FINAL REPORT

The Determination of Sediment Polycyclic Aromatic Hydrocarbon (PAH) Bioavailability using Direct Pore Water Analysis by Solid-Phase Microextraction (SPME)
ESTCP Project ER-200709

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AECOM

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Table of Contents

List of Appendices iv
List of Acronyms v
List of Figures vi
List of Tables vi
Acknowledgements vii
Executive Summary viii
1.0 INTRODUCTION 1
1.1 BACKGROUND 1
1.2 OBJECTIVE OF THE DEMONSTATION 3
1.3 REGULATORY DRIVERS 4
2.0 TECHNOLOGY 6
2.1 TECHNOLOGY DESCRIPTION 6
2.2 TECHNOLOGY DEVELOPMENT 8
2.3 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY 8
3.0 PERFORMANCE OBJECTIVES 10
3.1 PRESENCE OF IMPACTED SEDIMENTS 11
3.2 PRESENCE OF SEDIMENT PAHs 11
3.3 BIOAVAILABILITY ESTIMATION 11
3.4 RISK-BASED APPROACH TO SEDIMENT MANAGEMENT 12
3.5 ASTM METHOD APPROVAL 12
3.6 TECHNOLOGY TRANSFER TO NAVY RPMs AND RISK ASSESSORS 12
4.0 SITE DESCRIPTION 13
4.1 SITE SELECTION CRITERIA 13
4.2 TEST SITE HISTORY/CHARACTERISTICS 16
4.3 PRESENT OPERATIONS 18
5.0 TEST DESIGN 19
5.1 CONCEPTUAL EXPERIMENTAL DESIGN 19
5.2 BASELINE CHARACTERIZATION 20
5.3 TREATABILITY OR LABORATORY STUDY RESULTS 20

5.4 DESIGN AND LAYOUT OF TECHNOLOGY COMPONENTS 20
5.5 FIELD TESTING 20
5.6 SAMPLING METHODS 20
5.6.1 Sample Station Access 21
5.6.2 Sample Station Locations 22
5.6.3 Field Measurements 24
5.6.4 Sediment Sample Collection for Chemistry and Toxicity Testing 24
5.6.5 Field Observations 24
5.6.6 Sample Location Mapping 25
5.6.7 Sample Processing Procedures 25
5.6.8 Field and Sample Documentation 27
5.6.9 Sediment Sample Analysis 27
5.6.10 Sediment Characterization 29
5.6.11 Data Interpretation 31
5.6.12 Experimental Controls 32
5.7 SAMPLING RESULTS 32
5.7.1 General Sediment Physical and Chemical Properties 33
5.7.2 PAH Chemistry 33
5.7.3 Aquatic Toxicity Testing 43
5.7.4 Organic Carbon 45
5.7.5 Detailed Carbon Chemistry 45
5.7.6 Interpretation of analytical results 48
5.8 Practical Application of the Demonstration Results 58
6.0 PERFORMANCE ASSESSMENT 62
6.1 Confirm the presence of impacted sediments at the site that are toxic to the aquatic test organisms 62
6.2 Confirm the presence of sediment PAHs in the biologically-active zone of the site 62
6.3 Validate the use of the analytical method to estimate the bioavailability of sediment-bound PAHs by predicting aquatic toxicity 63
6.4 Develop a tiered, risk-based approach to management of PAH-impacted sediment based on direct chemical measures of PAH bioavailability and the hydrocarbon narcosis model 63
6.5 Assist the SCBA in securing final ASTM approval of the SPME pore water method 64
6.6 Transfer the technology to Navy RPMs and risk assessors 64

7.0 COST ASSESSMENT 67
7.1 Cost Model 67
7.2 Cost Drivers 67
7.3 Cost Analysis 68
7.3.1 Ideal site description 69
7.3.2 Cost analysis assumptions 69
7.3.3 Cost Comparison 69
8.0 IMPLEMENTATION ISSUES 70
9.0 REFERENCES 71

