) state (MDE, 2005).

Although the literature and mass balance measurements available indicate a high probability that chromium is not responsible for sediment toxicity in the Baltimore Harbor, efforts have been unable to establish clear causality between any pollutant(s) and observed toxicity. Thus, chromium is among the metals of potential concern in the Baltimore Harbor. Bioassay testing was conducted to determine if a link exists between the ingestion of chromium and observed toxicity in the Harbor. The results of the bioassay testing will also determine whether current chromium levels are contributing to the present levels of sediment impairment.

Experimental Approach

The purpose of this study was to determine what effect, if any, the addition of Cr(VI) has on the toxicity of selected Baltimore Harbor sediments via the ingestion pathway. CTFR investigated sediment toxicity following Cr(VI) addition by spiking Baltimore Harbor sediment samples with Cr(VI) and performing acute and chronic bioassays using the amphipod *L. plumulosus*. CTFR was the lead institution and employed EA Engineering, Science and Technology (EA), Inc under a subcontract arrangement. While CTFR conducted all of the spiking and analyses at the Homewood campus, the bioassay tests were performed by EA under the supervision of CTFR. EA's bioassay facility, located 30 minutes north of the Homewood Campus, provided a convenient location for testing.

Initially, this study was divided into five tasks.

Task 1. CTFR collected sediment samples, with EA's assistance and equipment, from five selected locations in the Baltimore Harbor and one reference location. Following collection, sediment samples were transported to EA's facility, by EA personnel, where a portion of the sediments were sieved to remove any indigenous organisms.

Task 2. Sieved sediment samples were then transported to CTFR for analysis. Data on sediment water content, total metals, acid volatile sulfides (AVS), total chromium concentration and chromium species concentrations were obtained and analyzed. The chromium concentrations obtained from these measurements are referred to as "baseline" concentrations throughout the remainder of the report. "Baseline" concentrations should not be interpreted as concentrations in sediments free of anthropogenic impact, but rather the native pre-spike concentrations of Cr(III) and Cr(VI).

Task 3. The sieved samples were spiked with Cr(VI). Three different spike concentrations were used for the sediment samples obtained from each test site. Spike A was designed to reflect environmentally relevant concentrations of total chromium and ranged from 383 to 677 µg/g dry weight. Spike B (ranged from 1250 to 1810 µg/g dry weight) and Spike C (ranged from 2000 to

4180 µg/g dry weight) contained concentrations well in excess of the levels of total chromium measured in Baltimore Harbor sediments. The measured concentrations and calculated spike concentration data are summarized in Table 1.

These spike concentrations were chosen to: 1) correspond with the range of total Cr concentrations measured in the Baltimore Harbor sediments (e.g., environmentally relevant) in the selected sites (Spike A); and 2) exceed measured total Cr concentrations (Spikes B and C). Spikes B and C were used, in part, to assess the level of Cr(VI) necessary to cause toxicity, in the event environmentally relevant concentrations showed no response. Spiked samples were then transported back to the EA Bioassay Lab. Prior to determining toxicity with spiked sediment samples, preliminary acute toxicity tests for each sample site were conducted to gauge the toxicity of unspiked sediments.

Task 4. Following delivery of the spiked sediment samples to EA, both acute and chronic toxicity studies were completed for each of the samples. A positive control sample spiked with a high concentration of the PAH naphthalene was also tested, along with a negative control reference sediment collected from the Wye River (a nearby site with similar sediment characteristics and negligible toxicity).

Task 5. At the conclusion of the bioassay tests, final characterization studies were conducted on each of the sediment samples. A flow chart summarizing the experimental approach is shown in Figure 1.

Table 1. Measured total Cr concentrations for spiked and unspiked Baltimore Harbor sediments used in bioassay.

Sample Location	Measured Baseline Total Cr (µg/g)	Measured Total Cr after Spike A (µg/g)	Calculated Spike A (µg/g)	Measured Total Cr after Spike B (µg/g)	Calculated Spike B (µg/g)	Measured Total Cr after Spike C (µg/g)	Calculated Spike C (µg/g)
BSM-33	823	1500	677	2470	1650	2820	2000
BSM-38	271	654	383	1580	1310	3360	3090
BSM-45	148	548	400	1820	1670	4330	4180
BSM-54	126	535	408	1380	1250	3050	2920
BSM-68	354	964	610	2160	1810	3560	3210

Task 6. Cr(VI) concentrations ranging from 1,500 ug/g to 3,000 ug/g dry weight were spiked into BSM-68 to determine the Cr(VI) spike concentration at which incomplete Cr(VI) reduction occurs. The experiment allowed for a more refined determination of the threshold concentration at which the Cr(VI) spike exceeded the reducing capacity of the sediment resulting in 100% mortality to amphipods.

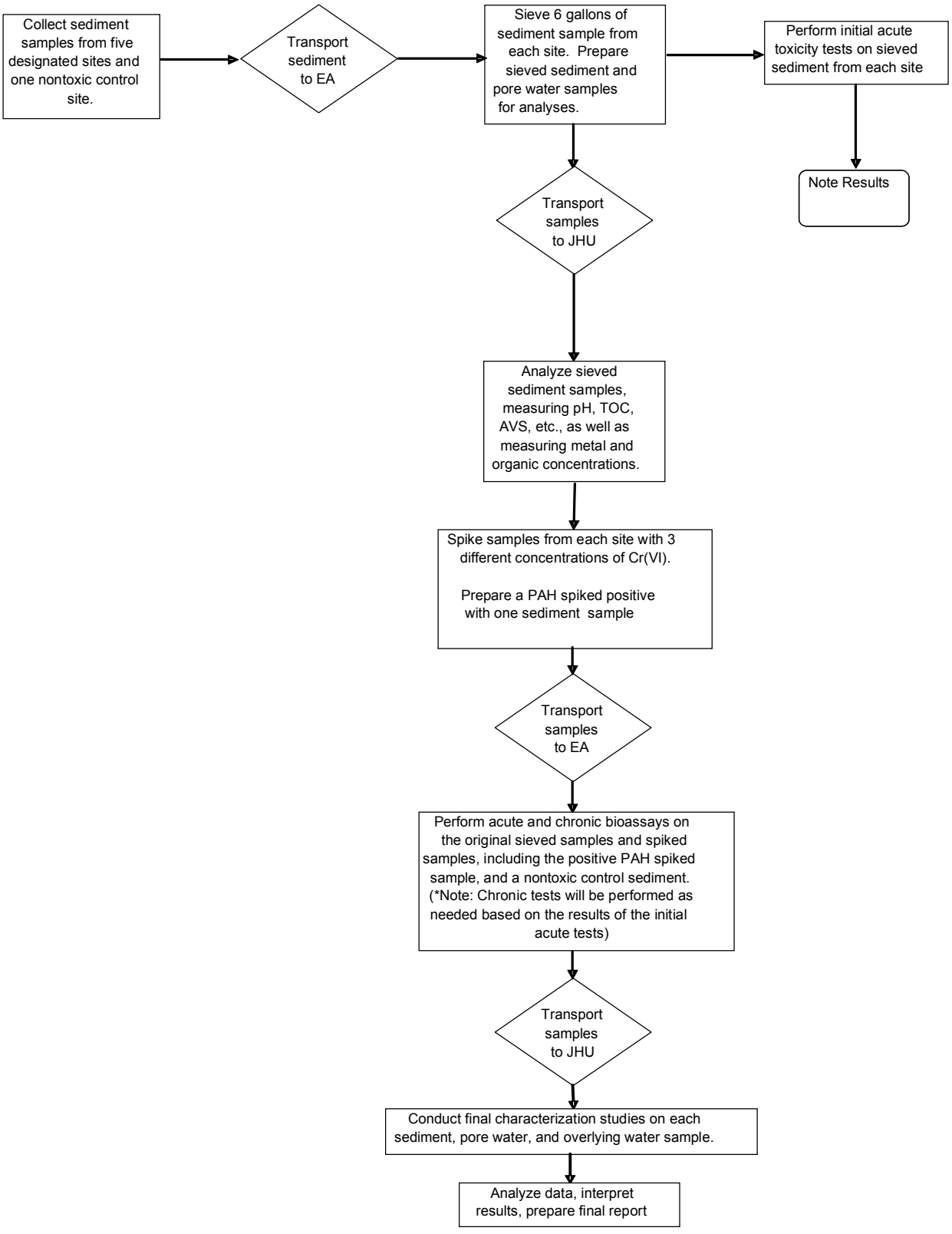

Figure 1. Flow chart of experimental approach.

Selection of Sample Sites

Sample collection for the bioassay studies took place at five selected sites around the Baltimore Harbor. The sites were chosen based on previous work detailed by McGee et al. (1999, 2004) and a recent University of Maryland TIE DRAFT Report (Klosterhaus *et al.,* 2006) that showed these sites to have elevated total chromium concentrations. Characterization data (TOC, grain size, etc) as well as contaminant analysis and toxicity data are detailed in the reports for each of the sites, thus providing important background information for the analysis to be completed by CTFR.

In selecting the sites, it was important that each site exhibit no more than 50% mortality of the test organism in an acute study (10 days) so that potential changes in toxicity with increasing Cr(VI) spike concentration would be observable. The selected sites are designated 33, 38, 45, 54, and 68. Figure 2 shows a map containing the chosen test sites along with other sites that have been characterized in the Baltimore Harbor and surrounding tributaries (McGee *et al.,* 1999). Data from the past studies for each of the test sites including pollutant concentrations and toxicity results are listed in Appendix A.

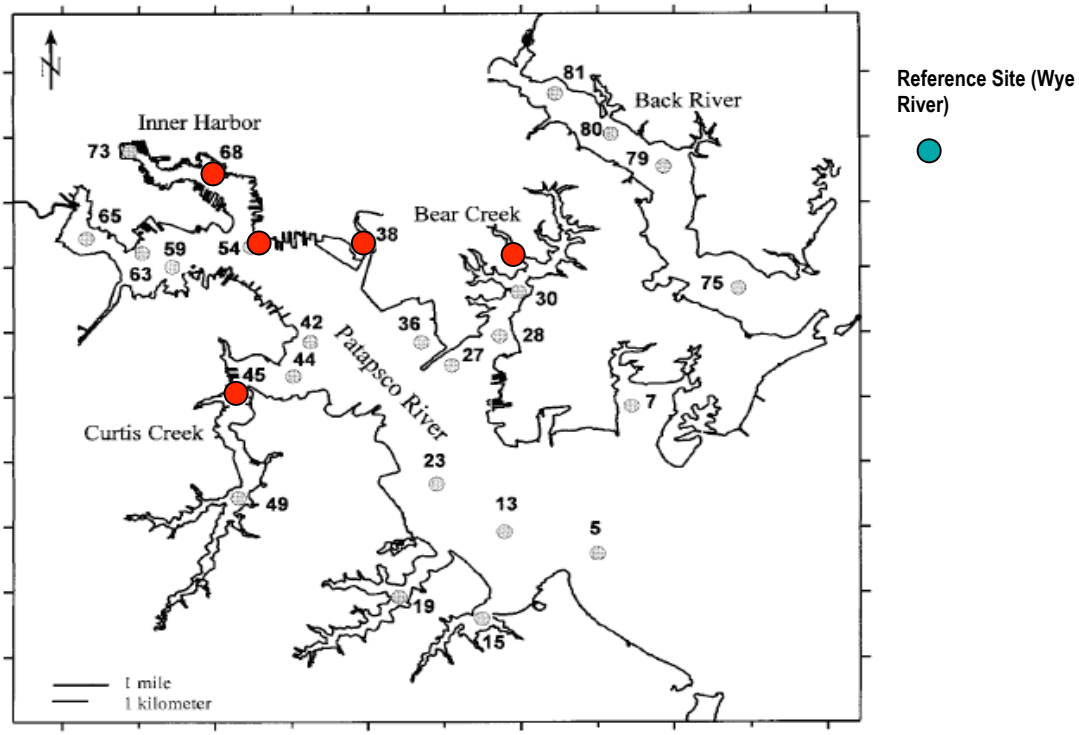

Figure 2. Baltimore Harbor test sites (McGee *et al.*, 1999). The sites shown with red dots and the Wye River reference site (off map) were sampled for this study.

Selection of Bioassay Test Organism

The estuarine amphipod *L. plumulosus* was used as the test species for the entirety of the bioassay experiments. This species was selected based on the organism's sensitivity and its presence in a wide range of sediment types and salinity ranges, as well as its tendency to reside in and ingest sediment. Because previously observed concentrations of metal contaminants in overlying water and pore water were quite low, ingestion of contaminated sediment was expected to be the primary route of exposure for aquatic organisms in the Harbor. Therefore, it was paramount that the chosen organisms ingest significant amounts of sediment as well as exhibit sensitivity to contaminants. As compared to polychaetes, clams, and other amphipods, *L. plumulosus* best matches the desired traits for the bioassay study (EPA, 2001). *L. plumulosus* is also indigenous to Baltimore Harbor sediments and can be cultured in the laboratory. This was advantageous since the cultured organisms could not have been previously exposed to environmental contaminants.

Selection of Cr(VI) Spiking Concentrations

Three different concentrations of Cr(VI) were spiked into sediment from each test site, and are noted as Spike A, Spike B, and Spike C throughout the report. Concentrations of chromium in dry sediment samples were previously found to range from approximately 200 and 850 µg/g dry weight at the five test sites (characterization data contained in Appendix A). These baseline concentrations were confirmed in the initial sediment analysis conducted as part of this experiment. These results are located in Table 1. Based on the observed chromium concentrations present, Spike A (range of 383-677 µg/g dry weight) was chosen to span the observed range of concentrations. Spike B (range of 1250-1810 µg/g dry weight) and Spike C (range of 2000 to 4180 µg/g dry weight) were chosen to assess the level of Cr(VI) necessary to cause acute and or chronic impacts to the bioassay organisms.

Selection of Test Type and Duration

Both acute (10 day) and chronic (28 day) bioassays were chosen for this study. In general, acute tests are more commonly performed in the field due to their ability to provide rapid evaluation of contaminated sediments. Chronic tests, however, have the ability to mimic conditions where sublethal concentrations of contaminants are present (McGee *et al*., 2004) and could lead to problems associated with long-term exposure. As can be seen from the site data in Appendix A, the chosen sites exhibit varying levels of toxicity, with some sites demonstrating changes in toxicity over the course of multiple tests. Thus, conducting both tests was intended to provide a more complete understanding of any effects the addition of Cr(VI) has on toxicity.

Experimental Procedures

Task 1: Sample Collection

Sediment samples were collected from the five site locations on 20-21 February 2007 by EA and CTFR personnel. A sediment sample from the non-toxic Wye River control site was also collected. Equipment for water quality analyses, including the boat, GPS system and sampler was provided by EA. The top 3 inches of sediment were collected from each station using a stainless steel Ponar sampler. Multiple grab samples were collected to make up a total of 10 gallons of sediment per site.

Five gallons from each site were used for toxicity and physicochemical characterization. The remaining five gallons of sediment were held in reserve at EA. Standard water quality parameters, temperature, pH, dissolved oxygen and salinity were measured at the bottom of each sampling station by EA personnel using a multimeter. The sediment samples were hand carried to EA's Ecotoxicology Laboratory in Sparks, Maryland on the day of collection, and placed in a temperature controlled chamber at 4°C. When not being used for testing, sediment samples remained stored in the dark at 4°C.

Task 2: Sample Preparation and Analysis

Once transferred to EA, a portion of the sediment samples were sieved and the remainder stored (in the dark, at 4°C) for any further use. A total of 5-gallons of sediment were press sieved using a 250 μm stainless steel wire mesh sieve, removing large particles and indigenous organisms. After sieving, each sample was homogenized. The five gallon sieved samples were then divided for characterization, spiking, and initial toxicity studies. Two gallons of the sieved sediment for each sampling site were hand carried to CTFR for analysis and spiking.

The remaining portion of sieved sediment from each test site was kept at EA where initial acute bioassay studies were performed to gauge the extent of mortality for the baseline mix of contaminants in the sediments. Analysis of the sediment samples included a number of parameters. Sediment water content was measured based on a protocol by Tan (1996). 10 g of wet sample was weighed and then dried in an oven at 105 °C. The final weight was then taken

and used to calculate the water content (Tan 1996). Sieve analysis was conducted by wet sieving sediment through 4.75 mm and 75 μm sieves. Particles retained on the 4.75 mm sieve were classified as gravel, those retained on the 75 μm sieve as sand, and those passing the 75 μm sieve as silt/clay. Total organic carbon content was determined by loss on ignition. Samples were placed in a muffle furnace at 550 ^{o}C for eight hours after first pretreating with 5 M HCl to remove carbonates.

The concentration of acid volatile sulfides (AVS) was also determined for each sieved sediment sample. AVS concentrations affect the reducing capacity of the sediments, as well as the bioavailability of metals; therefore, these values are important in examining causes of toxicity. AVS concentrations were determined using a method developed by Boothman and Helmstetter (1992).

Calibration standards were first prepared over a range of anticipated AVS concentrations. A sulfide stock solution was prepared from washed $Na_2S{\cdot}9H_2O$ crystals in deaerated deionized water. This sulfide solution was then standardized by iodimetric titration as described by Boothman and Helmstetter (1992). Calibration standards were prepared in a sulfide antioxidant buffer (SAOB) solution consisting of 2.0 M sodium hydroxide, 0.2 M EDTA, and 0.2 M ascorbic acid. A sulfide-specific electrode was used to measure the electrochemical potential of each standard, and a calibration curve was constructed. AVS was then recovered from the sediment samples using the purge and trap method developed by Boothman and Helmstetter (1992). Following extraction, the potential of each recovered solution was recorded and converted to concentration.

Determining chromium concentrations in the sediment samples was accomplished using two distinct analytical methods. Because chromium typically exists in aquatic environments in two valence states, different analytical approaches were used to measure both total chromium concentrations and concentrations of relevant individual chromium species, specifically Cr(VI). The use of the two analytical techniques enabled a mass balance to be obtained for chromium.

Initially, total chromium (both Cr(VI) and Cr(III)) was measured for each sieved sample using microwave assisted acid digestion coupled with inductively coupled plasma – mass spectrometry (ICP-MS). Concentrated HNO_3 was used for acid digestion of total metals following EPA Method 3051A (US EPA 1994). A polyatomic spectral interference at $m/z = 52$ caused by ArC^+ necessitated use of dynamic reaction cell (DRC) technology to remove the interference. The DRC utilized an ammonia reaction gas at a flow rate of 0.4 mL/min and a rejection parameter q of 0.4.

In addition to measurements of total chromium, the concentration of Cr(VI) in each sample was measured. Cr(VI) was extracted from sediment samples using an alkaline digestion protocol modified from EPA Method 3060A. Following alkaline digestion, samples were analyzed using reverse phase ion-pair HPLC (high performance liquid chromatography)-ICP-MS based upon a method developed by Chang and Jiang (2001). This method allowed for simultaneous measurements of Cr(VI) and Cr(III) in the digested samples. Separation of Cr(VI) and Cr(III) through HPLC-ICP-MS was achieved on a 3 mm i.d. by 3 cm C_8 column using a mobile phase flow rate of 1.5 mL/min and a mobile phase consisting of 2.0 mM tetrabutylammonium hydroxide (TBAH), an ion pairing reagent, and 0.6 mM of EDTA, a chelating agent, with the pH adjusted to 6.9 to 7.0.

Prior to analysis, alkaline digestates were diluted five-fold or greater into HPLC mobile phase and the pH of each sample was adjusted to ~7.0 with concentrated ultrapure HNO_3. Due to the alkaline digestion procedure, most of the chromium extracted was present as Cr(VI); however, the utilization of HPLC allowed for discrimination between Cr(VI) and any extracted Cr(III) at the lowest possible detection limits. The alkaline digestion procedure has been shown to minimize interconversion of Cr species during extraction (James *et al.*, 1995). The concentration of Cr(III) in the sediment samples could then be calculated as the difference between total Cr by acid digestion and Cr(VI) determined by alkaline digestion.