List of Appendices

Appendix A: Points of Contact
Appendix B: EPA SW-8272 Method
Appendix C: ASTM Provisional Method D-7363-07
Appendix D: Site-Specific Health and Safety Plan (HASP)
Appendix E: Field Documentation Forms
Appendix F: Chain of Custody Forms
Appendix G: Quality Assurance Project Plan (QAPP)
Appendix H: General Sediment Physical and Chemical Properties
Appendix I: EERC Laboratory Report: Total and SPME PAHs, TOC and SOC
Appendix J: ERDC Laboratory Report: Aquatic Toxicity Tests
Appendix K: UMBC Laboratory Report: Detailed Carbon Chemistry
Appendix L: Detailed Petrography Images Of 0.25-1.0 mm Lighter Density Sediment Fractions

List of Acronyms

ASTM Association of Standard and Testing Materials
AWQC Ambient water quality criteria
CERCLA Comprehensive Environmental Response, Compensation, and Liability Act
COC Chain of custody
DDOE Washington DC Department of the Environment
DOC Dissolved organic carbon
DoD Department of Defense
EA Environmental Assessment
EERC Energy and Environmental Research Center
ERDC Engineer Research and Development Center
EqP Equilibrium partitioning
ESB Equilibrium sediment benchmark
ESTCP Environmental Security Technology Certification Program
FCV Final chronic values
GSA General Services Administration
HASP Health and Safety Plan
HSWA Hazardous and Solid Waste Management Amendment
ITRC Interstate Technology Regulatory Council
MGP Manufactured gas plant
MNR Monitored natural recovery
NAPL Non-aqueous phase liquid
NFESC Naval Facilities Engineering Service Center
NOAA National Oceanographic and Atmospheric Administration
NPL National Priorities List
PAH Polycyclic aromatic hydrocarbon
PCB Polychlorinated biphenyl P
PE Personal protective equipment
QAPP Quality Assurance Project Plan
SARA Superfund Amendments and Reauthorization Act
SCBA Sediment Contaminant Bioavailability Alliance
SERDP Strategic Environmental Research and Development Project
SOC Soot organic carbon
SPME Solid phase microextraction
SQT Sediment quality triad
STL Severn Trent Laboratories
TIE Toxicity identification evaluation
TOC Total organic carbon
TPAH Total polycyclic aromatic hydrocarbons
TU Toxic units
VOC Volatile organic carbon
UMBC University of Maryland – Baltimore County
US EPA United States Environmental Protection Agency
WG&L Washington Gas and Light
WNY Washington Navy Yard

List of Figures

Figure 1-1. Conceptual model for applying various sediment assessment approaches in a tiered system to determine the risk of adverse effects due to PAHs in sediments (source: U.S. EPA, 2009)
Figure 2-2. SPME pore water versus pore water calculated using equilibrium partitioning
Figure 4-1. Sediment total PAH concentrations at the Washington Navy Yard
Figure 5-1. Sediment sampling locations
Figure 5-2. Bulk Sediment PAH_{16} concentrations compared to *H. azteca* survival
Figure 5-3. Bulk Sediment PAH_{34} concentrations compared to *H. azteca* survival
Figure 5-4. SPME pore water TU PAH_{34} compared to *H. azteca* survival
Figure 5-5. EqP-estimated pore water PAH_{34} compared to *H. azteca* survival
Figure 5-6. Relationship between published Koc and WNY site-specific Koc values obtained for sample AR02
Figure 5-7. TUs calculated using EqP and from SPME pore water analysis
Figure 5-8. Bulk sediment PAH_{16} concentrations compared to growth of *H. azteca*
Figure 5-9. SPME pore water TU PAH_{34} compared to growth of *H. azteca*
Figure 5-10. Total PAH concentration in the particulate fractions of four sediment samples
Figure 5-11. Bulk sediment total PAH_{16} concentrations compared to the threshold effects level (TEL) of 1.6 mg/kg and the probable effects level (PEL) of 22.8 mg/kg
Figure 5-12. Toxicity of PAHs estimated using equilibrium partitioning
Figure 5-13. Prediction of toxicity based on the *H. azteca* 28-day test for survival and the toxicity predicted using the direct analysis of pore water PAHs using SPME