Because of the multielement capability of ICP-MS, acid digestion coupled with ICP-MS, used to measure total Cr concentrations, was also simultaneously used to determine additional total metal concentrations. Cadmium, cobalt, copper, manganese, nickel, lead and zinc concentrations

were all analyzed in each sediment sample. Analysis of organics in the sediment samples, including PAHs, PCBs, and chlorinated pesticides, was carried out by Martel Laboratories, Inc., a local analytical lab.

Similar parameters measured for sediment samples were also determined for sediment pore water. Pore water was extracted from sediment samples using a centrifugation method. Centrifugation at 4,000 xg and 4°C was conducted on each sediment sample for 30 minutes to extract pore water (Bufflap and Allen, 1995; Winger *et al.,* 1998). Following centrifugation, the supernatant was filtered through a 0.2 μm nylon membrane filter to complete the pore water isolation. Chromium concentrations were then measured in the pore water samples. Total Cr and other metals were determined by ICP-MS following acidification of the pore water. Cr speciation in pore water was determined by HPLC-ICP-MS following dilution of pore water samples into the HPLC mobile phase. Measurements of ammonia concentrations for each sediment sample were completed by EA using an Accumet Model 25 pH/ion meter with a gas sensing ion selective electrode (Fisher Scientific). A more complete description of the analytical methods is provided in Appendix B.

Task 3: Cr(VI) Spiking and Additional Characterization

Sieved sediment samples from each site location were spiked with three different concentrations of Cr(VI). Spiking was accomplished by adding a standard solution of potassium dichromate to each sediment sample to achieve the desired spike concentration. Sediment samples were then mixed by hand in an attempt to achieve a uniform concentration. Analysis of samples following spiking and mixing indicated that mixing was likely insufficient as total Cr concentrations were not uniform throughout the spike container.

Based on previous studies performed by Berry *et al*. (2002), 24 to 48 hours of mixing was needed to ensure equilibration between the spiking solution and the sediments. However, Berry *et al*. (2002) did not measure the mixing time needed to achieve complete reduction of Cr(VI) in sediments. Based on observed kinetics of Cr(VI) reduction in batch Baltimore Harbor sediment suspensions conducted by the CTFR, a one-day equilibration period was determined to be

sufficient. After spiking was completed, sediment samples were refrigerated overnight before being hand carried back to EA for the bioassay testing. The bioassay tests were set up on the day of sediment delivery. A small aliquot from each spiked sediment sample was returned to CTFR for analysis of total Cr and Cr speciation.

A positive control was also prepared using naphthalene as a model PAH and sieved sediment from site BSM-38. The positive control was designed to be extremely toxic to the organisms, thus eliciting a positive response. Site BSM-38 was chosen for the PAH positive control based on data from the University of Maryland TIE Report (2006). Total PAHs were determined for each sample site, and site 38 exhibited a mid-range total concentration of PAHs. These data are located in Appendix A. A stock solution of naphthalene in acetone was prepared and then added to the sediment sample to a concentration of 350 mg/kg dry weight naphthalene in sediment. This value was chosen based on previous studies with similar PAHs (Verrhiest *et al.,* 2001).

All spiking studies at CTFR were initiated within 30 days of sample collection in order to ensure the anoxic nature of the sediments was preserved. In USEPA Method 3060A, "hexavalent chromium has been shown to be quantitatively stable in field-moist soil samples for 30 days from sample collection," (USEPA 1996). Thus, all spiking was completed within this timeframe. Due to the length of the chronic bioassays, final characterization was completed more than 30 days after Cr(VI) spiking.

Task 4: Sediment Bioassays

As discussed in ***Task 1***, each of the five sediment samples were initially screened for acute toxicity (10 day) to ensure each sample exhibited a moderate level of survival for baseline comparisons. The 10-day *L. plumulosus* acute toxicity testing was conducted by EA, in accordance with US EPA (2001) guidance. The *L. plumulosus* tests were conducted in 1-L beakers each containing 175 mL of sediment and 800 mL of overlying water. Artificial seawater was used as the overlying water for the toxicity testing. The artificial seawater was prepared by mixing Crystal Sea bioassay grade sea salts with dechlorinated tap water to a final salinity of 15

ppt. The test organisms were less than 48-hours old at test initiation. The sediment and overlying water were added to the chambers 24 hours prior to introduction of the test organisms.

The beakers were left undisturbed overnight to allow any suspended sediment particles in the water column to settle. Ten organisms were randomly introduced into each replicate beaker. The introduction of the test organisms to the test chambers marked the initiation of the toxicity test. The test was initiated 23 February 2007. Organisms were not fed during the initial 10-day acute toxicity screenings. The test chambers were maintained at a target temperature of 25±1°C with a 16-hour light/8-hour dark photoperiod. Temperature, pH, dissolved oxygen, and conductivity measurements were recorded daily for the overlying water. The test solutions were not renewed during the 10-day exposure period.

At the end of the 10-day exposure period, the surviving adult organisms from each replicate were retrieved by screening through a 250 µm sieve. The number of surviving adult *L. plumulosus* from each replicate was recorded.

Following the completion of sediment spiking and delivery of the sample to EA, both acute and chronic tests were performed on each sample. The 10-day acute toxicity tests were performed concurrently with the 28-day sediment toxicity test. The tests were conducted in 1-liter beakers each containing 175 mL of sediment and 800 mL of overlying water. The tests were performed using ten replicates per sediment sample, five for the acute test and five for the chronic series. The sediment and overlying water were added to the chambers 24 hours prior to introduction of the test organisms.

The beakers were left undisturbed overnight to allow any suspended sediment particles in the water column to settle. Twenty organisms were randomly introduced into each replicate beaker. The introduction of the test organisms to the test chambers marked the initiation of the toxicity tests. The test chambers were placed in an environmental chamber and maintained at a target temperature of 25±1°C with a 16-hour light/8-hour dark photoperiod. The overlying water was gently aerated at a rate of 100 bubbles per minute throughout the 10- and 28-day exposure

period. During the 10-day exposure period, the *L. plumulosus* were fed three times a week with 1 mL/replicate of a 20 mg/mL slurry of finely ground Tetramin in deionized water.

The overlying water in the exposure chambers was renewed three times each week by siphoning 400 mL of the old overlying water from each test chamber, and then slowly siphoning fresh replacement water into the chamber, taking care not to disturb the sediment. Temperature, pH, dissolved oxygen, and conductivity in the overlying water were recorded daily for one replicate of each sediment sample.

At the end of the 10-day exposure period, the five acute toxicity beakers for each spike sample were taken down for analysis. The surviving adult organisms from each replicate were retrieved by screening through a 250 µm sieve. The number of surviving adult *L. plumulosus* from each replicate was recorded.

The 28-day chronic toxicity beakers were maintained for an additional eighteen days. As with the acute toxicity beakers, the *L. plumulosus* for the chronic study were fed three times a week with 1 mL/replicate of a 20 mg/mL slurry of finely ground Tetramin in deionized water during the first two weeks of the exposure period. This feeding schedule was maintained during weeks three and four, however the concentration of the slurry was increased to 40 mg/ml Tetramin, to provide additional food for the older (larger) test organisms.

As was done throughout the acute toxicity test, the overlying water in the exposure chambers was renewed three times per week. Water quality parameters, such as temperature, pH, dissolved oxygen, and conductivity measurements were recorded daily for the overlying water in one replicate of each sediment for the duration of the 28 day test. Ammonia measurements were conducted on composite samples of overlying water from each sediment sample at test initiation.

At the end of the 28-day exposure period, the surviving adult organisms from each replicate were retrieved by screening through a 500 µm sieve. The number of surviving adult *L. plumulosus* from each replicate was recorded, and the surviving adults from each replicate were placed in a dried, pre-weighed tin and placed in a drying oven overnight at 100°C. The tins were then

removed from the oven and placed in a desiccator to cool. Each pan was weighed to the nearest 0.01 mg to determine a mean dry weight per replicate, obtained by dividing the total organism dry weight per replicate by the number of surviving organisms per replicate. Material that had passed through the 500 µm sieve was then retained on a 250 µm sieve to retrieve the offspring. Amphipods and residual sediments that were retained on the 250 µm sieve were rinsed with freshwater to remove salts, and washed into a sample jar. The offspring were stained with a 1 g/L solution of rose bengal, and preserved with 70% alcohol. The offspring were counted, and the reproduction endpoint was calculated as the number of offspring per surviving adult.

Task 5: Final Characterization

At the completion of the chronic studies by EA, further sediment characterization was performed by CTFR. Portions of sediment from each test sample were returned to CTFR for final characterization. Total Cr concentrations and Cr(VI) sediment concentrations were measured for each sample using the protocols outlined in ***Task 2.*** Overlying water from the beakers was also tested for total Cr and Cr(VI). In addition, pore water was isolated from each sediment sample using centrifugation as described in ***Task 2.*** Total Cr concentrations and species concentrations (when relevant) were measured in the pore water for each sample.

Task 6: Cr(VI) Re-spiking and Characterization

After completing the final characterization, it was determined that Cr speciation data were needed for spiked sediment 24 hours after Cr(VI) addition. The additional measurements were used to determine if the added chromium had been completely reduced to Cr(III) at the time the bioassays were set up (24 hours after spiking). A range of chromium concentrations, from 1,500-3,000 µg/g dry weight were spiked into BSM-68 to determine the threshold concentration where Cr(VI) exceeds the reducing capacity of the sediment.

To better mimic bioassay testing conditions, 1-L beakers were set up, each containing 150 grams (approximately 175 mL) of BSM-68 sediment. The sediments were then spiked with varying concentrations of Cr(VI). The concentrations used were 1500 µg/g, 1750 µg/g, 2000 µg/g, 2250

µg/g, 2500 µg/g, 2750 µg/g, and 3000 µg/g. Following spiking, sediments were hand mixed within the beakers. The beakers were then covered and refrigerated overnight. After a 24 hour period, 800 mL of overlying water was added to the beakers. The beakers were then allowed to sit for an additional 24 hours, for a total equilibration period of 48 hours. The overlying water in each beaker was then observed to determine beakers with yellow tinted water, indicating Cr(VI) in the water and incomplete reduction. Each sediment sample was then analyzed for total Cr and Cr(VI).

Results

Water Quality Parameters at Sediment Collection Sites

Standard water quality parameters including temperature, pH, dissolved oxygen and conductivity were measured at the surface, mid-point (half of total depth at each site), and bottom of each sampling station by EA personnel, with the exception of BSM 45, BSM-2 and Wye River. Data were not collected at these locations. These values are displayed in Table 2, below. Temperature and pH were fairly consistent between the sites, measured at approximately 2 ºC and pH 8, respectively. Conductivity, dissolved oxygen (DO), and turbidity showed more variability from location to location. Dissolved oxygen was greatest in the near surface waters and ranged from 1.52 to 4.41 mg/L for sampled stations. While DO was lower at depth for all sampled stations, only site BSM-33 showed a significant difference (~1.9 mg/L) between surface and bottom water DO concentrations. Conductivity was greatest in the bottom waters near the sediment-water interface and ranged from 281.0 to 323.5 µS/m. Turbidity was also greatest for bottom waters, ranging from 3.2 to 5.2 NTU for bottom waters.

Table 2. Water quality parameters measured at sampling locations.

BSM 33		Parameters				
		Temperature (ºC)	Conductivity (µS/m)	DO (mg/L)	pH	Turbidity (NTU)
21-Feb-2007	Surface	2.20	230.4	4.41	7.91	1.8
	Middle	2.55	253.3	3.53	8.13	2.0
	Bottom	2.04	281.0	2.54	8.01	5.0
	Latitude	39.25354488 N				
	Longitude	76.49044867 W				
	Depth (ft)	10				

BSM 38		Parameters				
		Temperature (ºC)	Conductivity (µS/m)	DO (mg/L)	pH	Turbidity (NTU)
21-Feb-2007	Surface	2.31	310.7	1.76	8.16	3.5
	Middle	1.81	315.4	1.66	8.18	3.1
	Bottom	1.71	317.0	1.55	8.05	3.4
	Latitude	39.2564 N				
	Longitude	76.5361 W				
	Depth (ft)	20				

BSM 54		Parameters				
		Temperature (°C)	Conductivity (µS/m)	DO (mg/L)	pH	Turbidity (NTU)
20-Feb-2007	Surface	1.96	318.8	1.52	8.00	4.1
	Middle	1.88	319.9	1.45	7.99	2.7
	Bottom	1.99	323.5	1.34	7.91	5.2
	Latitude	39.2583 N				
	Longitude	76.5683 W				
	Depth (ft)	29				

BSM 68		Parameters				
		Temperature (°C)	Conductivity (µS/m)	DO (mg/L)	pH	Turbidity (NTU)
20-Feb-2007	Surface	2.37	298.6	2.71	7.83	2.1
	Middle	2.01	318.9	2.22	7.84	2.1
	Bottom	2.16	319.2	2.16	7.82	3.2
	Latitude	39.2778 N				
	Longitude	76.5833 W				
	Depth (ft)	12				

Initial Sediment and Water Parameters

Results from measurements of sediment water content, grain-size, and TOC for the five test sites are displayed in Table 3. Water content was fairly similar among the five test sites and averaged about 49 weight percent. Grain-size analysis revealed that the sediments should be classified as silty muds with roughly 82-97% of the total sediment weight in the silt/clay size fraction, with the exception of site BSM-54 which was appreciably sandier. TOC content averaged about 6-7% and showed little site to site variation, with the exception of site BSM-54 (TOC = 4.9%)

Table 3. Percent water content, grain-size distribution, and total organic carbon content for the five ecotoxicity test sites.

	Water Content (%)	% Gravel (>4.75 mm)	% Sand (0.075-4.75 mm)	% Silt/Clay (<0.075 mm)	% TOC
BSM-33	57%	0%	2.7%	97.3%	7.3%
BSM-38	43%	0%	12.2%	87.8%	6.1%
BSM-45	47%	0%	17.6%	82.4%	7.0%
BSM-54	51%	0%	39.8%	60.2%	4.9%
BSM-68	46%	0%	5.6%	94.4%	7.1%

The concentration of acid volatile sulfides (AVS) was also determined for each sieved sediment sample. The results of this analysis are displayed in Figure 3. As can be observed from the graph, the AVS concentrations are similar across the sediment samples with the exception of BSM-33. Measuring over 500 µmole/g dry weight, the AVS concentration in sediment from BSM-33 is a factor of 10 greater than the concentrations in BSM-68 and BSM-54 sediments.

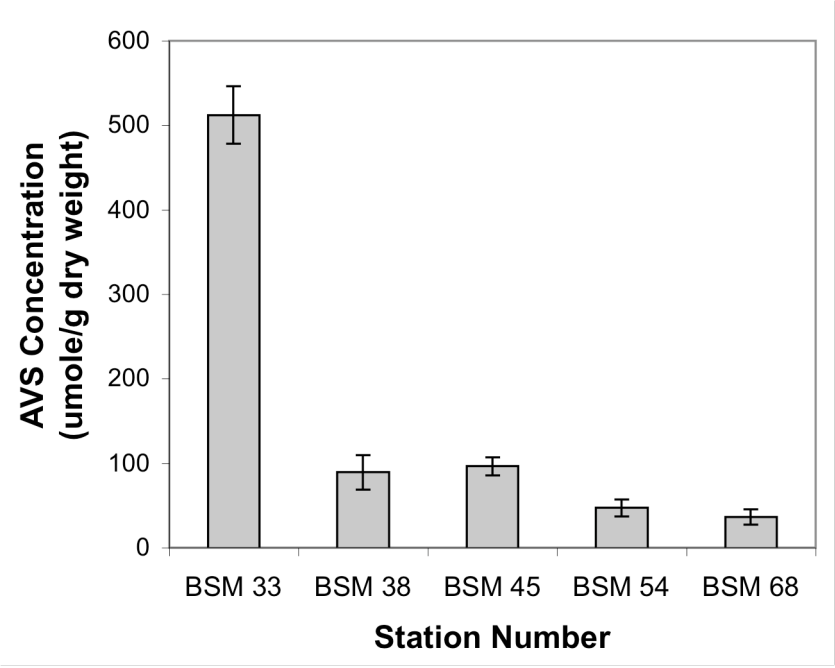

Figure 3. AVS content of sediments used in the bioassay. Error bars indicate 95% confidence limits.

During pre-spike sediment characterization, total Cr was measured for each sample, as well as the concentration of Cr(VI). The concentration of Cr(III) in the sediment samples was then calculated by subtracting the Cr(VI) concentration from the total Cr concentration. The total Cr concentration, Cr(VI) concentration and resulting balance of Cr(III) for each sediment is displayed in Table 4. As can be seen from Table 4, total chromium concentrations for the five test sites in Baltimore Harbor are elevated with concentrations ranging from 126 to 822 µg/g dry weight. Cr(VI) concentrations in sediment, however, are quite low, ~4 orders of magnitude lower than total Cr, meaning that the bulk of Cr in these sediments is present in the trivalent form.

Table 4. Total Cr, Cr(VI) and Cr(III) balance for each unspiked sediment sample. All concentrations are dry weight basis.

	Total Cr (ug/g)	Cr(VI) (ug/g)	Cr(III) (Balance) (ug/g)
BSM 33	823	0.066	823
BSM 38	271	0.055	271
BSM 45	148	0.055	148
BSM 54	126	0.050	126
BSM 68	354	0.078	354

Further analysis of sediment samples was performed to measure other contaminant concentrations, including cadmium, cobalt, copper, manganese, nickel, lead and zinc. Table 5 displays the total metal results. Concentrations of Cu, Zn, and Pb were elevated for most of the test sites with concentrations generally in the several hundred (Cu and Pb) to thousands (Zn) of µg/g dry weight. Concentrations of other heavy metal contaminants (Co, Ni, Ag, and Cd) were considerably lower with concentrations generally below 50 µg/g dry weight. A hot spot of Cd contamination was evident for site BSM-38 in Colgate Creek that contained Cd levels nearly a full order of magnitude above the other sampled stations. The data for total metals collected in this study are consistent with previous measures of total metals for these sites shown in Appendix A.

Table 5. Total metal concentrations in Baltimore Harbor sediments. All concentrations are dry weight basis.

	[Mn] (ug/g)	[Co] (ug/g)	[Ni] (ug/g)	[Cu] (ug/g)	[Zn] (ug/g)	[Ag] (ug/g)	[Cd] (ug/g)	[Pb] (ug/g)
BSM-33	144	11.4	16.5	97.4	2100	1.5	8.3	174
BSM-38	373	12.7	35.5	379	1380	1.5	77.7	426
BSM-45	411	8.7	27.7	193	281	0.7	1.0	145
BSM-54	309	10.9	26.6	223	1254	1.2	29.0	248
BSM-68	376	13.3	30.2	250	311	1.7	1.5	178

Sediment concentrations of organics, including PAHs, PCBs, and chlorinated pesticides, were determined by the contracted analytical laboratory, Martel Laboratories, Inc. A large suite of target analytes were analyzed and the full results for each sediment sample are included in Appendix C. Organic analytes were generally below detection with few exceptions. Common contaminants such as benzo[a]pyrene, 1,2 dichlorobenzene, 2,4, dichlorophenol, hexachlorobenzene, naphthalene, nitrobenzene, phenanthrene, and phenol were not observed at concentrations above the detection limit of 500 µg/g dry weight for any of the sediments sampled in this study. Common PCB Aroclor mixtures were also generally below detection (0.05 µg/g dry weight) with the exception of Aroclor PCB-1260, which was found at concentrations of 0.12 and 0.10 µg/g dry weight at sites BSM-38 and BSM-45, respectively. Chlorinated pesticides were also generally observed at concentrations below detection (generally 5 µg/kg dry weight) with the exception of dieldrin which was observed at 5.3 µg /kg dry weight at site BSM-38 and 9 µg/kg dry weight at site BSM-45.

In addition to sediment, analysis of the pore water was also conducted. Total Cr results for pore water are displayed in Table 6 below. Because total Cr concentrations were well below ambient water quality criteria for Cr(VI) (11 µg/L for freshwater and 50 µg/L for saltwater), Cr speciation in unspiked samples was not determined. Previous work by the CTFR has shown pore water Cr(VI) concentrations throughout Baltimore Harbor to be less than 1 µg/L.