List of Tables

Table 3-1. Performance objectives
Table 4-1. Site selection criteria and final sample results
Table 5-1. Sample selection locations based on past PAH analyses
Table 5-2. Sediment samples and those selected for further analysis
Table 5-3. Sample handling requirements
Table 5-4. Analytical testing program
Table 5-5. Laboratory methods
Table 5-6. Sample grain size and basic chemistry
Table 5-7. Sediment total PAH concentrations and TUs
Table 5-8. Sediment SPME pore water PAH concentrations and TUs
Table 5-9. Aquatic toxicity test results
Table 5-10. Sediment TOC and SOC
Table 5-11. Properties of the four samples used for detailed carbon analysis
Table 5-12. Petrographic Compositional Analysis of the Sediment Organic Particle Fraction in the Size Range of 0.25 – 1.0 mm
Table 7-1. Cost model for the characterization of toxicity to benthic organisms using the SPME direct pore water PAH analysis and aquatic toxicity testing

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Dr. David V. Nakles of Carnegie Mellon University (formerly of RETEC/ENSR/AECOM) was the Principle Investigator (PI) for the project. AECOM provided overall project management, collected sediment samples, provided data analysis, wrote the interim and final reports, and served as liaison with the SCBA. Other AECOM employees who helped on this project include Dr. Stephen C. Geiger, Dr. Kathleen McDonough, Mr. Nicholas Azzolina, Ms. Helen Jones, and Ms. Heather Albert. Stephen Geiger took over project management responsibilities in the Spring of 2008.

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Executive Summary

Polycyclic aromatic hydrocarbons (PAHs) are often detected in many sediments adjacent to sites where industrial processes have operated. By their nature, PAHs are very hydrophobic, and tend to be tightly bound to the organic materials within sediments, making them unavailable for exposure to aquatic organisms. As a result of this binding phenomenon, there is often no correlation between the measured total PAH concentrations in sediments and those concentrations that adversely affect benthic organisms. Rather, these adverse effects are correlated to the dissolved-phase PAHs that are detected in sediment pore water. In spite of these observations, most PAH-contaminated sediment sites are evaluated and managed based on the total PAH concentrations determined on whole sediment samples.

To be protective of the environment and at the same time provide a more realistic process for evaluating the risks of PAHs to benthic organisms, a framework was developed by the U.S. EPA (EPA) entitled *Evaluating Ecological Risk to Invertebrate Receptors from PAHs in Sediments at Hazardous Waste Sites* (EPA-600-R-06-162F)(U.S. EPA, 2009). This framework proceeds in a sequential tiered manner, as follows:

1. Tier 1: Conduct an assessment of PAH bioavailability based on the analysis of whole sediments, using either empirical guidelines as screening levels or by modeling sediment pore water concentrations using equilibrium partitioning. If Tier 1 values are exceeded, action can be taken or, alternatively, a Tier 2 analysis can be conducted;

2. Tier 2: Conduct an assessment of PAH bioavailability based on the direct analysis of interstitial waters, i.e., pore water, of sediment. If significant bioavailable concentrations are detected, then action can be taken or, alternatively, a Tier 3 analysis can be conducted; and

3. Tier 3: Conduct sediment toxicity testing which reflects the bioavailability of the PAHs that are measured in the whole sediment. If there is significant toxicity, then there is a probable risk of adverse effects and appropriate action should be taken.

Note that the process can stop after completion of any tier of the framework, although it is possible that additional remedial action may be required since each level of analysis generally provides a more accurate assessment of the risk associated with the impacted sediment.