Table 6. Total chromium concentrations in pore water isolated from unspiked Baltimore Harbor sediments.

	Total Cr (ug/L)
BSM33	3.0
BSM38	3.7
BSM45	1.2
BSM54	1.0
BSM68	4.8

Initial Acute Toxicity Tests

Table 7 presents the results of the initial 10-day acute toxicity sediment tests. At completion of the test, survival in the BSM38, BMS68 and the reference sediment (52, 61 and 64 percent survival respectively) was significantly different (p=0.05) from survival in the Wye River control, which had 82 percent survival. Survival in the BSM33, BSM45 and BSM54 samples was 65, 81 and 78 percent survival, respectively, and was not significantly different from control survival. Although some sediment samples exhibited greater toxicity than the Wye River control, the conclusion of the initial acute toxicity screens was that no sediment sample was sufficiently toxic to exclude it from the chronic toxicity sediment spiking study. Temperature, pH, dissolved oxygen, and conductivity measurements were recorded daily on the overlying water in one replicate of each sediment sample, and ammonia was measured in sediment pore water at test initiation. Temperature, pH, DO and salinity were determined to be fairly constant between the samples. Ammonia concentrations, however, exhibited some variability, ranging from 0.10 to 14.2 mg/L. Water quality measurements for the initial 10-day bioassay are summarized in Table 8.

Table 7. 10-day % survival for initial acute toxicity tests.

	10-Day Survival (%)
BSM33	65
BSM38	52(a)
BSM45	81
BSM54	78
BSM68	61(a)
BSM2 Reference	64(a)
Wye River Control	82

(a) Significantly different from the Wye River control (p=0.05).

Table 8. Water quality measurements for initial acute toxicity tests.

	Temperature (°C)(b)	pH	Dissolved Oxygen (mg/L)	Salinity (ppt)	Ammonia (mg/L)(c)
BSM33	23.8-25.9	7.8-8.2	4.9-6.9	14.2-15.7	14.2
	25.0 (±0.6)	7.9 (±0.1)	6.2 (±0.6)	14.9 (±0.6)	
BSM38	24.0-25.7	7.8-8.1	5.3-6.7	14.5-16.6	6.85
	24.9 (±0.5)	7.9 (±0.1)	6.2 (±0.4)	15.2 (±0.6)	
BSM45	24.3-25.9	7.7-8.5	5.7-6.8	14.3-16.3	11
	25.2 (±0.5)	8.2 (±0.3)	6.2 (±0.4)	14.9 (±0.6)	
BSM54	23.9-25.5	7.9-8.6	5.1-6.9	14.9-16.8	7.77
	24.7 (±0.5)	8.2 (±0.2)	6.2 (±0.5)	15.6 (±0.5)	
BSM68	24.2-25.9	7.9-8.0	5.4-7.0	14.5-15.5	9.94
	25.1 (±0.6)	8.0 (±0.1)	6.2 (±0.5)	14.8 (±0.3)	
BSM2 REF	24.1-25.9	7.8-8.0	5.9-7.2	14.5-17.6	1.52
	25.1 (±0.6)	7.9 (±0.1)	6.7 (±0.4)	15.4 (±0.1)	
Wye River Control	23.7-25.3	7.7-8.3	5.4-6.9	15.1-17.1	<0.10 - 6.61
	24.7 (±0.5)	8.0 (±0.2)	6.3 (±0.5)	15.9 (±0.6)	

(a) Range and mean (± standard deviation).
(b) Temperatures of test solutions were within the acceptable 3°C deviation as defined by USEPA (2002).
(c) Ammonia measured on overlying water at test initiation and termination.

Characterization of Spiked Sediments

Following spiking of sediment samples, samples were taken to EA for use in the bioassays. Unused sediment was returned to CTFR for analysis for total chromium. These results are presented in Table 1.

Acute Toxicity Tests

Results of the acute sediment toxicity tests for the various sediment samples are summarized in Table 9. For the BSM 33 sediment samples, there was 93 percent survival for spike C (2000 µg/g Cr dry weight), 90 percent survival for spike B (1650 µg/g Cr dry weight) and 92 percent survival for spike A (677 µg/g Cr dry weight) after 10 days. The unspiked baseline sediment had 90 percent survival. Sediment samples from BSM 38 displayed similar results.

After 10 days of exposure, there was 96 percent survival for spike C (3090 µg/g Cr dry weight), 90 percent survival for spike B (1310 µg/g Cr dry weight), 96 percent survival for spike A (383 µg/g Cr dry weight), and 91 percent survival in the unspiked baseline sediment. For BSM 45, there was 100 percent survival for spike C (4180 µg/g Cr dry weight), 94 percent survival for spike B (1670 µg/g Cr dry weight) and 98 percent survival for spike A (400 µg/g Cr dry weight). The unspiked baseline sediment had 97 percent survival.

After 10 days of *L. plumulosus* exposure to the BSM 54 sediment samples, there was 88 percent survival in spike C (2920 µg/g Cr dry weight), 89 percent survival in spike B (1250 µg/g Cr dry weight) and 93 percent survival in spike A (408 µg/g Cr dry weight). The unspiked BSM-54 baseline sample had 92 percent survival. Unlike, the other four sediment samples BSM-68 displayed acute toxicity at the highest spike concentration (spike C). After 10 days of exposure, there were no surviving organisms in the 3210 µg/g Cr(VI) spiked sediment, which was significantly different from survival in the unspiked baseline sample (73 percent survival). Survival in spikes B (1810 µg/g Cr dry weight) and A (610 µg/g Cr dry weight) was 75 and 81 percent survival, respectively, which was not significantly different from the baseline survival.

Table 9. Percent survival for each sediment sample at the completion of the 10 day acute toxicity test. For specific values of the spike concentrations of spikes A to C, refer to the text or Table 1. Error estimates indicate 95% confidence limits.

	BSM-33 (%)	BSM-38 (%)	BSM-45 (%)	BSM-54 (%)	BSM-68 (%)	Wye River (%)	PAH Control (%)
Baseline	90.0 (±14.6)	91.0 (±5.2)	97.0 (±12.1)	92.0 (±7.1)	73.0 (±9.4)	99.0(±2.8)	80.0(±28.1)
Spike A	92.0 (±5.6)	96.0 (±2.8)	98.0 (±3.4)	93.0 (±7.1)	81.0 (±9.2)		
Spike B	90.0 (±11.6)	90.0 (±9.8)	94.0 (±8.1)	89.0 (±5.2)	75.0 (±13.2)		
Spike C	93.0 (±8.3)	96.0 (±11.9)	100.0 (±8.8)	88.0 (±11.3)	0 (±0)		

Chronic Toxicity Tests

Results of the chronic sediment toxicity tests for the various sediment samples are summarized in Table 10. For the BSM-33 sediment samples, there was a substantial drop in survival from 10 days to 28 days; however, survival was similar among the Cr spiked sediments and not significantly different from the unspiked baseline sediment. For BSM-33 spikes A and C (677 and 2000 µg/g Cr dry weight, respectively), 33 percent survival was observed. The spike B sample had 47 percent survival at test termination and the baseline unspiked sediment had 32 percent survival. At the 95% confidence level, these values were not significantly different. Mean biomass in the spiked sediment samples ranged from 0.37 to 0.53 mg/exposed organism, which was not statistically significant from the unspiked baseline sediment (0.29 mg/organism). Mean reproduction also appeared to be unaffected by additional chromium. Mean reproduction for spikes A, B, and C was 3.6, 5.2 and 2.2 neonates per surviving organism, respectively. Mean reproduction in the unspiked baseline sample was 2.4 neonates per surviving organism.

For sediment from site BSM-38, at 28-days, there was 37 percent survival in the baseline sediment. Survival in the spiked sediments ranged from 50 to 60 percent survival, and was not significantly different from the baseline sample at the 95% confidence level. Mean biomass in the spiked sediment samples ranged from 0.40 to 0.71 mg/exposed organism which was not significantly different from mean biomass in the unspiked baseline sample (0.46 mg/exposed

organism). Mean reproduction for spikes A, B, and C was 1.6, 0.7 and 1.8 neonates per surviving organism, respectively. The unspiked BSM-38 sediment had a mean reproduction of 1.3 neonates per surviving organism.

In sediment samples from site BSM-45, there was 72 percent survival for spike A (400 µg/g Cr dry weight), 87 percent survival for spike B (1670 µg/g Cr dry weight) and 91 percent survival for spike C (4180 µg/g Cr dry weight). The baseline sediment sample had 85 percent survival. Mean biomass in the spiked sediment samples ranged from 1.07 to 1.1 mg/exposed organism and was not significantly different from the unspiked baseline sample (1.04 mg/exposed organism). Mean reproduction in spikes A, B, and C was 1.9, 3.2 and 2.5 neonates per organism, respectively. As with the biomass measurements, mean reproduction in spiked and unspiked sediments were not significantly different. The unspiked mean reproduction was measured at 0.8 neonates per surviving organism.

At 28-days, survival in BSM-54 sediment ranged from 73-82% for spiked sediments and 76% for the unspiked sediment. Compared to the unspiked baseline sediment, survival at 10-days and 28-days was not significantly altered by spiking with Cr(VI). Mean biomass in the spiked sediment samples ranged from 1.04 to 1.65 mg/exposed organism which was not significantly different from mean biomass in the unspiked baseline control (0.99 mg/exposed organism). Mean reproduction also was unaffected by Cr(VI) addition. Mean reproduction for spikes A, B, and C was 2.9, 2.3 and 4.3 neonates per surviving organism, respectively, which was not significantly different from mean reproduction in the unspiked baseline sample (2.5 neonates per surviving organism).

As with the acute toxicity test, BSM-68 displayed chronic toxicity at the highest spike concentration (3210 µg/g Cr dry weight). At the end of the 28-day testing period, there was 0% survival at this spike level. Survival for the remaining spiked sediments (67% for spike A and 57% for spike B) was not significantly different from survival in the unspiked sediment (56 percent survival). Mean biomass for spikes A and B was 0.54 and 0.50 mg/exposed organism, which was not significantly different from the unspiked baseline sample (0.39 mg/organism) at the 95% confidence level. Mean reproduction for spikes A and B was 0.2 neonates per surviving

organism. The unspiked baseline had a mean reproduction of 0.60 neonates per surviving organism.

Table 10. Percent survival, mean biomass, and mean reproduction for each sediment sample at the completion of the 28 day chronic toxicity test. Error estimates indicate 95% confidence limits.

	28-Day Survival (%)	Mean Biomass (mg/organism exposed)	Mean Reproduction (neonates per surviving organism)
BSM-33			
Baseline	32.0 (±14.3)	0.29 (±0.08)	2.4 (±2.1)
Spike A (677 ug/g)	33.0 (±12.1)	0.37 (±0.21)	3.6 (±2.8)
Spike B (1650 ug/g)	47.0 (±24.3)	0.53 (±0.28)	5.2 (±2.0)
Spike C (2000 ug/g)	34.0 (±16.1)	0.40 (±0.19)	2.2 (±1.2)
BSM-38			
Baseline	37.0 (±19.4)	0.47 (±0.40)	1.3 (±0.8)
Spike A (383 ug/g)	50.0 (±9.8)	0.68 (±0.33)	1.6 (±0.5)
Spike B (1310 ug/g)	53.0 (±16.2)	0.40 (±0.30)	0.7 (±1.0)
Spike C (3090 ug/g)	60.0 (±15.2)	0.72 (±0.35)	1.8 (±1.7)
BSM-45			
Baseline	86.0 (±6.8)	1.04 (±0.27)	0.8 (±0.7)
Spike A (400 ug/g)	72.0 (±26.2)	1.07 (±0.0.63)	1.9 (±1.1)
Spike B (1670 ug/g)	88.0 (±10.4)	1.51 (±0.14)	3.2 (±2.4)
Spike C (4180 ug/g)	93.0 (±8.3)	1.44 (±0.48)	2.5 (±1.6)
BSM-54			
Baseline	76.0 (±16.1)	0.99 (±0.41)	2.5 (±2.0)
Spike A (408 ug/g)	78.0 (±9.4)	1.38 (±0.43)	2.9 (±1.8)
Spike B (1250 ug/g)	73.0 (±16.8)	1.04 (±0.63)	2.3 (±1.0)
Spike C (2920 ug/g)	82.0 (±12.1)	1.65 (±0.45)	4.3 (±1.1)
BSM-68			
Baseline	56.0 (±27.9)	0.39 (±0.26)	0.6 (±0.6)
Spike A (610 ug/g)	67.0 (±12.1)	0.54 (±0.27)	0.2 (±0.4)
Spike B (1810 ug/g)	57.0 (±10.4)	0.50 (±0.28)	0.2 (±0.3)
Spike C (3210 ug/g)	--	--	--

Final Characterization

Following completion of the chronic bioassay studies, a representative sediment sample of each site and spike concentration was transported to CTFR for final characterization. Total Cr and Cr(VI) concentrations in sediment were measured and Cr(III) calculated by difference. These data are displayed in Table 11. Cr(VI) concentrations in sediment in spiked sediments were virtually the same as unspiked sediment with the exception of BSM 68 (spike C) which contained Cr(VI) in sediment approximately 150 times above baseline concentrations. Despite the increased Cr(VI) concentration for this sample, most of the Cr(VI) added was still reduced for this sediment, with greater than 99 percent of the total mass of Cr in the sediment in the form of Cr(III). From these speciation results, it is apparent that for all samples, nearly all of the Cr(VI) added was reduced to the trivalent form.

Table 11. Total Cr, Cr(VI) and Cr(III) balance for each sediment sample following Cr(VI) addition to the sediment. All concentrations are dry weight basis.

Site	Total Cr (µg/g)	Cr(VI) (µg/g)	Cr(III) Balance (µg/g)
BSM-33			
Baseline	823	0.05	823
Spike A	1325	0.00	1325
Spike B	2466	0.00	2466
Spike C	2820	0.02	2820
BSM-38			
Baseline	271	0.05	271
Spike A	654	0.01	654
Spike B	1580	0.01	1580
Spike C	3360	0.03	3360
BSM-45			
Baseline	148	0.05	148
Spike A	548	0.01	548
Spike B	1820	0.00	1820
Spike C	4330	0.02	4330
BSM-54			
Baseline	126	0.08	126
Spike A	535	1.37	534
Spike B	1380	0.27	1380
Spike C	3050	0.80	3049
BSM-68			
Baseline	354	0.06	354
Spike A	964	0.36	964
Spike B	2160	1.10	2159
Spike C	3560	9.57	3550

Overlying water concentrations were measured for each representative sediment sample of each site and spike concentration. Total Cr and Cr(VI) were both measured and Cr(III) calculated by difference for each sample. These values are reported in Table 12. Total Cr in the overlying water was generally below 10 µg/L with only a few exceptions. The overlying water for the baseline test reactor for site BSM-38 was anomalously high, averaging about 13 µg/L; however, no Cr(VI) was detected in the overlying water for this sample. The only overlying water that contained any quantifiable Cr(VI) was collected from the test beakers containing BSM 68 sediment spiked with 3210 µg/g dry weight Cr(VI). Total Cr in the overlying water for these test

conditions were 1455.9 µg/L, with Cr(VI) averaging 1054.1 µg/L and Cr(III) 401.8 µg/L. It should be pointed out, however, that the overlying water was changed periodically throughout the duration of the bioassay tests, and the chromium concentration in the overlying water was diluted by 60% with every water change. For BSM-68 where incomplete Cr(VI) reduction occurred, accumulation of Cr(VI) in the overlying water may have been appreciably higher than the reported values in the initial stages of the bioassay.

Table 12. Total Cr, Cr(VI) and Cr(III) balance for overlying water taken from bioassay test beakers.

Site	Total Cr (ug/L)	Cr(VI) (ug/L)	Cr(III) Balance (ug/L)
BSM-33			
Baseline	0.05	ND	0.05
Spike A (677 ug/g)	ND	ND	ND
Spike B (1650 ug/g)	ND	ND	ND
Spike C (2000 ug/g)	ND	ND	ND
BSM-38			
Baseline	13.4	ND	13.4
Spike A (383 ug/g)	ND	ND	ND
Spike B (1310 ug/g)	1.6	ND	1.6
Spike C (3090 ug/g)	1.6	ND	1.6
BSM-45			
Baseline	0.1	ND	0.1
Spike A (400 ug/g)	0.3	ND	0.3
Spike B (1670 ug/g)	1.5	ND	1.5
Spike C (4180 ug/g)	1.3	ND	1.3
BSM-54			
Baseline	0.5	ND	0.5
Spike A (408 ug/g)	ND	ND	ND
Spike B (1250 ug/g)	2.7	ND	2.7
Spike C (2920 ug/g)	9.2	ND	9.2
BSM-68			
Baseline	0.8	ND	0.8
Spike A (610 ug/g)	1.7	ND	1.7
Spike B (1810 ug/g)	3.1	ND	3.1
Spike C (3210 ug/g)	1455.9	1054.1	401.8

In addition, pore water was isolated from each sediment sample using centrifugation as described in ***Task 2.*** Total Cr concentrations were measured in the pore water for each sample at CTFR. The results are displayed in Table 13. Total Cr concentrations in pore water did not show much variation except for site BSM-33 that showed total Cr concentrations ranging from 0.9 to 27.2 µg/L. Although we observed high Cr(VI) concentrations in the overlying water of test beakers for BSM-68 spike C, we observed relatively low (10.3 µg/L) total Cr concentrations in the pore water of this sample. This finding may also be related to the periodic water renewal throughout the duration of the bioassay.

Table 13. Total Cr concentrations in pore water of spiked and unspiked Baltimore Harbor sediments.

Site	Total Cr (ug/L)
BSM-33	
Baseline	3.0
Spike A (677 ug/g)	13.8
Spike B (1650 ug/g)	27.2
Spike C (2000 ug/g)	1.0
BSM-38	
Baseline	3.7
Spike A (383 ug/g)	2.8
Spike B (1310 ug/g)	14.3
Spike C (3090 ug/g)	5.2
BSM-45	
Baseline	1.2
Spike A (400 ug/g)	1.5
Spike B (1670 ug/g)	1.2
Spike C (4180 ug/g)	0.9
BSM-54	
Baseline	1.0
Spike A (408 ug/g)	4.0
Spike B (1250 ug/g)	1.1
Spike C (2920 ug/g)	1.9
BSM-68	
Baseline	4.8
Spike A (610 ug/g)	6.1
Spike B (1810 ug/g)	4.8
Spike C (3210 ug/g)	10.3

Cr(VI) Re-spiking and Characterization

For site BSM-68 sediment, we observed toxicity to *L. plumulosus* at a Cr(VI) spike concentration of 3210 µg/g dry weight but not a spike concentration of 1810 µg/g dry weight, implying that the true lowest observed adverse effects level (LOAEL) for site BSM-68 lies somewhere between 1810 and 3210 µg/g dry weight of Cr(VI). In order to more accurately determine the threshold spike concentration required to elicit a toxic response, a range of Cr(VI) concentrations, from 1,500-3,000 µg/g dry weight, were spiked into BSM-68 sediment. The resulting Cr(VI) concentrations in the water column, pore water, and sediment are shown in Table 14. The results are also depicted graphically. As can be seen, the threshold spiking concentration appears to be approximately 2,250 µg/g. At this concentration, Cr(VI) is detected in pore water and the water column concentration increases significantly. Relatively little Cr(VI) was found to be associated with the solid phase (Table 14), indicating that little of the Cr(VI) remaining was adsorbed to the sediment.

The water column and pore water data are also graphed against the corresponding spike concentration in Figure 4. The data also show that a linear relationship exists between water column and pore water and the spiking concentration once the threshold Cr(VI) spiking concentration has been exceeded. Figure 5 shows a linear regression of the relationship between water column and pore water concentrations with the spiking concentrations above the threshold value. Also shown in Figure 5 is the AVS content of BSM-68 sediment. To first approximation, the Cr(VI) concentration in the pore water and overlying water appears to increase linearly once the Cr(VI) spike concentration exceeds the molar equivalent AVS concentration.

Table 14. Concentration of Cr(VI) in water column, pore water, and sediment samples for re-spiking experiment with BSM-68 sediment.

	Concentration of Cr(VI)		
	Water Column (ug/L)	**Porewater (ug/L)**	**Solid-associated Cr(VI) (ug/g sediment)**
BSM 68-1500 ug/g	4	-	-
BSM 68-1750 ug/g	133	-	-
BSM 68-2000 ug/g	252	-	-
BSM 68-2250 ug/g	3400	56.8	-
BSM 68-2500 ug/g	26200	28300	0.3
BSM 68-2750 ug/g	39300	94704	1.3
BSM 68-3000 ug/g	61700	170000	2.5

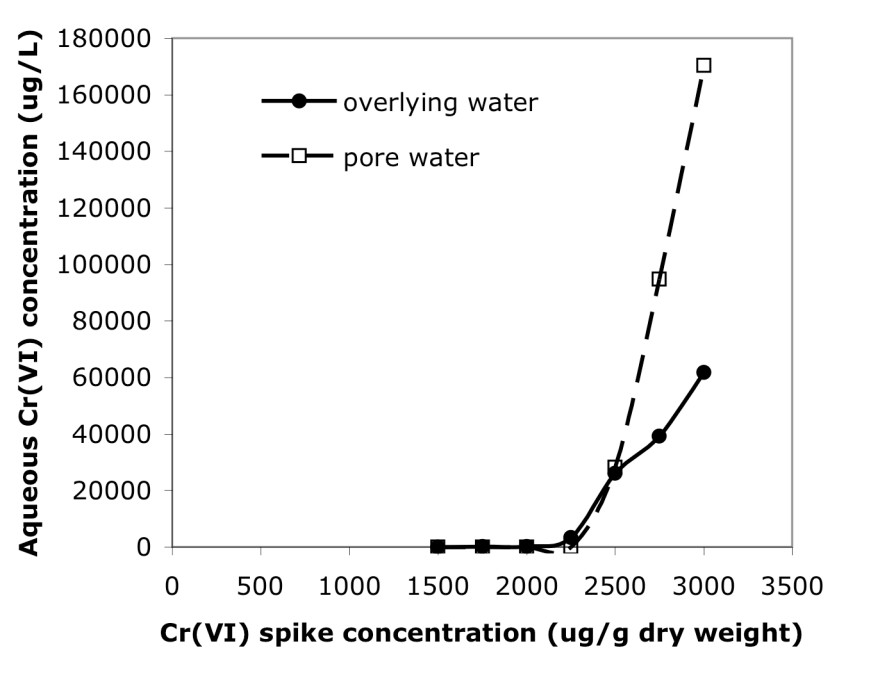

Figure 4. Overlying water and pore water concentrations vs. Cr(VI) spike concentrations for BSM-68 sediment.

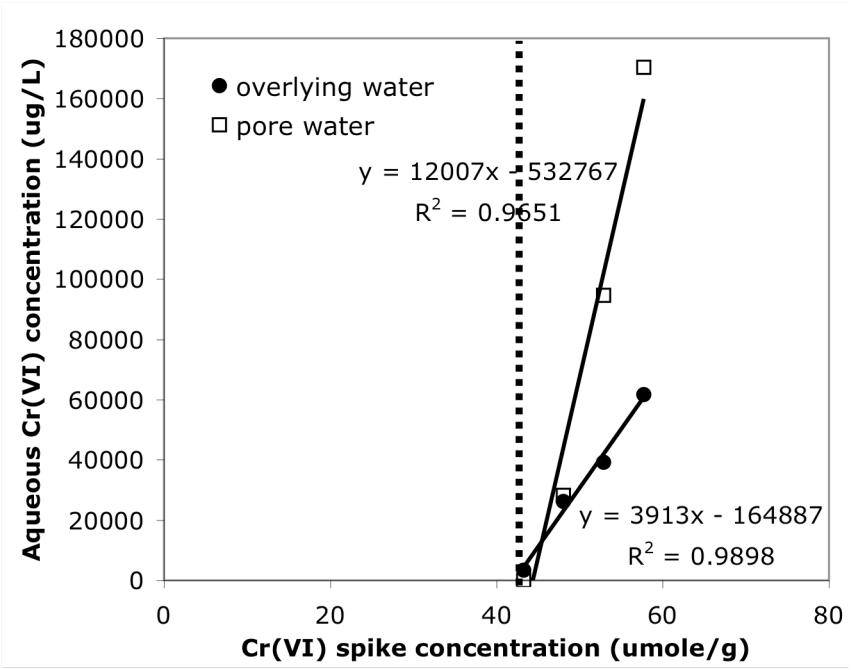

Figure 5. Linear relationship between overlying water and pore water concentrations and Cr(VI) spike concentrations for BSM-68 sediment. The dashed line indicates the AVS concentration of the sediment in µmole/g dry weight.

Analysis of Results

Sediment Toxicity

The results of both the acute and chronic toxicity data indicate that none of the spiked sediment samples displayed elevated toxicity when compared with baseline concentrations, with the exception of BSM-68 spiked at 3210 µg/g dry weight. To illustrate these trends, bar graphs of the toxicity data for each site are displayed below, with total Cr concentrations for the baseline and three spike levels. The acute toxicity data are provided in Figure 6, followed by the chronic toxicity data in Figure 7.

Looking at the graphs, it is clear that with the exception of BSM-68 spiked at 3210 µg/g, toxicity values do not vary from that observed for the baseline concentrations. Similar to previous findings (Berry *et al*, 2004; Besser *et al*., 2004), no correlation was observed between sediment toxicity and total Cr concentrations. Interestingly, some pre-existing chronic toxicity is observed for the baseline sediment samples; however, levels of chronic toxicity in the spiked samples are not elevated with respect to the baseline samples, excluding BSM-68 spiked with Cr(VI) at 3210 µg/g dry weight.

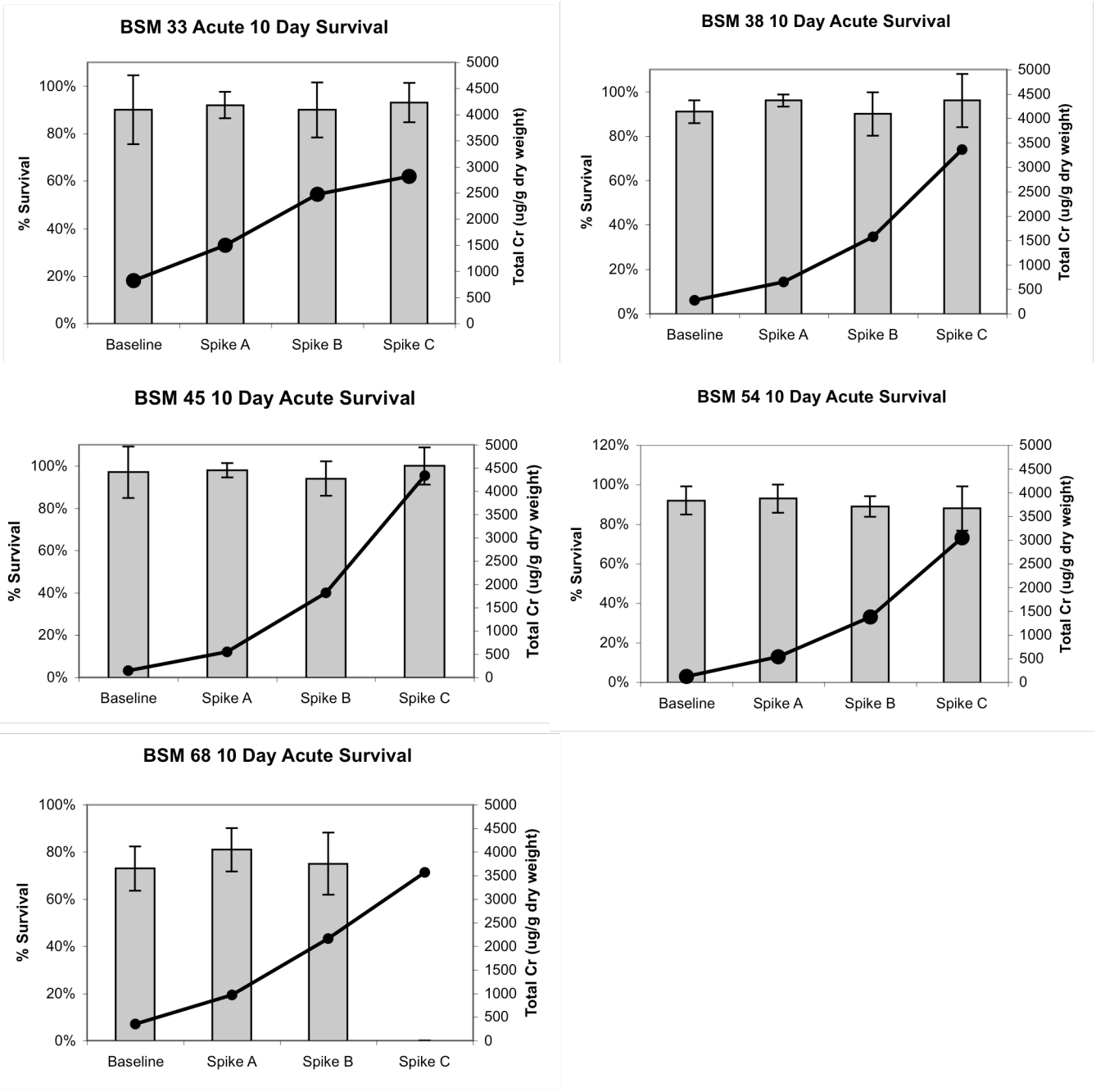

Figure 6. Acute toxicity and total Cr concentrations of BSM-33, BSM-38, BSM-45, BSM-54, and BSM-68 baseline and spiked sediments. Bars represent the mean acute 10 day percent survival for each of the tests and the filled circles represent the dry weight total Cr concentrations. Error bars indicate 95% confidence limits on toxicity data.

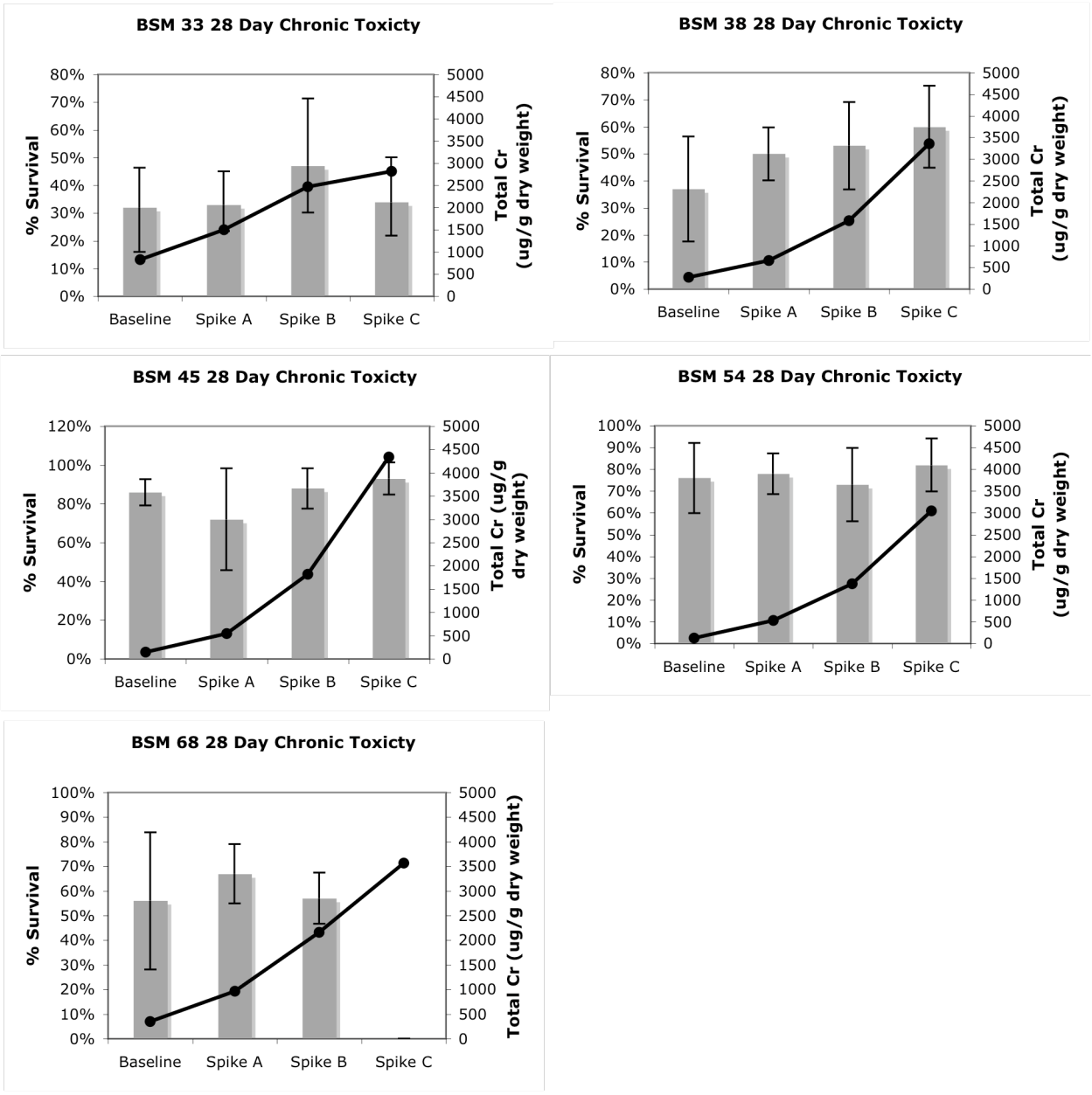

Figure 7. Chronic toxicity and total chromium concentrations of BSM-33, BSM-38, BSM-45, BSM-54, and BSM-68 baseline and spiked sediments. Bars indicate chronic 28 day percent survival and filled circles represent the total Cr concentration. Error bars indicate the 95% confidence limits for toxicity data.

The addition of Cr(VI) to sediments at concentrations at or exceeding environmentally relevant concentrations caused no changes in observed toxicity (e.g., no dose-response), with the exception of BSM-68 spiked at 3210 μg/g dry weight. Moreover, conclusions can also be drawn about the effects of residual chromium in the baseline sediment. Based on these findings, it can be concluded that the chromium already present in baseline sediment samples does not contribute to any observed toxicity. If chromium already present was contributing to acute or chronic toxicity, increases in this toxicity would be expected with even slight additions of chromium. Thus it can be concluded that chromium is not responsible for any of the observed acute or chronic toxicity in the Baltimore Harbor sediments sampled in this study. The data also lend further support to the hypothesis that ingestion of even high levels of trivalent chromium in sediment does not result in toxicity to the indigenous, sensitive amphipods used in these bioassays.

Cr(VI)/AVS Ratios as Predictors of Toxicity

The initial AVS data coupled with measured Cr concentrations for each sediment sample and spike concentration were used to calculate the ratio of added Cr(VI) to AVS for each sample. Numerous studies suggest that the metal/AVS ratio can be used as an indicator of toxicity (DiToro *et al*., 1992, Berry *et al.,* 1996). In the case of chromium, AVS constituents including FeS(s) and H_2S reduce Cr(VI) to Cr(III). As long as the total concentration of available sulfides exceeds the total Cr(VI) added to the sediment, all Cr(VI) should be quickly reduced to Cr(III). The results of these calculations for the sediment samples are displayed in the Table 15 below. AVS concentrations exceed spiked Cr(VI) concentrations in all sediments except for BSM-54 (spike C) and BSM-68 (spike C). It should be noted that both of these samples had Cr(VI) added/AVS molar ratios greater than unity but only the BSM-68 (spike C) sample showed acute or chronic toxicity. Incremental spiking of BSM-68 sediment revealed that aqueous Cr(VI) concentrations rapidly climbed above ambient water quality criteria once the Cr(VI) spike addition exceeded the reducing capacity of the sediment.

When excess reducing capacity remains, presumably in the form of AVS, aqueous and solid phase Cr(VI) concentrations are negligible. Because no toxicity was observed at a ratio slightly above one for BSM-54 (spike C) it seems probable that BSM-54 contained non-AVS reducing capacity (e.g. labile organic matter) that resulted in complete reduction of Cr(VI). It should also be noted that stoichiometries for Cr(VI) reduction by AVS are largely speculative, as AVS is a poorly defined operational quantity. Although a 1:1 stoichiometry for Cr(VI) reduction by FeS appears possible (Mullet *et al*., 2004), it cannot be concluded with absolute certainty that Cr(VI) will persist whenever Cr(VI)/AVS is greater than one. Rather, it appears that Cr(VI) is unlikely to persist and elicit toxic responses when the Cr(VI)/AVS ratio is less than one.

Table 15. Molar ratio of Cr(VI) added to AVS present for the five test sites and three different spike concentrations. For values of the various spike concentrations refer to the text or Table 1.

[Cr(VI)] added/AVS Molar Ratio					
	BSM-33	***BSM-38***	***BSM-45***	***BSM-54***	***BSM-68***
Spike A	0.03	0.08	0.08	0.17	0.32
Spike B	0.06	0.28	0.33	0.51	0.96
Spike C	0.11	0.67	0.83	1.19	1.71

Re-spiking and Characterization

In a re-spiking experiment, a range of chromium concentrations, from 1,500-3,000 µg/g, were spiked into BSM-68 sediment. The results allowed for determination of the threshold Cr(VI) spike concentration at which the reducing capacity of BSM-68 sediment would be exceeded. Based on the overlying water and pore water measurements of Cr(VI), the threshold concentration was measured at 2,250 µg/g. This spike concentration represented the lowest concentration where Cr(VI) was measured in the overlying water and pore water. One interpretation of these results is that the 2,250 µg/g Cr(VI) spike concentration likely represents the true lowest observable adverse effects level (LOAEL) for BSM-68. The bioassay data constrained this threshold to between 1810 and 3210 µg/g dry weight, so the result found here is

consistent with the bioassay data. Interestingly, nearly all of the added Cr(VI) remaining was present in the pore water and overlying water, with solid-associated Cr(VI) representing a negligible contribution.

Cr(VI)/AVS Ratio for Re-spiked Sediments

In the re-spiking experiments, the Cr:AVS ratio was again calculated for each of the spikes. For this calculation, the target spike concentrations were used to calculate the ratios, and AVS was measured again for BSM-68 at the time of the re-spikes due to the potential for change in AVS concentrations over time. The calculated ratios are displayed in Table 16. Looking at the table, at the spike concentration of 2,250 μg/g, the Cr(VI) concentration just exceeds the available AVS (with a ratio of 1.02). Because the Cr(VI) exceeds the AVS, it is possible that Cr(VI) added will be incompletely reduced, leading to the observed accumulation of Cr(VI) in the overlying water and pore water.

Table 16. Ratio of added Cr(VI) to AVS for re-spiked BSM-68 sediments.

BSM-68 Dry Weight Spike (ug/g)	Dry Weight Spike (umole/g)	Dry weight AVS (umole/g)	Total Cr :AVS Ratio
1500	28.8	42.3	0.68
1750	33.7	42.3	0.80
2000	38.5	42.3	0.91
2250	43.3	42.3	**1.02**
2500	48.1	42.3	**1.14**
2750	52.9	42.3	**1.25**
3000	57.7	42.3	**1.36**

Cr Calculations and Mass Balance for Re-spiked Sediments

Measurement of Cr(VI) in overlying water, pore water, and sediment permits mass balance on the Cr(VI) spikes added to BSM-68, allowing for calculation of the total mass of Cr(VI) reduced for the seven different spike concentrations. The overall percent reduction may be compared with the expected Cr(VI) reduction based on a 1:1 stoichiometry for Cr(VI) reduction by AVS. This comparison is plotted in Figure 8. We obtained relatively good agreement between experimental Cr(VI) reduction and predicted reduction, but, because the slope of the line in

Figure 8 is slightly less than 1, a 1:1 stoichiometry for Cr(VI) reduction by AVS actually under-predicts the observed experimental Cr(VI) reduction by BSM-68 sediment. This under-prediction may be due to less than 100% recovery of remaining Cr(VI) after spiking, the presence of other non-AVS reductants such as natural organic matter, or due to an alternative stoichiometry of Cr(VI) reduction by AVS.