Traditionally, PAHs in sediments have been assessed through a comparison to screening levels/guidelines (i.e., Tier 1 analysis). A more site-specific assessment involves the use of the equilibrium partitioning theory to estimate pore water PAH concentrations from the whole sediment concentrations, followed by a comparison of pore water concentrations to published water-based effects levels. However, this approach has been shown to overestimate the impact of PAHs to benthic organisms in industrialized, urban waterways, because the partitioning coefficients used for this calculation generally do not account for the presence of anthropogenic carbon. It has been shown that anthropogenic carbon is more sorptive of PAHs than naturally-occurring organic carbon, which is the basis for the partition coefficients that are found in the literature.

Aquatic toxicity testing (i.e., a Tier 3 analysis) is another site-specific approach for assessing the effects of PAH impacts on benthic organisms. While aquatic toxicity tests reflect the bioavailability of the whole sediment PAHs, they do not take into account the presence of other contaminant effects, and are generally expensive, time consuming, and often difficult to interpret.

The EPA framework recognizes the need for a more precise, site-specific assessment and proposes the characterization of dissolved-phase PAHs in the sediment pore water as a measure of bioavailability that can be used to predict the toxicity of whole sediment PAHs to benthic organisms (i.e., a Tier 2 analysis). To this end, a consortium of industries called the Sediment Contaminant Bioavailability Alliance (SCBA) developed a laboratory method to directly determine the concentrations of PAHs in dissolved sediment pore water (EPA method SW-8272 and ASTM provisional method D-7363-07). These concentrations are compared to published water-based effects levels. To date, the SCBA has analyzed over 250 sediment samples at 18 industrial sites, mostly manufactured gas plants and aluminum smelters. The method utilizes solid-phase microextraction (SPME) on a very small sample of sediment (20 ml to < 40 ml) to provide PAH concentration data of sediment pore water, which has been shown to be correlated to the results of aquatic toxicity tests.

This demonstration project was designed to assess whether the SCBA protocol was applicable to Department of Defense (DoD) sites. Sediments at the Washington Navy Yard (WNY) in Washington DC were targeted for this demonstration, since they contain PAHs at concentrations which exceed published screening levels, and do not contain high concentrations of other potential compounds which might affect the health of benthic organisms. Fifteen surficial sediment samples were collected from the WNY and analyzed for total PAHs, pore water PAHs using SPME, total organic carbon (TOC), soot organic carbon (SOC), and general physical and chemical parameters. In addition, acute (i.e., survival) and chronic (i.e., growth) toxicity was assessed in a 28-day test involving the freshwater amphipod *Hyalella azteca*. A detailed analysis of the carbon types in four of the sediments was also conducted to provide more information on the processes involved in bioavailability of PAHs in sediments.

All of the sediments collected from the WNY had total PAH concentrations which exceeded the published screening levels (the total and probable effects levels of 1.6 and 22.8 mg/kg, respectively). However, the aquatic toxicity tests indicated that there was only one sample with reduced survival of *H. azteca*. The use of equilibrium partitioning to estimate pore water PAH concentrations did not result in a more accurate assessment of adverse effects when compared to the results of the aquatic toxicity test. However, the pore water PAHs directly analyzed using SPME did accurately predict the response of *H. azteca* observed in the aquatic toxicity tests. The detailed carbon analysis showed that the sediment sample exhibiting toxicity contained soot carbon, but was coated in oil, which prevented PAHs from partitioning into the sediment carbon.

This demonstration showed that the SPME analytical method and bioavailability assessment protocol developed by the SCBA may be applicable to PAHs associated with DoD sites, and that the results of this analysis are consistent with an industry database that was developed using several other PAH-impacted sediment sites.