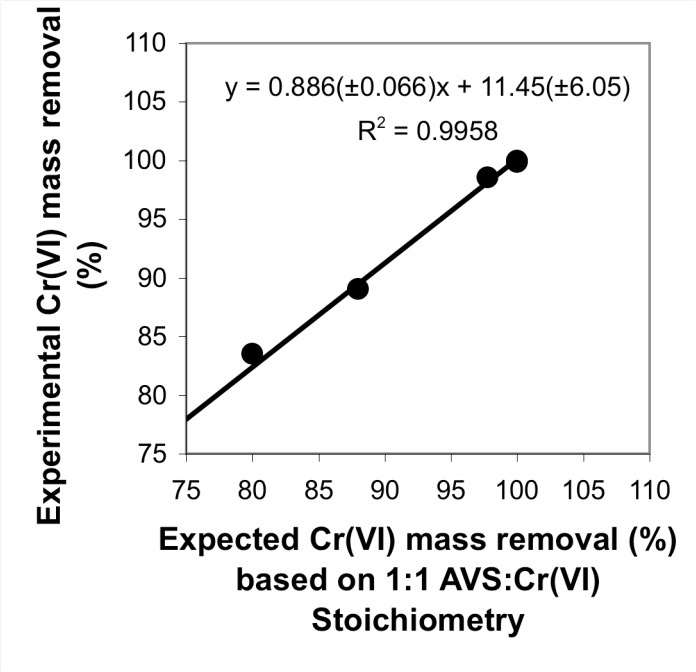

Figure 8. Expected Cr(VI) mass reduction based on 1:1 Cr(VI)/AVS stoichiometry versus observed experimental Cr(VI) mass reduction for BSM-68 sediments spiked at 1500, 1750, 2000, 2250, 2500, 2750, and 3000 μg/g.

Conclusions

The results of these experiments provide many insights into the nature of chromium in Baltimore Harbor sediments. It can be concluded that the addition of Cr(VI) concentrations, significantly above environmentally relevant levels, to Baltimore Harbor sediments does not cause additional acute or chronic toxicity to the amphipod *L. plumulosus*. In these studies, no spiked sediments demonstrated additional toxicity as a result of spiking with Cr(VI), with the exception of the

single sample spiked with 3210 µg/g dry weight of Cr(VI). Further, due to the lack of additional toxicity observed in conjunction with spiking sediment with Cr(VI), it can also be concluded that current chromium concentrations in Baltimore Harbor sediments are not causing the acute or chronic toxicity observed in baseline samples.

This research also generated evidence that supports the theory that AVS is an important mediator of chromium sediment toxicity. Looking at both the initial experiment, as well as the re-spiking studies, there was a high correlation between Cr(VI) reduction and sediment AVS concentrations. In the initial experiment, Cr(VI) was not completely reduced in only one sediment sample- BSM-68 spiked with 3210 µg/g dry weight Cr(VI). This also represented the only spike that exceeded a Cr(VI):AVS molar ratio of 1. Similarly, the experimentally determined threshold concentration for incomplete Cr(VI) reduction roughly corresponded to the predicted threshold concentration based on a 1:1 stoichiometry for Cr(VI) reduction by AVS. The Cr(VI) spike of 2,250 µg/g was the lowest spike where Cr(VI) was observed in the overlying water and pore water. It was also the lowest spike concentration that exceeded the AVS concentration on a molar basis.

In conclusion, these experiments produced three important insights regarding chromium and ecotoxicity of Baltimore sediments.

- First, it was demonstrated that current chromium concentrations are not responsible for observed toxicity to *L. plumulosus*.
- Second, the addition of Cr(VI), at concentrations well above environmentally relevant levels, to Baltimore Harbor sediments should not cause any additional toxicity.
- Finally, these experiments support the hypothesis that AVS constituents are the major contributors to Cr(VI) reduction in anoxic sediment. It also supports the hypothesis that the stoichiometry of Cr(VI) reduction by AVS occurs on a 1:1 basis, and AVS concentrations can be used as a predictor of potential toxicity due to Cr(VI) addition to sediment.

Acknowledgements

The authors wish to express thanks to Honeywell International, Inc. for financial support of the research described in this report.

References

American Public Health Association (APHA), American Waterworks Association, Water Environment Federation. 1998. Standard Methods for Examination of Water and Wastewater, 20th edition or most recent version. APHA, Washington, D.C.

Baker, J., R. Mason, J. Cornwell, J. Ashley, J. Halka, and J. Hill. 1997. *Spatial Mapping of Sedimentary Contaminants in the Baltimore Harbor/Patapsco River/Back River System*. Report to the Maryland Department of Environment: Baltimore, Maryland. UMCES [CBL] 97-142

Becker, D. S., E. R. Long, D. M. Proctor, and T. C. Ginn. 2006. Evaluation of potential toxicity and bioavailability of chromium in sediments with chromite ore processing residue. *Environ. Toxicol. Chem.* 25: 2576-2583.

Berry, W.J., D. J. Hansen, J. D. Mahony, D. L. Robson, D. M. Di Toro, B. P. Shipley, B. Rogers, J. M. Corbin, and W.S. Boothman. 1996. Predicting the toxicity of metal-spiked laboratory sediments using acid-volatile sulfide and interstitial water normalizations. *Environ. Toxicol. Chem.* 15: 2067-2079.

Berry, W. J., W. S. Boothman, J. R. Serbst, and P. A. Edwards. 2002. Effects of Chromium in Sediment: 1. Toxicity Tests with Saltwater Field Sediments. U.S. Environmental Protection Agency, National Health and Environmental Effects Research Laboratory , Atlantic Ecology Division. Narragansett, RI. Presented at SETAC 23rd Annual Meeting Salt Lake City, Utah. 16 - 20 November 2002.

Berry, W. J., W. S. Boothman, J. R. Serbst, and P. A. Edwards. 2004. Predicting the toxicity of chromium in sediments. *Environ. Toxicol. Chem*. 23: 2981-2992.

Besser, J. M., W. G. Brumbaugh, N. E. Kemble, T. W. May, and C. G. Ingersoll. 2004. Effects of sediment characteristics on the toxicity of chromium(III) and chromium(VI) to the amphipod, hyalella azteca. *Environ. Sci. Technol.* 38: 6210-6216.

Boothman, W. S. and A. Helmstetter. 1992. Vertical and seasonal variability of acid volatile sulfides in marine sediments. U.S. EPA Report 600/X-93/036. Narragansett, RI.

Boursiquot, S., M. Mullet, and J.-J. Ehrhardt. 2002. XPS study of the reaction of chromium(VI) with mackinawite (FeS). *Surface and Interface Science* 34: 293-297.

Bufflap, S. E. and H. E. Allen. 1995. Comparison of pore water sampling techniques for trace metals. *Water Research* 29: 2051-2054.

Chang, Y. L. and S. J. Jiang. 2001. Determination of chromium species in water samples by liquid chromatography-inductively coupled plasma-dynamic reaction cell-mass spectrometry. *J. Anal. At. Spectrom.* 16: 858-862.

Di Toro, D. M., J. D. Mahony, D. J. Hansen, K. J. Scott, A. R. Carlson, and G. T. Ankley. 1992. Acid volatile sulfide predicts the acute toxicity of cadmium and nickel in sediments. *Environ. Sci. Technol.* 26: 96-101.

Driscoll, S. K., G. A. Harkey, and P. F. Landrum. 1997. Accumulation and toxicokinetics of flouranthene in sediment bioassays with freshwater amphipods. *Environ. Toxicol. Chem.* 16: 742-753.

Ho. K. T., R. M. Burgess, M. C. Pelletier, J. R. Serbst, S. A. Ryba, M. G. Cantwell, A. Kuhn, and P. Raczelowski. 2002. An overview of toxicant identification in sediments and dredged materials. *Marine Pollution Bulletin* 44: 286-293.

James, B. R., J. C. Petura, R. J. Vitale, and G. R. Mussoline. 1995. Hexavalent Chromium Extraction from Soils: A Comparison of Five Methods. *Environ. Sci. Technol.* 29: 2377-2381

Klosterhaus, S., J. Baker, G. Zeigler, and D. Fisher. 2006. Toxicity Identification and Evaluation and Long-Term Contaminant Trends in the Baltimore Harbor, DRAFT. Submitted to: Technical and Regulatory Services Administration, Maryland Department of the Environment

Lee, B.-W., S. B. Griscom, J.-S. Lee, H. J. Choi, C.-H. Koh, S. N. Luoma, and N. S. Fisher. 2000. Influences of Dietary Uptake and Reactive Sulfides on Metal Bioavailability from Aquatic Sediments. *Science* 287: 282-284.

Long, E. R., D. D. MacDonald, J. C. Cubbage, and C. G. Ingersoll. 1998. Predicting the toxicity of sediment-associated trace metals with simultaneously extracted trace metal: acid-volatile sulfide concentrations and dry weight-normalized concentrations: a critical comparison. *Environ. Toxicol. Chem.* **17:** 972-974.

Maryland Department of the Environment. 2005. Water Quality Analysis of Chromium in the Inner Harbor/Northwest Branch and Bear Creek Portions of Baltimore Harbor in Baltimore City and Baltimore County, Maryland. Submitted to: Watershed Protection Division, USEPA.

McGee, B. L., D. J. Fisher, L. T. Yonkos, G. P. Ziegler, and S. Turley. 1999. Assessment of sediment contamination, acute toxicity, and population viability of the estuarine amphipod Leptocheirus plumulosus in Baltimore Harbor, Maryland, USA. Environ. Toxicol. Chem. 18: 2151-2160.

McGee, B. L., D. J. Fisher, D. A. Wright, L. T. Yonkos, G. P. Ziegler, S. D. Turley, J. D. Farrar, D. W. Moore, and T. S. Bridges. 2004. A field test and comparison of acute and chromic sediment toxicity tests with the estuarine amphipod *Leptocheirus plumulosus* in Chesapeake Bay, USA. *Environ. Toxicol. Chem.* 23: 1751-1761.

Mullet, M., S. Boursiquot, and J.-J. Ehrhardt. 2004. Removal of hexavalent chromium from solutions by mackinawite, tetragonal FeS. *Colloids and Surfaces A: Physiochemical and Engineering Aspects* 244: 77-85.

Murdoch, M. H., P. M. Chapman, D. M. Norman, and V. M. Quintino. 1997. Spiking sediment with organochlorines for toxicity testing. *Environ. Toxicol. Chem.* 16: 1504-1509.

Oshida, P.S., L. S. Word, and A. J. Mearns. 1981. Effects of Hexavalent and Trivalent Chromium on the Reproduction of *Neanthes arenaceodetata* (Polychaeta). *Mar. Env. Res.* 5: 41-49

Rickard, D. and J. W. Morse. 2005. Acid volatile sulfide (AVS). *Marine Chemistry*. 97:141-197.

Rifkin, E., P. Gwinn, and E. Bouwer. 2004. Chromium and sediment toxicity. *Environ. Sci. Technol.* 38: 267A-271A.

Tan, K. H. 1996. Soil Sampling, Preparation, and Analysis. Marcel Dekker, NY, 480 p.

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

U.S. Environmental Protection Agency. 1994. EPA Method 3051A- Microwave assisted acid digestion of sediments, sludges, soils, and oils. SW-846.

U.S. Environmental Protection Agency. 1996. EPA Method 3060A- Alkaline Digestion for Hexavalent Chromium. SW-846.

U.S. Environmental Protection Agency. 1997. The Incidence and Severity of Sediment

Contamination in Surface Waters of the United States: National Sediment Quality Survey, Second Edition. EPA/823/R-97-006. Office of Science and Technology, Washington, D.C.

U.S. Environmental Protection Agency. 2001. Methods for Assessing the Chronic Toxicity of Marine and Estuarine Sediment-associated Contaminants with the Amphipod *Leptocheirus plumulosus*, First Edition. EPA/600/R-01020. Office of Research and Development, Washington, D.C.

U.S. Environmental Protection Agency. 2004. The Incidence and Severity of Sediment Contamination in Surface Waters of the United States: National Sediment Quality Survey, Second EditionEPA/823/R-04-007. Office of Science and Technology, Washington, D.C.

U.S. Environmental Protection Agency. 2004b. EPA Method 9045D- Soil and Waste pH. SW-846.

U.S. Environmental Protection Agency. 2004c. EPA Method 9060A- Total Organic Carbon.

Verrhiest, G., B. Clement, and G. Blake. 2001. Single and combined effects of sediment associated PAHs on three species of freshwater macroinvertebrates. *Ecotoxiciology.* 10: 363-372.

Watlington, K., A. Graham, and E. Bouwer. 2007. The Sediment Ingestion Pathway as a Source of Toxicity in Baltimore Harbor, Literature Review Report. Submitted to Technical and Regulatory Services Administration, Maryland Department of the Environment, Baltimore, MD.

Winger, P.V., P.J. Lasier, and B.P. Jackson. 1998. The influence of extraction procedure on ion concentrations in sediment pore water. *Archives Environ. Contam. Toxicol.* 35: 8-13.

Appendix A: Previous site characterization data from the University of Maryland DRAFT TIE report (Klosterhaus *et al.*, 2006)

General Data for Sample Sites (University of Maryland TIE Report 2006)							
Site Name		**Latitude**	**Longitude**	**Sediment Carbon (%)**	**Pore Water DOC (mg/L)**	**Pore Water Ammonia (mg/L)**	**Pore Water Sulfide (mg/L)**
BSM33	Bear Creek #6	39.2536	76.4903	6	22.3	10.2	<0.18
BSM38	Colgate Creek	39.2564	76.5361	4.3	10.5	55.6	<0.18
BSM45	Curtis Bay	39.2175	76.5767	5.3	27	9.9	<0.18
BSM54	Lazaretto Pt.	39.2583	76.5683	3.9	13.4	11	<0.18
BSM68	Northwest Branch	39.2778	76.5833	5.7	14.5	11.6	<0.18

Sediment Grain Information for Sample Sites (University of Maryland TIE Report 2006)							
Site Name	**%H20**	**Bulk Density**	**% Gravel**	**% Sand**	**% Silt**	**%Clay**	**Shepard's Classification**
BSM33	68.2	1.25	0	14.69	38.47	46.84	Silty-Clay
BSM38	71.06	1.22	0	14.15	34.18	50.87	Silty-Clay
BSM45	66.68	1.27	0	13.93	29.71	56.36	Silty-Clay
BSM54	67.8	1.26	1.1	20.53	26.48	51.89	Sand-Silt-Clay
BSM68	70.94	1.23	0	14.81	37.58	47.62	Silty-Clay

	Metals in sediment (ug/g dry) (University of Maryland TIE Report 2006)						
Site Name	**Cr**	**Cu**	**Zn**	**Cd**	**Pb**	**As**	**Ag**
BSM33	860	211	1640	7.97	265	41.2	2.44
BSM38	213	226	449	16	184	28.8	1.84
BSM45	265	269	487	1.42	270	46.5	1.98
BSM54	246	150	335	1.75	134	23.9	1.72
BSM68	472	356	529	2.95	335	30	3.07

	Metals in Baltimore Harbor pore water (µg/L) (University of Maryland TIE Report 2006)						
Site Name	**Cr**	**Cu**	**Zn**	**Cd**	**Pb**	**As**	**Ag**
BSM33	4.33	20.5	1.35	0.061	0.042	10.2	<MDL
BSM38	11.6	62.7	1.28	0.02	0.056	12.3	0.038
BSM45	1.88	24.3	2.22	0.023	0.026	25.1	<MDL
BSM54	3.24	31.7	1.95	0.021	0.045	23.9	0.023
BSM68	4.54	24.8	1.51	0.042	0.103	11	0.053

	Organics in Baltimore Harbor Sediments (ng/g) (University of Maryland TIE Report 2006)					
Site Name	**Total PAHs**	**Total PCBs**	**Total BDEs**	**TBT**	**DBT**	**MBT**
BSM33	6700	180	360	26	<DL	<DL
BSM38	9100	150	90	74	<DL	<DL
BSM45	11600	440	130	31	2	<DL
BSM54	6500	90	60	130	2	<DL
BSM68	15100	210	100	97	30	<DL

	Organics in Baltimore Harbor Pore Water (ng/g) (University of Maryland TIE Report 2006)					
Site Name	**Total PAHs**	**Total PCBs**	**Total BDEs**	**TBT**	**DBT**	**MBT**
BSM33	140	3.8	0.1	<DL	<DL	<DL
BSM38	300	2.5	<DL	<DL	<DL	<DL
BSM45	180	23.6	<DL	<DL	<DL	<DL
BSM54	110	2.2	<DL	<DL	<DL	<DL
BSM68	210	8.7	0.1	<DL	<DL	<DL

	Toxicity of Baltimore Harbor Sediments, (McGee 1999[1], 2004[2])	
Site Name	**Acute Toxicity (% Survival)**	**Chronic Toxicity (% Survival)**
BSM33	65.7[2]	39.0[2]; 80.5[2]
BSM38	55[1]	
BSM45	76.4[1]; 46.7[2]	68.0[2]; 99.6[2]
BSM54	83.1[1]	
BSM68	53.0[1]	

Appendix B: Sample Characterization Protocols

Digestion for Cr(VI) – A Modified Version of EPA 3060A – Alkaline Digestion for Hexavalent Chromium

I. Materials

- Teflon microwave digestion vessels
- CEM MARS X-press Microwave Digestion Unit
- 0.2 um nylon membrane syringe filters
- 10 mL polypropylene syringes
- 15 mL polypropylene centrifuge tubes
- pH meter
- Analytical balance
- NIST approved temperature measurement device

II. Reagents

- 5.0 M HNO_3
- Anhydrous Na_2CO_3
- NaOH pellets
- Lead chromate (insoluble matrix spike) – 10-20 mg $PbCrO_4$
- Digestion Solution: 20.0 g NaOH and 30.0 g Na_2CO_3 in 1 L flask and dilute to mark (**IMPT**: pH must be greater than 11.5)
- $K_2Cr_2O_7$ spiking solution (1000 mg/L Cr(VI)): dissolve 2.829 g of dried (105 ^oC) $K_2Cr_2O_7$ in 1 L flask and dilute to mark (certified primary standard may used instead)
- Matrix spiking solution (100 mg/L): add 10 mL of 1000 mg/L Cr(VI) solution to 100 mL flask and dilute to volume

III. Procedure

1. Weight ~1.0 g of field moist sediment into microwave digestion vessel.
2. Add 10 mL of alkaline digestion solution (described above)
3. Hand mix sediment and alkaline digestion solution
4. Microwave for 60 min at 95 ^0C
5. Allow vessels to cool, and pour contents into 15 mL centrifuge tubes
6. Centrifuge for 30 min at 4000xg
7. Filter supernatant through 0.2 um nylon membrane filter
8. Dilute filtered supernatant at least 5x into HPLC mobile phase (2 mM TBAH, 0.6 mM EDTA, at pH 6.9-7.0)
9. Adjust pH of sample to ~7 with concentrated HNO_3 (generally need to add about 10 uL of concentrated nitric to a 1 mL sample)
10. Transfer quantitatively contents of vessel to 100 mL flask and adjust volume to 100 mL with reagent water.

IV. QA/QC

1. Preparation blank prepared and analyzed with each digestion batch – detected Cr(VI) must be below detection limit or 10% regulatory limit
2. Laboratory control sample (LCS) – utilize matrix spike solution or solid matrix spiking agent into 50 mL of digestion solution

3. Separately prepared duplicate samples analyzed once per batch (Relative percent difference less than 20% required)
4. Soluble and insoluble pre-digestion matrix spikes analyzed once per batch of 20 samples – Acceptable spike recovery 75-125%
    a. Soluble spike – 1.0 mL of spiking solution (40 mg/kg) or at twice sample concentration (greater of two)
    b. Insoluble spike – 10-20 mg $PbCrO_4$ to sample
5. Compare LCS and matrix spike data. If LCS data is good, but matrix spike a failure, sediment may be highly reducing and low recovery would be expected. Measure pH and oxidation reduction potential (convert to E_h) of sediment and evaluate reducing or oxidizing properties of sediment.
6. If 0% matrix spike obtained – perform mass balance on total Cr analyzed for two samples:
    a. Separate unspiked aliquot of sample used for spiking
    b. Digested solids remaining after alkaline digestion and filtration – difference should equal to matrix spike added.
7. If 0% matrix spike recovery obtained, perform a series of spike additions of increasing spike concentration to exceed reducing capacity. Determine if response beyond reducing capacity is linear and quantitative.
8. Post-digestion Cr(VI) matrix spike once per batch (40 mg/kg or twice sample concentration, whichever is greater) – 85-115% recovery guideline

V. References

James, B. R.; J. C. Petura; R. J. Vitale; and G. R. Mussoline. Hexavalent chromium extraction from soils: a comparison of five methods. *Environ. Sci. Technol.* **1995,** *29*, 2377-2381.

Pettine, M. and S. Capri. Digestion treatments and risks of Cr(III)-Cr(VI) interconversions during Cr(VI) determination in soils and sediments -- a review. *Analytica Chimica Acta* **2005,** *540*, 231-238.

U.S. Environmental Protection Agency. EPA Method 3060A- Alkaline Digestion for Hexavalent Chromium. SW-846. Revised December, 1996.

Vitale, R. J.; G. R. Mussoline; K. A. Rinehimer; J. C. Petura; and B. R. James. Extraction of sparingly soluble chromate from soils: evaluation of methods and E_h -- pH effects. *Environ. Sci. Technol.* **1997,** *31*, 390-394.

Digestion for Total Metals – EPA Method 3051 – Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils

I. Materials

- Volumetric grad cylinder – 50 or 100 mL
- Filter paper
- Filter funnel
- Analytical balance
- NIST approved temperature measurement device
- Fluorocarbon beaker

II. Reagents

- High purity concentrated HNO_3
- Reagent water
- 2% v/v HNO_3 solution prepared using concentrated nitric and DI water

III. Procedure

1. Weigh the PFA or TFM vessel, cap assembly, and valve to nearest 0.001 g
2. Weigh well-mixed sample to nearest 0.001 g using no more than 0.500 g (recommend 0.250 g or 0.100 g if carbonates or easily oxidized organics present in sample)
3. In fume hood, add 10 +/- 0.1 mL HNO_3 (If reaction occurs, allow reaction to stop before capping)
4. Cap vessels and weigh to nearest 0.001 g
5. Place carousel in microwave and connect pressure vessels to central overflow vessel (**IMPT** – All sample locations should be filled – fill extra vessels with 10 mL of HNO_3 if necessary)
6. Irradiate for 10 min. Temperature should rise to 175 ^oC in less than 5.5 min and remain between 170-180 ^oC for balance of 10 min period. Pressure should peak at ~ 6 atm.
7. Allow vessels to cool for at least 5 min before removing from microwave
8. Weigh each vessel assembly – if total weight has decreased by more than 10%, sample must be discarded
9. Uncap and vent each bottle in fume hood
10. Transfer sample to acid cleaned bottle
11. Centrifuge (2,000-3,000 rpm for 10 min) and settle
12. Dilute supernatant to 2% v/v HNO_3 using DI water
13. Filter diluted sample through 0.2 um nylon membrane to remove any remaining particles
14. Dilute to known volume using 2% v/v HNO_3 as diluent.

IV. QA/QC

- Duplicate samples
- Standard Reference Material every batch/20 samples – 85-115% recovery required. If standard not met, SRM run again. If satisfactory result still not obtained, entire batch of samples must be run again following identification of potential error.
- Sample Spikes every batch/20 samples
- Digestion with HF will permit determination of fraction of Cr associated with mineral phases not digested using nitric acid

V. References

U.S. Environmental Protection Agency. EPA Method 3051- Microwave assisted acid digestion of sediments, sludges, soils, and oils. SW-846. Revised September, 1994.

Cr Speciation Determination by HPLC-ICP-MS

I. Materials

- Autosampler vials
- Volumetric flasks for standard prep

II. Reagents

- 1.0 M solution of tetrabutylammonium hydroxide (TBAH)
- potassium salt of EDTA

- MeOH
- High purity concentrated HNO_3
- Mixed calibration stds – dilute stock-standards to levels in linear range for instrument in 1% v/v HNO_3 and reagent water to obtain appropriate concentration for each analyte of interest
- Cr(VI) std solution – prepared from $K_2Cr_2O_7$
- Cr (III) std solution – prepared from $CrCl_3$
- Cr(III) and Cr(VI) calibration standards in range of 0.5 to 100 ug/L diluted in HPLC mobile phase
- Calibration blank for ICP-MS (1% v/v HNO_3 in reagent water)
- Cal. Blank for HPLC-ICP-MS

III. Procedure

Mobile Phase Prep

- 2 mM TBAH
- 0.6 mM EDTA
- pH adjusted to 6.9-7.0 with concentrated HNO_3

Column Preparation

- Upon arrival column should be washed for 90 min with 100% MeOH at 1.0 mL/min
- Prior to daily use, run mobile phase through column for 30 min.
- Wash at end of day with 70/30 v/v MeOH/water

General ICP-MS Operating Procedure

1. Allow greater than 30 min for instrument equilibration
2. Verify instrument stability with tuning solution (at least 4 times with RSD less than 5%
    a. Mass calibration should not differ more than 0.1 amu from true value; mass calibration should be adjusted to correct value
    b. Resolution should be less than 0.9 amu full width at 10% peak height
3. Calibrate for analytes of interest – blank and at least a single calibration std. Flush with rinse blank b/w each std. Use average of more than 3 integrations for calibration and sample analysis
4. Flush with rinse blank (30 s) until signal returns to quantitation level. Nebulize sample until steady state signal is achieved. Analyze calibration check standard and calibration blank at least once every 10 samples

HPLC-ICP-MS Calibration Procedure

a. Calibrate from 50 uL injections of 0.1, 0.5, 1, 5, and 10 ug/L of each Cr species in mobile phase

Table 1 – Operating Conditions for ICP-MS and DRC – from PE pamphlet and Chang and Jiang, 2001

Plasma Conditions	
rf power	1050 – 1175 W
Ar plasma flow	15 L/min
Auxiliary Ar flow	1.2 L/min
Nebulizer Ar flow	0.95 L/min
Mass spectrometer settings	
Dwell time	300-1000 ms

Sweeps	1
Readings	250
Isotopes Monitored	52Cr (Chang and Jiang also monitored 53Cr)
DRC parameters	
NH3 reaction gas flow	0.40 mL/min to 0.65 mL/min
Quadropole rod offset	-8.5 V
Cell path voltage	-29 V
Cell rod offset	0 V
Rejection parameter q	0.4 to 0.70
Rejection parameter a	0
Autolens	On

Table 2 – HPLC conditions – modified from PE pamphlet and Chang and Jiang, 2001

Mobile Phase	2 mM TBAH+ (Chang and Jiang, 2001 used TBAP) 0.6 mM EDTA (potassium salt); pH 6.9
Flow Rate	1.0 mL/min to 1.5 mL/min
Column	3x3 CR C8
Sample volume	50 uL to 100 uL
Autosampler Flush Solvent	5% MeOH/95% water

IV. QA/QC

1. Instrument detection limit – calculated as std deviation of three runs on three non-consecutive days of blank with seven consecutive measurements per day (DL must be determined every three months)
2. Dilution test – analysis of five fold dilution must agree with in 10% of original determination otherwise interference suspected. Run dilution test once per 20 samples
3. Post-digestion spike addition – 75-125% recovery required
4. Laboratory control sample – same sample prep, analytical methods and QA/QC procedures as test samples. Run once per 20 samples
5. Results of calibration blank must be within 3 x DL for each element
6. Check standards should be within 10% of expected values – run with each batch of samples.

V. References

Chang, Y-L and S-J Jiang. Determination of chromium in water and urine by reaction cell inductively coupled plasma mass spectrometry. *J. Anal. At. Spectrom.* **2001,** *16*, 1434-1438.

Montes-Bayon, M.; K. DeNicola; and J. A. Caruso. Liquid chromatography-inductively coupled plasma mass spectrometry. *J. Chromatography A* **2003,** *1000*, 457-476.

Neubauer, K.; W. Reuter; and P. Perrone. Chromium speciation in water by HPLC/ICP-MS. Perkin Elmer Bulletin. **2003**.

Pantsar-Kallio, M. and P. K. G. Manninen. Speciation of chromium by coupled column HPLC-ICP-MS-the effects of interfering ions. *Fresenius J. Anal. Chem*. **1996,** *355*, 716-718.

Powell, M. J.; D. W. Boomer; and D. R. Wiederin. Determination of chromium species in environmental samples using high-pressure liquid chromatography direct injection nebulization and inductively coupled plasma mass spectrometry. *Anal. Chem*. **1995,** *67*, 2474-2478.

Seby, F.; S. Charles; M. Gagean; H. Garraud; and O. F. X. Donard. Chromium speciation by hyphenation of high-performance liquid chromatography to inductively coupled plasma-mass spectrometry -- study of the influence of interfering ions. *J. Anal. At. Spectrom.* **2003,** *18*, 1386-1390.

U.S. Environmental Protection Agency. EPA Method 6020- Inductively Coupled Plasma-Mass Spectroscopy. SW-846. Revised September, 1994.

Pore water Extraction by Centrifugation

I. Materials

- High speed centrifuge
- 250 mL polycarbonate centrifuge tubes
- Analytical balance
- 0.2 um membrane filters
- disposable plastic syringes with plastic tips

II. Reagents

- LC mobile phase for dilution

III. Procedure

1. Weigh centrifuge tube and cap
2. Weigh out desired sediment quantity (50 g) into centrifuge tube
3. Run for 20-30 min at 4 ^{o}C at 4000xg or 10000xg
4. Withdraw sample with syringe and filter supernatant through 0.2 um membrane filter into cleaned sample vial
5. Dilute to appropriate volume with LC mobile phase

IV. QA/QC

1. Mass balance to determine extraction efficiency – comparison of weight of pore water extracted vs. total pore water weight determined by sample drying
2. Duplicate extractions

V. References

Ankley, G. T., and M.K. Schubauer-Berigan. 1994. Comparison of techniques for the isolation of sediment pore water for toxicity testing. *Archives Environ. Contam. Toxicol*. **27**, 507-512.

Annual Book of ASTM Standards, 2002. ASTM E 1391-94: Standard guide for collection, storage, characterization, and manipulation of sediments for toxicological testing. ASTM Int.

Carignan, R., F. Rafin, and A. Tessier. 1985. Sediment pore water sampling for metal analysis: A comparison of techniques. *Geochim. Cosmo. Acta*, **49**, 2493-2497

Winger, P.V., P.J. Lasier, and B.P. Jackson. 1998. The Influence of extraction procedure on ion concentrations in sediment pore water. *Archives Environ. Contam. Toxicol*. **35**, 8-13.

Determination of Acid Volatile Sulfides – from Boothman and Helmstetter, 1993

I. Materials

- Impinger bottles
- Round bottom flasks
- N_2 gas cylinder
- Tygon tubing
- Flow controller
- O_2 scrubber
- pH meter
- sulfide electrode
- reference electrode
- 100 mL volumetric flasks
- 150 mL beakers
- magnetic stirrer and stir bars
- 1 L volumetric flasks
- Plastic syringes
- Needles (Luer tip, 10 mL)
- Analytical balance
- 125 mL Erlenmeyer flasks
- glass pipettes – assorted

II. Reagents

- Deaerated DI water (DDIW)
    - Bubble N_2 through 2.5 L of DI water for 1 hour
- 6 M HCl
- SAOB solution (2 M NaOH, 0.2 M EDTA, 0.2 M ascorbic acid)
    - Dissolve 80.00 g NaOH slowly in 700 mL DDIW
    - When cool, add 74.45 g EDTA (disodium form) and stir
    - Add 35.23 g ascorbic acid
    - Pour solution into 1 L volumetric flask and dilute to mark
- Primary Sulfide Standard
    - Wash crystals of $Na_2S{\cdot}9H_2O$ with DDIW
    - Weigh out 12 g $Na_2S{\cdot}9H_2O$ and dissolve in 900 mL DDIW
    - Pour into 1 L volumetric flask and dilute to volume

- SAOB diluent
  - Mix 300 mL of SAOB with 300 mL DDIW
- Working stock solution
  - 50 mL of SAOB diluent in 100 mL volumetric
  - Pipette in appropriate volume of primary standard and dilute to volume
- Standard Iodine Solution (0.025 N)
  - Dissolve 20-25 g KI in 100 mL DI water
  - Weigh out 3.2 g I_2 and dissolve in KI solution
  - Pour solution into 1 L volumetric flask and dilute to mark
- Thiosulfate titrant (0.025 N)
- Starch indicator
  - 1.0 g starch in 100 mL boiling DI water

III. Procedure

Standardization of Primary Stock Solution

1. Pipette 10.00 mL of standard iodine solution into each of two 125 mL Erlenmeyer flasks
2. Pipette 2.000 mL of sulfide primary stock solution into one flask, and 2.000 mL DDIW into the other
3. Add 5.00 mL of 6 M HCl into each flask, swirl, cover, and place in dark for 5 min
4. Titrate each with 0.025 N thiosulfate solution, adding starch indicator when yellow iodine color fades. End point is reached when blue color disappears.
5. Sulfide concentration is calculated as:

$$\text{sulfide } (\mu\text{mol/mL}) = \frac{(V_{tblank}-V_{tsample})[S_2O_3^{2-}]}{V_{sample}} \times \frac{1 \text{ mole } S^{2-}}{2 \text{ equiv } S^{2-}} \times \frac{1000\mu\text{mole}}{1\text{mmol}}$$

where: V_{tblank}= volume of titrant added to blank
V_{tsampe}= volume of titrant added to sample
V_{sample} = volume of sample (2.000 mL)
$[S_2O_3^{2-}]$ = concentration of $S_2O_3^{2-}$ in mmol/mL

Calibration of Standards

1. Prepare 6 calibration standards from working stock solution that cover range of expected AVS concentrations
2. Pour standard into 150 mL beaker, add magnetic stir bar and place on stirrer. Stir with minimum agitation so as to minimize oxidation
3. Rinse electrodes with DDIW and blot dry. Immerse electrodes into sample
4. Allow 8-10 min for electrode response to stabilize and then record the reading in mV
5. Construct a calibration curve by plotting potential in mV vs. log concentration.

AVS recovery

1. Purge apparatus shown below with N_2 for at least 30 min prior to sample introduction
2. Fill impinger bottles with 50 mL of SAOB and 30 mL of DDIW
3. Weigh out 1-2 g of wet sediment into 250 mL round bottom flask. Add 50 mL DDIW to cover sediment. Add stir bar
4. Run purge gas flow at 100 mL/min for 10 min. Then reduce flow to 40 mL/min
5. Stop gas flow and slowly inject 10 mL of 6 M HCL with syringe through septum sidearm
6. Resume gas flow at 40 mL/min and stir well for 30 min.
7. Stop gas flow. Rinse impinger bottles with DDIW into 100 mL volumetric flask. Dilute to volume
8. Pour contents into 150 mL and immerse electrodes. Record measurement in mV.

IV. QA/QC

1. Duplicate analyses – calculated concentrations should differ by no more than 15% of the mean of the two values.
2. Calibration blank
    a. Add 25 mL SAOB and 15 mL of DDIW to 50 mL volumetric flask
    b. Add 1-4 mL of secondary stock solution. Dilute to volume
    c. Measured concentration should be within 15% of the expected value
3. Blank spike
    a. Prepare apparatus as if sample, but without sediment.
    b. Add 50 mL of DDIW and spike with 1-5 mL secondary stock solution
    c. Calculate % recovery – should be between 85-115%. If not, examine apparatus for sources of error. Correct problems and demonstrate successful blank spike recovery before analyzing sediment samples

4. Sample Spike
    a. Prepare sediment for analysis that has already been well characterized for AVS concentration
    b. Add 1-5mL spike from secondary stock solution
    c. Measure sulfide as before and calculate percent recovery – should fall between 85-115%. Repeat spike if desired recovery not obtained. If recovery is still low, attempt spike with lower sediment sample quantity to help reduce matrix interferences.

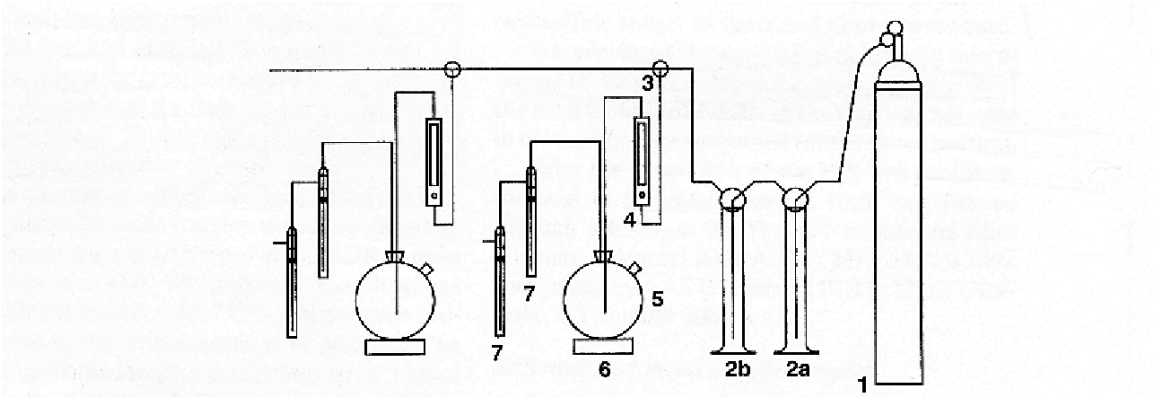

Fig. 1. Apparatus for AVS determination: (1) N_2 cylinder; (2) gas-washing bottles—(a) oxygen scrubbing solution or an oxygen trap may be used in replacement of this gas-washing bottle, (b) deionized water; (3) three-way stopcock; (4) flow controller; (5) reaction flask; (6) magnetic stirrer; (7) impingers with nonfritted outlets.

V. References

Allen H.E., Fu G., Deng B. Analysis of acid-volatile sulfide (AVS) and simultaneously extracted metals (SEM) for the estimation of potential toxicity in aquatic sediments. *Environ. Toxicol. Chem*. **1993,** 12:1–13.

Berry, W.J., W.S. Boothman, J.R. Serbst, and P.A. Edwards. Predicting the toxicity of chromium in sediments. *Environ. Toxicol. Chem.* **2004,** *23*, 2981-2992.

Boothman, W.S. and A. Helmstetter. Determination of acid-volatile sulfide and simultaneously-extracted metals in sediments using sulfide-specific detection. AVSSEM Standard Operating Protocol v. 2.0, **1993**, US EPA Environmental Research Laboratory, Narragansett, RI Internal Document.

Cornwell, J.C. and J.W. Morse. The characterization of iron sulfide minerals in marine sediments. *Marine Chemistry*. **1987**, 22, 193-206

Soil and Waste pH – EPA Method 9045 D

I. Materials

- pH meter
- glass electrode
- reference electrode
- 50 mL beaker
- thermometer or temperature sensor
- analytical balance

II. Reagents

- NIST primary buffer solutions

III. Procedure

1. Calibrate the pH meter using pH 2, 4, 7, and 10 NIST standard buffer solutions
2. Add 20 g of soil/sediment to 50 mL beaker and add 20 mL reagent water. Cover and stir for 5 min.
3. Let suspension stand for 1 hour to settle or centrifuge
4. Immerse glass electrode into clear supernatant solution
5. If sample temperature differs by more than 2 ^{o}C from buffer solution, apply temperature correction
6. Report temperature at which pH of soil in water was measured.

IV. QA/QC

1. Duplicate samples
2. Electrode thoroughly rinsed between samples

V. References

U.S. Environmental Protection Agency. EPA Method 9045D- Soil and Waste pH. SW-846. Revised November, 2004.

Gravimetric Method for Determining Sediment Water Content – Adapted from Tan, 1996

I. Materials

- Oven at 100 – 100 ^{o}C
- Analytical Balance
- Flasks with ground stoppered lid
- Dessicator with magnesium perchlorate or calcium sulfate

II. Procedure

1. Weigh flask and stopper
2. Add 5-10 g of wet sample, stopper the flask, and weigh
3. Remove lid from flask and place in oven to dry for 24 hours at 105 ^{o}C.
4. Remove sample from oven and place in dessicator to cool
5. Place stopper on flask and record weight
6. wet mass % H_2O = wet soil mass – dry soil mass /wet soil mass

III. QA/QC

1. Return sample and dry for several hours, cool in dessicator and determine weight. Repeat until no difference in consecutive measurements.
2. Duplicate samples

IV. References

Tan, K.H. Soil Sampling, Preparation, and Analysis. **1996.** Marcel Dekker, NY, 480 p.

Appendix C: Organics Analysis Results

MARTEL

Sensible Scientific Solutions

Certificate of Analysis

Tuesday, June 5, 2007

Prepared expressly for:

Johns Hopkins Enterprise
313 Ames Hall
3400 N. Charles St.
Baltimore, MD 21218

Attention: Dr. Ed Bower

Report for Lab No: 44929.
P.O. Number: 2000104074
Project Identification: Sediment Study - JHU / Honeywell - 4/20/07

MARTEL NO. 44929 000001 | CLIENT SAMPLE IDENTIFICATION: Wye River Control | Sample Date/Time: 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Base/Neutral/Acid Extractables			EPA 8270C		05/09/2007 19:47 CJD
					/ /
Acenaphthene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Acenaphthylene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Benzo[a]anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Benzo[b]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Benzo[k]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Benzo[ghi]perylene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Benzo[a]pyrene	<300	ug/kg	EPA 8270	300	05/09/2007 19:47 CJD
Bis-(2-chloroethoxy)methane	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Bis-(2-chloroethyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Bis(2-chloroisopropyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
4-Bromophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Benzyl butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Carbazole	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
4-Chloroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
4-Chloro-3-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
2-Chloronaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
2-Chlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
4-Chlorophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Chrysene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Dibenz[a,h]anthracene	<300	ug/kg	EPA 8270	300	05/09/2007 19:47 CJD
Dibenzofuran	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Di-n-butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
1,2-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 19:47 CJD
1,3-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 19:47 CJD
1,4-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 19:47 CJD
3,3'-Dichlorobenzidine	<300	ug/kg	EPA 8270	300	05/09/2007 19:47 CJD
2,4-Dichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Diethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
2,4-Dimethylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Dimethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD

MARTEL NO. 44929 000001 | CLIENT SAMPLE IDENTIFICATION: Wye River Control | Sample Date/Time: 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
4,6-Dinitro-2-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
2,4-Dinitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
2,4-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
2,6-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Di-n-octyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Bis-(2-ethylhexyl)-phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Fluorene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Hexachlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Hexachlorocyclopentadiene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Hexachloroethane	<300	ug/kg	EPA 8270	300	05/09/2007 19:47 CJD
Indeno-(1,2,3-cd)-pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Isophorone	<300	ug/kg	EPA 8270	300	05/09/2007 19:47 CJD
2-Methylnaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
2-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
4-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Naphthalene	<300	ug/kg	EPA 8270	300	05/09/2007 19:47 CJD
2-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
3-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
4-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Nitrobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
2-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
4-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
N-Nitrosodiphenylamine	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
N-Nitroso-di-N-propylamine	<300	ug/kg	EPA 8270	300	05/09/2007 19:47 CJD
Pentachlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Phenanthrene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Phenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
1,2,4-Trichlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
2,4,5-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
2,4,6-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
N-Nitrosodimethylamine	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
Hexachlorobutadiene	<500	ug/kg	EPA 8270	500	05/09/2007 19:47 CJD
					/ /
Surrogate Spike					/ /
					/ /
2,4,6-Tribromophenol	86	%	EPA 8270		05/09/2007 19:47 CJD
2-Fluorobiphenyl	75	%	EPA 8270		05/09/2007 19:47 CJD
2-Fluorophenol	62	%	EPA 8270		05/09/2007 19:47 CJD
Nitrobenzene-d5	62	%	EPA 8270		05/09/2007 19:47 CJD
Phenol-d6	65	%	EPA 8270		05/09/2007 19:47 CJD
Terphenyl-d14	80	%	EPA 8270		05/09/2007 19:47 CJD
					/ /
Solids (Total)	55	%	EPA 160.3		04/26/2007 13:10 GS

MARTEL NO. 44929 000001 | CLIENT SAMPLE IDENTIFICATION: Wye River Control | Sample Date/Time: 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Diesel Range Organics by GC/FID	18	mg/kg	EPA 8015B		05/08/2007 15:32 AK
Gasoline Range Organics by GC-FID	<0.25	mg/kg	EPA 8015B	0.25	05/15/2007 18:11 MW
PCB's as Aroclors by Capillary GC			EPA 8082		05/16/2007 20:39 AK
					/ /
PCB-1016	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 20:39 AK
PCB-1221	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 20:39 AK
PCB-1232	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 20:39 AK
PCB-1242	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 20:39 AK
PCB-1248	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 20:39 AK
PCB-1254	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 20:39 AK
PCB-1260	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 20:39 AK
PCB-1262	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 20:39 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	60	%	EPA 8082		05/16/2007 20:39 AK
Decachlorobiphenyl	59	%	EPA 8082		05/16/2007 20:39 AK
					/ /
pH	7.59		EPA 150.1		04/26/2007 13:50 SK
Organochlorine Pesticides and PCB's			EPA 8081A		05/31/2007 20:20 AK
					/ /
Aldrin	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
a-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
b-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
g-BHC (Lindane)	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
d-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Chlordane	<50	ug/kg	EPA 8081	50	05/31/2007 20:20 AK
4,4'-DDD	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
4,4'-DDE	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
4,4'-DDT	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Dieldrin	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Endosulfan I	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Endosulfan II	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Endosulfan Sulfate	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Endrin	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Endrin Aldehyde	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Heptachlor	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Heptachlor Epoxide	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Methoxychlor	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Endrin Ketone	<5	ug/kg	EPA 8081	5	05/31/2007 20:20 AK
Toxaphene	<50	ug/kg	EPA 8081	50	05/31/2007 20:20 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	49	%	EPA 8081		05/31/2007 20:20 AK

MARTEL NO.		CLIENT SAMPLE IDENTIFICATION	Sample Date/Time
44929	000001	Wye River Control	04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Decachlorobiphenyl	66	%	EPA 8081		05/31/2007 20:20 AK
					/ /

MARTEL NO.		CLIENT SAMPLE IDENTIFICATION	Sample Date/Time
44929	000002	BSM 33	04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Base/Neutral/Acid Extractables			EPA 8270C		05/09/2007 20:58 CJD
					/ /
Acenaphthene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Acenaphthylene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Benzo[a]anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Benzo[b]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Benzo[k]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Benzo[ghi]perylene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Benzo[a]pyrene	<300	ug/kg	EPA 8270	300	05/09/2007 20:58 CJD
Bis-(2-chloroethoxy)methane	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Bis-(2-chloroethyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Bis(2-chloroisopropyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
4-Bromophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Benzyl butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Carbazole	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
4-Chloroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
4-Chloro-3-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
2-Chloronaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
2-Chlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
4-Chlorophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Chrysene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Dibenz[a,h]anthracene	<300	ug/kg	EPA 8270	300	05/09/2007 20:58 CJD
Dibenzofuran	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Di-n-butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
1,2-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 20:58 CJD
1,3-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 20:58 CJD
1,4-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 20:58 CJD
3,3'-Dichlorobenzidine	<300	ug/kg	EPA 8270	300	05/09/2007 20:58 CJD
2,4-Dichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Diethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
2,4-Dimethylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Dimethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
4,6-Dinitro-2-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
2,4-Dinitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
2,4-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
2,6-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Di-n-octyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD

MARTEL NO. 44929 000002 BSM 33

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Bis-(2-ethylhexyl)-phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Fluorene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Hexachlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Hexachlorocyclopentadiene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Hexachloroethane	<300	ug/kg	EPA 8270	300	05/09/2007 20:58 CJD
Indeno-(1,2,3-cd)-pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Isophorone	<300	ug/kg	EPA 8270	300	05/09/2007 20:58 CJD
2-Methylnaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
2-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
4-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Naphthalene	<300	ug/kg	EPA 8270	300	05/09/2007 20:58 CJD
2-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
3-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
4-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Nitrobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
2-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
4-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
N-Nitrosodiphenylamine	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
N-Nitroso-di-N-propylamine	<300	ug/kg	EPA 8270	300	05/09/2007 20:58 CJD
Pentachlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Phenanthrene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Phenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
1,2,4-Trichlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
2,4,5-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
2,4,6-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
N-Nitrosodimethylamine	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
Hexachlorobutadiene	<500	ug/kg	EPA 8270	500	05/09/2007 20:58 CJD
					/ /
Surrogate Spike					/ /
					/ /
2,4,6-Tribromophenol	79	%	EPA 8270		05/09/2007 20:58 CJD
2-Fluorobiphenyl	62	%	EPA 8270		05/09/2007 20:58 CJD
2-Fluorophenol	53	%	EPA 8270		05/09/2007 20:58 CJD
Nitrobenzene-d5	51	%	EPA 8270		05/09/2007 20:58 CJD
Phenol-d6	54	%	EPA 8270		05/09/2007 20:58 CJD
Terphenyl-d14	79	%	EPA 8270		05/09/2007 20:58 CJD
					/ /
Solids (Total)	14	%	EPA 160.3		04/26/2007 13:10 GS
Diesel Range Organics by GC/FID	170	mg/kg	EPA 8015B		05/08/2007 16:05 AK
Gasoline Range Organics by GC-FID	<0.25	mg/kg	EPA 8015B	0.25	05/09/2007 19:57 MW
PCB's as Aroclors by Capillary GC			EPA 8082		05/16/2007 21:13 AK
					/ /
PCB-1016	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:13 AK

MARTEL NO. 44929 000002 BSM 33

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
PCB-1221	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:13 AK
PCB-1232	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:13 AK
PCB-1242	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:13 AK
PCB-1248	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:13 AK
PCB-1254	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:13 AK
PCB-1260	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:13 AK
PCB-1262	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:13 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	77	%	EPA 8082		05/16/2007 21:13 AK
Decachlorobiphenyl	64	%	EPA 8082		05/16/2007 21:13 AK
					/ /
pH	8.54		EPA 150.1		04/26/2007 13:50 SK
Organochlorine Pesticides and PCB's			EPA 8081A		05/31/2007 20:57 AK
					/ /
Aldrin	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
a-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
b-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
g-BHC (Lindane)	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
d-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Chlordane	<50	ug/kg	EPA 8081	50	05/31/2007 20:57 AK
4,4'-DDD	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
4,4'-DDE	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
4,4'-DDT	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Dieldrin	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Endosulfan I	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Endosulfan II	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Endosulfan Sulfate	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Endrin	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Endrin Aldehyde	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Heptachlor	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Heptachlor Epoxide	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Methoxychlor	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Endrin Ketone	<5	ug/kg	EPA 8081	5	05/31/2007 20:57 AK
Toxaphene	<50	ug/kg	EPA 8081	50	05/31/2007 20:57 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	86	%	EPA 8081		05/31/2007 20:57 AK
Decachlorobiphenyl	80	%	EPA 8081		05/31/2007 20:57 AK
					/ /

MARTEL NO. 44929 000003 BSM 45

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Base/Neutral/Acid Extractables			EPA 8270C		05/09/2007 20:22 CJD
					/ /
Acenaphthene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Acenaphthylene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Benzo[a]anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Benzo[b]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Benzo[k]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Benzo[ghi]perylene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Benzo[a]pyrene	<300	ug/kg	EPA 8270	300	05/09/2007 20:22 CJD
Bis-(2-chloroethoxy)methane	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Bis-(2-chloroethyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Bis(2-chloroisopropyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
4-Bromophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Benzyl butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Carbazole	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
4-Chloroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
4-Chloro-3-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
2-Chloronaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
2-Chlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
4-Chlorophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Chrysene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Dibenz[a,h]anthracene	<300	ug/kg	EPA 8270	300	05/09/2007 20:22 CJD
Dibenzofuran	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Di-n-butyl phthalate	1300	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
1,2-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 20:22 CJD
1,3-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 20:22 CJD
1,4-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 20:22 CJD
3,3'-Dichlorobenzidine	<300	ug/kg	EPA 8270	300	05/09/2007 20:22 CJD
2,4-Dichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Diethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
2,4-Dimethylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Dimethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
4,6-Dinitro-2-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
2,4-Dinitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
2,4-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
2,6-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Di-n-octyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Bis-(2-ethylhexyl)-phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Fluorene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Hexachlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Hexachlorocyclopentadiene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Hexachloroethane	<300	ug/kg	EPA 8270	300	05/09/2007 20:22 CJD
Indeno-(1,2,3-cd)-pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD

MARTEL NO. 44929 000003 BSM 45

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Isophorone	<300	ug/kg	EPA 8270	300	05/09/2007 20:22 CJD
2-Methylnaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
2-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
4-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Naphthalene	<300	ug/kg	EPA 8270	300	05/09/2007 20:22 CJD
2-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
3-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
4-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Nitrobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
2-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
4-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
N-Nitrosodiphenylamine	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
N-Nitroso-di-N-propylamine	<300	ug/kg	EPA 8270	300	05/09/2007 20:22 CJD
Pentachlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Phenanthrene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Phenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
1,2,4-Trichlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
2,4,5-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
2,4,6-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
N-Nitrosodimethylamine	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
Hexachlorobutadiene	<500	ug/kg	EPA 8270	500	05/09/2007 20:22 CJD
					/ /
Surrogate Spike					/ /
					/ /
2,4,6-Tribromophenol	90	%	EPA 8270		05/09/2007 20:22 CJD
2-Fluorobiphenyl	75	%	EPA 8270		05/09/2007 20:22 CJD
2-Fluorophenol	65	%	EPA 8270		05/09/2007 20:22 CJD
Nitrobenzene-d5	61	%	EPA 8270		05/09/2007 20:22 CJD
Phenol-d6	66	%	EPA 8270		05/09/2007 20:22 CJD
Terphenyl-d14	76	%	EPA 8270		05/09/2007 20:22 CJD
					/ /
Solids (Total)	27	%	EPA 160.3		04/26/2007 13:10 GS
Diesel Range Organics by GC/FID	99	mg/kg	EPA 8015B		05/08/2007 16:39 AK
Gasoline Range Organics by GC-FID	<0.25	mg/kg	EPA 8015B	0.25	05/09/2007 20:35 MW
PCB's as Aroclors by Capillary GC			EPA 8082		05/16/2007 21:47 AK
					/ /
PCB-1016	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:47 AK
PCB-1221	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:47 AK
PCB-1232	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:47 AK
PCB-1242	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:47 AK
PCB-1248	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:47 AK
PCB-1254	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:47 AK
PCB-1260	0.1	mg/kg	EPA 8082	0.05	05/16/2007 21:47 AK
PCB-1262	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 21:47 AK

MARTEL NO. 44929 000003 | CLIENT SAMPLE IDENTIFICATION | Sample Date/Time / /

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	67	%	EPA 8082		05/16/2007 21:47 AK
Decachlorobiphenyl	70	%	EPA 8082		05/16/2007 21:47 AK
					/ /
pH	7.14		EPA 150.1		04/26/2007 13:50 SK
Organochlorine Pesticides and PCB's			EPA 8081A		05/31/2007 21:33 AK
					/ /
Aldrin	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
a-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
b-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
g-BHC (Lindane)	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
d-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Chlordane	<50	ug/kg	EPA 8081	50	05/31/2007 21:33 AK
4,4'-DDD	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
4,4'-DDE	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
4,4'-DDT	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Dieldrin	9.0	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Endosulfan I	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Endosulfan II	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Endosulfan Sulfate	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Endrin	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Endrin Aldehyde	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Heptachlor	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Heptachlor Epoxide	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Methoxychlor	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Endrin Ketone	<5	ug/kg	EPA 8081	5	05/31/2007 21:33 AK
Toxaphene	<50	ug/kg	EPA 8081	50	05/31/2007 21:33 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	58	%	EPA 8081		05/31/2007 21:33 AK
Decachlorobiphenyl	79	%	EPA 8081		05/31/2007 21:33 AK
					/ /

MARTEL NO. 44929 000004 BSM 54 | CLIENT SAMPLE IDENTIFICATION | Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Base/Neutral/Acid Extractables			EPA 8270C		05/09/2007 21:33 CJD
					/ /
Acenaphthene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Acenaphthylene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD

MARTEL NO. 44929 000004 BSM 54

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Benzo[a]anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Benzo[b]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Benzo[k]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Benzo[ghi]perylene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Benzo[a]pyrene	<300	ug/kg	EPA 8270	300	05/09/2007 21:33 CJD
Bis-(2-chloroethoxy)methane	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Bis-(2-chloroethyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Bis(2-chloroisopropyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
4-Bromophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Benzyl butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Carbazole	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
4-Chloroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
4-Chloro-3-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
2-Chloronaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
2-Chlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
4-Chlorophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Chrysene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Dibenz[a,h]anthracene	<300	ug/kg	EPA 8270	300	05/09/2007 21:33 CJD
Dibenzofuran	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Di-n-butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
1,2-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 21:33 CJD
1,3-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 21:33 CJD
1,4-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 21:33 CJD
3,3'-Dichlorobenzidine	<300	ug/kg	EPA 8270	300	05/09/2007 21:33 CJD
2,4-Dichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Diethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
2,4-Dimethylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Dimethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
4,6-Dinitro-2-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
2,4-Dinitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
2,4-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
2,6-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Di-n-octyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Bis-(2-ethylhexyl)-phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Fluorene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Hexachlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Hexachlorocyclopentadiene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Hexachloroethane	<300	ug/kg	EPA 8270	300	05/09/2007 21:33 CJD
Indeno-(1,2,3-cd)-pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Isophorone	<300	ug/kg	EPA 8270	300	05/09/2007 21:33 CJD
2-Methylnaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
2-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
4-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Naphthalene	<300	ug/kg	EPA 8270	300	05/09/2007 21:33 CJD

MARTEL NO. 44929 000004 BSM 54

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
2-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
3-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
4-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Nitrobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
2-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
4-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
N-Nitrosodiphenylamine	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
N-Nitroso-di-N-propylamine	<300	ug/kg	EPA 8270	300	05/09/2007 21:33 CJD
Pentachlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Phenanthrene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Phenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
1,2,4-Trichlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
2,4,5-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
2,4,6-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
N-Nitrosodimethylamine	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
Hexachlorobutadiene	<500	ug/kg	EPA 8270	500	05/09/2007 21:33 CJD
					/ /
Surrogate Spike					/ /
					/ /
2,4,6-Tribromophenol	75	%	EPA 8270		05/09/2007 21:33 CJD
2-Fluorobiphenyl	59	%	EPA 8270		05/09/2007 21:33 CJD
2-Fluorophenol	48	%	EPA 8270		05/09/2007 21:33 CJD
Nitrobenzene-d5	46	%	EPA 8270		05/09/2007 21:33 CJD
Phenol-d6	50	%	EPA 8270		05/09/2007 21:33 CJD
Terphenyl-d14	64	%	EPA 8270		05/09/2007 21:33 CJD
					/ /
Solids (Total)	33	%	EPA 160.3		04/26/2007 13:10 GS
Diesel Range Organics by GC/FID	71	mg/kg	EPA 8015B		05/08/2007 17:13 AK
Gasoline Range Organics by GC-FID	<0.25	mg/kg	EPA 8015B	0.25	05/09/2007 21:13 MW
PCB's as Aroclors by Capillary GC			EPA 8082		05/16/2007 22:21 AK
					/ /
PCB-1016	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:21 AK
PCB-1221	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:21 AK
PCB-1232	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:21 AK
PCB-1242	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:21 AK
PCB-1248	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:21 AK
PCB-1254	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:21 AK
PCB-1260	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:21 AK
PCB-1262	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:21 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	72	%	EPA 8082		05/16/2007 22:21 AK
Decachlorobiphenyl	63	%	EPA 8082		05/16/2007 22:21 AK

MARTEL NO. 44929 000004 | CLIENT SAMPLE IDENTIFICATION | Sample Date/Time / /

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
					/ /
pH	7.91		EPA 150.1		04/26/2007 13:50 SK
Organochlorine Pesticides and PCB's			EPA 8081A		05/31/2007 22:09 AK
					/ /
Aldrin	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
a-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
b-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
g-BHC (Lindane)	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
d-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Chlordane	<50	ug/kg	EPA 8081	50	05/31/2007 22:09 AK
4,4'-DDD	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
4,4'-DDE	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
4,4'-DDT	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Dieldrin	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Endosulfan I	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Endosulfan II	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Endosulfan Sulfate	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Endrin	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Endrin Aldehyde	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Heptachlor	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Heptachlor Epoxide	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Methoxychlor	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Endrin Ketone	<5	ug/kg	EPA 8081	5	05/31/2007 22:09 AK
Toxaphene	<50	ug/kg	EPA 8081	50	05/31/2007 22:09 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	66	%	EPA 8081		05/31/2007 22:09 AK
Decachlorobiphenyl	75	%	EPA 8081		05/31/2007 22:09 AK
					/ /

MARTEL NO. 44929 000005 BSM 68 | CLIENT SAMPLE IDENTIFICATION | Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Base/Neutral/Acid Extractables			EPA 8270C		05/09/2007 22:09 CJD
					/ /
Acenaphthene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Acenaphthylene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Benzo[a]anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Benzo[b]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Benzo[k]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Benzo[ghi]perylene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Benzo[a]pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD

MARTEL NO. 44929 000005 BSM 68

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Bis-(2-chloroethoxy)methane	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Bis-(2-chloroethyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Bis(2-chloroisopropyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
4-Bromophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Benzyl butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Carbazole	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
4-Chloroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
4-Chloro-3-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
2-Chloronaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
2-Chlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
4-Chlorophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Chrysene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Dibenz[a,h]anthracene	<300	ug/kg	EPA 8270	300	05/09/2007 22:09 CJD
Dibenzofuran	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Di-n-butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
1,2-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 22:09 CJD
1,3-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 22:09 CJD
1,4-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 22:09 CJD
3,3'-Dichlorobenzidine	<300	ug/kg	EPA 8270	300	05/09/2007 22:09 CJD
2,4-Dichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Diethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
2,4-Dimethylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Dimethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
4,6-Dinitro-2-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
2,4-Dinitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
2,4-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
2,6-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Di-n-octyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Bis-(2-ethylhexyl)-phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Fluorene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Hexachlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Hexachlorocyclopentadiene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Hexachloroethane	<300	ug/kg	EPA 8270	300	05/09/2007 22:09 CJD
Indeno-(1,2,3-cd)-pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Isophorone	<300	ug/kg	EPA 8270	300	05/09/2007 22:09 CJD
2-Methylnaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
2-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
4-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Naphthalene	<300	ug/kg	EPA 8270	300	05/09/2007 22:09 CJD
2-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
3-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
4-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Nitrobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
2-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD

MARTEL NO. 44929 000005 BSM 68

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
4-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
N-Nitrosodiphenylamine	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
N-Nitroso-di-N-propylamine	<300	ug/kg	EPA 8270	300	05/09/2007 22:09 CJD
Pentachlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Phenanthrene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Phenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
1,2,4-Trichlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
2,4,5-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
2,4,6-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
N-Nitrosodimethylamine	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
Hexachlorobutadiene	<500	ug/kg	EPA 8270	500	05/09/2007 22:09 CJD
					/ /
Surrogate Spike					/ /
					/ /
2,4,6-Tribromophenol	86	%	EPA 8270		05/09/2007 22:09 CJD
2-Fluorobiphenyl	66	%	EPA 8270		05/09/2007 22:09 CJD
2-Fluorophenol	55	%	EPA 8270		05/09/2007 22:09 CJD
Nitrobenzene-d5	53	%	EPA 8270		05/09/2007 22:09 CJD
Phenol-d6	56	%	EPA 8270		05/09/2007 22:09 CJD
Terphenyl-d14	80	%	EPA 8270		05/09/2007 22:09 CJD
					/ /
Solids (Total)	26	%	EPA 160.3		04/26/2007 13:10 GS
Diesel Range Organics by GC/FID	49	mg/kg	EPA 8015B		05/08/2007 17:46 AK
Gasoline Range Organics by GC-FID	<0.25	mg/kg	EPA 8015B	0.25	05/09/2007 21:51 MW
PCB's as Aroclors by Capillary GC			EPA 8082		05/16/2007 22:55 AK
					/ /
PCB-1016	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:55 AK
PCB-1221	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:55 AK
PCB-1232	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:55 AK
PCB-1242	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:55 AK
PCB-1248	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:55 AK
PCB-1254	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:55 AK
PCB-1260	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:55 AK
PCB-1262	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 22:55 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	59	%	EPA 8082		05/16/2007 22:55 AK
Decachlorobiphenyl	58	%	EPA 8082		05/16/2007 22:55 AK
					/ /
pH	7.99		EPA 150.1		04/26/2007 13:50 SK
Organochlorine Pesticides and PCB's			EPA 8081A		05/31/2007 22:45 AK
					/ /
Aldrin	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK

MARTEL NO. 44929 000005 BSM 68 | CLIENT SAMPLE IDENTIFICATION | Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
a-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
b-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
g-BHC (Lindane)	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
d-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Chlordane	<50	ug/kg	EPA 8081	50	05/31/2007 22:45 AK
4,4'-DDD	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
4,4'-DDE	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
4,4'-DDT	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Dieldrin	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Endosulfan I	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Endosulfan II	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Endosulfan Sulfate	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Endrin	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Endrin Aldehyde	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Heptachlor	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Heptachlor Epoxide	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Methoxychlor	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Endrin Ketone	<5	ug/kg	EPA 8081	5	05/31/2007 22:45 AK
Toxaphene	<50	ug/kg	EPA 8081	50	05/31/2007 22:45 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	55	%	EPA 8081		05/31/2007 22:45 AK
Decachlorobiphenyl	70	%	EPA 8081		05/31/2007 22:45 AK
					/ /

MARTEL NO. 44929 000006 BSM 38 | CLIENT SAMPLE IDENTIFICATION | Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Base/Neutral/Acid Extractables			EPA 8270C		05/09/2007 22:45 CJD
					/ /
Acenaphthene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Acenaphthylene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Benzo[a]anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Benzo[b]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Benzo[k]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Benzo[ghi]perylene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Benzo[a]pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Bis-(2-chloroethoxy)methane	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Bis-(2-chloroethyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Bis(2-chloroisopropyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
4-Bromophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Benzyl butyl phthalate	1200	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD

MARTEL NO. 44929 000006 BSM 38

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Carbazole	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
4-Chloroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
4-Chloro-3-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
2-Chloronaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
2-Chlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
4-Chlorophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Chrysene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Dibenz[a,h]anthracene	<300	ug/kg	EPA 8270	300	05/09/2007 22:45 CJD
Dibenzofuran	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Di-n-butyl phthalate	7600	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
1,2-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 22:45 CJD
1,3-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 22:45 CJD
1,4-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 22:45 CJD
3,3'-Dichlorobenzidine	<300	ug/kg	EPA 8270	300	05/09/2007 22:45 CJD
2,4-Dichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Diethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
2,4-Dimethylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Dimethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
4,6-Dinitro-2-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
2,4-Dinitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
2,4-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
2,6-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Di-n-octyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Bis-(2-ethylhexyl)-phthalate	13000	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Fluoranthene	580	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Fluorene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Hexachlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Hexachlorocyclopentadiene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Hexachloroethane	<300	ug/kg	EPA 8270	300	05/09/2007 22:45 CJD
Indeno-(1,2,3-cd)-pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Isophorone	<300	ug/kg	EPA 8270	300	05/09/2007 22:45 CJD
2-Methylnaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
2-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
4-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Naphthalene	<300	ug/kg	EPA 8270	300	05/09/2007 22:45 CJD
2-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
3-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
4-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Nitrobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
2-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
4-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
N-Nitrosodiphenylamine	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
N-Nitroso-di-N-propylamine	<300	ug/kg	EPA 8270	300	05/09/2007 22:45 CJD
Pentachlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Phenanthrene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD

MARTEL NO. 44929 000006 BSM 38

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Phenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
1,2,4-Trichlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
2,4,5-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
2,4,6-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
N-Nitrosodimethylamine	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
Hexachlorobutadiene	<500	ug/kg	EPA 8270	500	05/09/2007 22:45 CJD
					/ /
Surrogate Spike					/ /
					/ /
2,4,6-Tribromophenol	92	%	EPA 8270		05/09/2007 22:45 CJD
2-Fluorobiphenyl	65	%	EPA 8270		05/09/2007 22:45 CJD
2-Fluorophenol	60	%	EPA 8270		05/09/2007 22:45 CJD
Nitrobenzene-d5	54	%	EPA 8270		05/09/2007 22:45 CJD
Phenol-d6	62	%	EPA 8270		05/09/2007 22:45 CJD
Terphenyl-d14	94	%	EPA 8270		05/09/2007 22:45 CJD
					/ /
Solids (Total)	34	%	EPA 160.3		04/26/2007 13:10 GS
Diesel Range Organics by GC/FID	290	mg/kg	EPA 8015B		05/09/2007 13:28 AK
Gasoline Range Organics by GC-FID	2.4	mg/kg	EPA 8015B	0.25	05/09/2007 22:29 MW
PCB's as Aroclors by Capillary GC			EPA 8082		05/16/2007 23:29 AK
					/ /
PCB-1016	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 23:29 AK
PCB-1221	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 23:29 AK
PCB-1232	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 23:29 AK
PCB-1242	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 23:29 AK
PCB-1248	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 23:29 AK
PCB-1254	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 23:29 AK
PCB-1260	0.12	mg/kg	EPA 8082	0.05	05/16/2007 23:29 AK
PCB-1262	<0.05	mg/kg	EPA 8082	0.05	05/16/2007 23:29 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	126	%	EPA 8082		05/16/2007 23:29 AK
Decachlorobiphenyl	67	%	EPA 8082		05/16/2007 23:29 AK
					/ /
pH	7.44		EPA 150.1		04/26/2007 13:50 SK
Organochlorine Pesticides and PCB's			EPA 8081A		05/31/2007 23:21 AK
					/ /
Aldrin	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
a-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
b-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
g-BHC (Lindane)	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
d-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Chlordane	<50	ug/kg	EPA 8081	50	05/31/2007 23:21 AK

MARTEL NO. 44929 000006 BSM 38

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
4,4'-DDD	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
4,4'-DDE	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
4,4'-DDT	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Dieldrin	5.3	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Endosulfan I	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Endosulfan II	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Endosulfan Sulfate	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Endrin	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Endrin Aldehyde	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Heptachlor	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Heptachlor Epoxide	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Methoxychlor	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Endrin Ketone	<5	ug/kg	EPA 8081	5	05/31/2007 23:21 AK
Toxaphene	<50	ug/kg	EPA 8081	50	05/31/2007 23:21 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	58	%	EPA 8081		05/31/2007 23:21 AK
Decachlorobiphenyl	70	%	EPA 8081		05/31/2007 23:21 AK
					/ /

MARTEL NO. 44929 000007 BSM 2 Reference

CLIENT SAMPLE IDENTIFICATION

Sample Date/Time 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Base/Neutral/Acid Extractables			EPA 8270C		05/09/2007 17:59 CJD
					/ /
Acenaphthene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Acenaphthylene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Benzo[a]anthracene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Benzo[b]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Benzo[k]fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Benzo[ghi]perylene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Benzo[a]pyrene	<300	ug/kg	EPA 8270	300	05/09/2007 17:59 CJD
Bis-(2-chloroethoxy)methane	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Bis-(2-chloroethyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Bis(2-chloroisopropyl)ether	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
4-Bromophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Benzyl butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Carbazole	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
4-Chloroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
4-Chloro-3-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
2-Chloronaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
2-Chlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD

MARTEL NO. 44929 000007 | CLIENT SAMPLE IDENTIFICATION: BSM 2 Reference | Sample Date/Time: 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
4-Chlorophenyl phenyl ether	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Chrysene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Dibenz[a,h]anthracene	<300	ug/kg	EPA 8270	300	05/09/2007 17:59 CJD
Dibenzofuran	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Di-n-butyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
1,2-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 17:59 CJD
1,3-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 17:59 CJD
1,4-Dichlorobenzene	<300	ug/kg	EPA 8270	300	05/09/2007 17:59 CJD
3,3'-Dichlorobenzidine	<300	ug/kg	EPA 8270	300	05/09/2007 17:59 CJD
2,4-Dichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Diethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
2,4-Dimethylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Dimethyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
4,6-Dinitro-2-methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
2,4-Dinitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
2,4-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
2,6-Dinitrotoluene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Di-n-octyl phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Bis-(2-ethylhexyl)-phthalate	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Fluoranthene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Fluorene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Hexachlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Hexachlorocyclopentadiene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Hexachloroethane	<300	ug/kg	EPA 8270	300	05/09/2007 17:59 CJD
Indeno-(1,2,3-cd)-pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Isophorone	<300	ug/kg	EPA 8270	300	05/09/2007 17:59 CJD
2-Methylnaphthalene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
2-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
4-Methylphenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Naphthalene	<300	ug/kg	EPA 8270	300	05/09/2007 17:59 CJD
2-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
3-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
4-Nitroaniline	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Nitrobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
2-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
4-Nitrophenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
N-Nitrosodiphenylamine	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
N-Nitroso-di-N-propylamine	<300	ug/kg	EPA 8270	300	05/09/2007 17:59 CJD
Pentachlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Phenanthrene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Phenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Pyrene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
1,2,4-Trichlorobenzene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
2,4,5-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
2,4,6-Trichlorophenol	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD

MARTEL NO. 44929 000007 | CLIENT SAMPLE IDENTIFICATION: BSM 2 Reference | Sample Date/Time: 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
N-Nitrosodimethylamine	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
Hexachlorobutadiene	<500	ug/kg	EPA 8270	500	05/09/2007 17:59 CJD
					/ /
Surrogate Spike					/ /
					/ /
2,4,6-Tribromophenol	74	%	EPA 8270		05/09/2007 17:59 CJD
2-Fluorobiphenyl	63	%	EPA 8270		05/09/2007 17:59 CJD
2-Fluorophenol	62	%	EPA 8270		05/09/2007 17:59 CJD
Nitrobenzene-d5	57	%	EPA 8270		05/09/2007 17:59 CJD
Phenol-d6	66	%	EPA 8270		05/09/2007 17:59 CJD
Terphenyl-d14	77	%	EPA 8270		05/09/2007 17:59 CJD
					/ /
Solids (Total)	34	%	EPA 160.3		04/26/2007 13:10 GS
Diesel Range Organics by GC/FID	53	mg/kg	EPA 8015B		05/08/2007 18:54 AK
Gasoline Range Organics by GC-FID	<0.25	mg/kg	EPA 8015B	0.25	05/09/2007 23:07 MW
PCB's as Aroclors by Capillary GC			EPA 8082		05/17/2007 00:03 AK
					/ /
PCB-1016	<0.05	mg/kg	EPA 8082	0.05	05/17/2007 00:03 AK
PCB-1221	<0.05	mg/kg	EPA 8082	0.05	05/17/2007 00:03 AK
PCB-1232	<0.05	mg/kg	EPA 8082	0.05	05/17/2007 00:03 AK
PCB-1242	<0.05	mg/kg	EPA 8082	0.05	05/17/2007 00:03 AK
PCB-1248	<0.05	mg/kg	EPA 8082	0.05	05/17/2007 00:03 AK
PCB-1254	<0.05	mg/kg	EPA 8082	0.05	05/17/2007 00:03 AK
PCB-1260	<0.05	mg/kg	EPA 8082	0.05	05/17/2007 00:03 AK
PCB-1262	<0.05	mg/kg	EPA 8082	0.05	05/17/2007 00:03 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	147	%	EPA 8082		05/17/2007 00:03 AK
Decachlorobiphenyl	146	%	EPA 8082		05/17/2007 00:03 AK
					/ /
pH	7.53		EPA 150.1		04/26/2007 13:50 SK
Organochlorine Pesticides and PCB's			EPA 8081A		05/31/2007 23:57 AK
					/ /
Aldrin	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
a-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
b-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
g-BHC (Lindane)	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
d-BHC	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Chlordane	<50	ug/kg	EPA 8081	50	05/31/2007 23:57 AK
4,4'-DDD	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
4,4'-DDE	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
4,4'-DDT	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Dieldrin	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Endosulfan I	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK

MARTEL NO. 44929 000007 | CLIENT SAMPLE IDENTIFICATION: BSM 2 Reference | Sample Date/Time: 04/20/2007 12:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Endosulfan II	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Endosulfan Sulfate	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Endrin	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Endrin Aldehyde	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Heptachlor	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Heptachlor Epoxide	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Methoxychlor	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Endrin Ketone	<5	ug/kg	EPA 8081	5	05/31/2007 23:57 AK
Toxaphene	<50	ug/kg	EPA 8081	50	05/31/2007 23:57 AK
					/ /
Surrogate Spike					/ /
					/ /
2,4,5,6-Tetrachlorometaxylene	54	%	EPA 8081		05/31/2007 23:57 AK
Decachlorobiphenyl	58	%	EPA 8081		05/31/2007 23:57 AK
					/ /

MARTEL NO. 44929 0008TB | CLIENT SAMPLE IDENTIFICATION: Trip Blank | Sample Date/Time: 04/20/2007 00:00

Compound	Test Value	Test Unit	Method	Detection Limit	Analysis Date/Time/Initial
Gasoline Range Organics by GC-FID	<0.25	mg/kg	EPA 8015B	0.25	05/04/2007 18:17 MW

Martel Laboratories *JDS* **Inc.**

1025 Cromwell Bridge Road - Baltimore, Maryland 21286
PH 410-825-7790 FAX 410-821-1054 EMAIL: vk@martellabs.com

All Procedures used are in accordance with the following methods:

"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, U.S. EPA Washington D.C., Third Edition, December 1996.

QC _____ *Approved* _____

Date 6/5/7 *Date* 6/5/07

CHAIN OF CUSTODY / SAMPLE INFORMATION FORM

Martel Laboratories JDS Inc. • 1025 Cromwell Bridge Road • Baltimore, MD 21286 • (410) 825-7790 • FAX (410) 821-1054

Martel Log # 44929 Client Code

Client Name/Phone/FAX: 434-250-1219 (cell)

Client Address: Kwatlin1@jhu.edu (email)

Invoice Address:

Sampler: Katie Watlington

Project Name/#: JHU / Honeywell

Contract/P.O Number

Sample Turnaround Time

Station No./ Sample ID	Station Location	Matrix	Container Description/ Preservation Status	Potentially Hazardous?	# of Containers	Date	Time	Analyses Required/Comments
	Wye River Control	Sediment			3	04/20	12:00	
	BSM 33	"			"	"	"	
	BSM 45	"			"	"	"	
	BSM 54	"			"	"	"	
	BSM 68	"			"	"	"	
	BSM 38	"			"	"	"	
	BSM 2 Reference	"			"	"	"	

Transferred by: Katie Watlington	Received by:	Date 04/20	Time 1200
Transferred by: Katie Watlington	Received by:	Date 4/25/07	Time 1710
Transferred by:	Received by:	Date	Time

Cooler Receipt Information **(LAB USE ONLY)**

Sufficient ice? - (Yes)/No If No, temp.= _____

Sample containers pres'd? - (Yes)/No If No, explain

Custody Seal present/intact? - Yes/No (N/A)

Initials: SK Date: 4/25/07