1.0 INTRODUCTION

1.1 BACKGROUND

The Department of Defense (DoD) currently has environmental liabilities associated with polycyclic aromatic hydrocarbon (PAH) impacted sediments at many of their sites. The magnitude of these liabilities is determined by assessments of the ecological risks that are represented by these impacted sediments. One of the primary goals of these ecological risk assessments is to determine what concentration of PAHs can remain in the sediments without causing an unacceptable risk to the environment. Studies of the bioavailability of sediment-bound PAHs and their toxicity to aquatic organisms have shown that the use of the total PAH concentrations in the sediment to predict toxicity often overestimates the ecological risk, which can lead to additional remediation costs with no additional reduction in risk. The observed lack of toxicity at elevated total PAH concentrations in sediments has been attributed to the fact that these compounds are much more strongly bound to sediment organic carbon than is assumed by the standard equilibrium partitioning model, reducing their bioavailability to the receptor organisms.

In 2003, the EPA produced a document entitled *Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: PAH Mixtures* (EPA-600-R-02-013). This document provides a framework for assessing the environmental impact of sediment PAHs on benthic organisms. The document suggests that benthic organisms are most sensitive to pore water PAH concentrations and not total bulk sediment PAH concentrations. Since PAHs are generally metabolized by most benthic organisms (i.e., they do not bioaccumulate), and these organisms by definition are in intimate contact with sediments, they were selected as appropriate environmental receptors by which PAH-induced effects could be measured. This document presents an analytical method for accurately measuring the dissolved-phase concentrations of PAHs in sediment pore water and demonstrates the ability to use these data to accurately predict the survival of a benthic organism when exposed to these impacted sediments. These results are compared to alternative assessment approaches that involve the comparison of whole sediment PAH concentrations to published screening levels as well as the use of equilibrium partitioning (EqP) as a means to estimate the concentrations of PAHs in sediment pore water from the concentrations in the solid-phase (i.e., whole) sediment. In both approaches where the PAH concentrations in pore water available (measured or estimated) the pore water PAH concentrations are then related to threshold ambient water criteria (AWQC) which were derived to assess dissolved-phase impacts to benthic organisms. In the case of PAHs, the effect is general (i.e., non-specific) and is termed hydrocarbon narcosis. This document also presented a probability distribution of benthic invertebrate final acute values, indicating which organisms would be most susceptible to PAH impacts.

Historically, the major problem with the determination of pore water PAH concentrations by traditional means (i.e., direct centrifugation of sediment to isolate the pore water) is that a very large volume of sediment is required. In addition, the pore water that is collected in this manner may contain PAHs associated with particulate and dissolved organic carbon, in addition to the truly dissolved pore water PAHs. This confounds the assessment since it has been shown that it is the concentration of dissolved pore water PAHs which correlate with effects of PAHs to

benthic organisms. Furthermore, the traditional EqP method of estimating pore water PAH concentrations from bulk sediment concentrations assumes that the organic carbon in the bulk sediment is similar to naturally-occurring organic matter. However, in most industrialized and urban waterways of the United States, another carbon phase (black carbon or soot carbon) is ubiquitous. This soot carbon more tightly sequesters PAHs than naturally-occurring organic carbon, and thus the equilibrium partitioning coefficients from the literature which are used to estimate pore water concentrations from bulk sediment often overestimate the pore water PAH concentrations by up to four orders of magnitude. When these derived pore water concentrations are related to effects-level water concentrations for benthic organisms, they in turn overestimate the potential toxicity of the sediment to these organisms.

To better predict the impact of sediment PAHs to benthic organisms, an industry-led organization, the Sediment Contaminant Bioavailability Alliance (SCBA)[1], was formed to develop an analytical method to 1) directly determine dissolved-phase PAH concentrations in sediment pore water, thus alleviating the need to model or predict pore water PAH concentrations based on equilibrium partitioning from multiple phases of carbon that might be present, 2) enable the collection of a very small quantity of sediment (20 to 40 ml), and 3) provide low detection limits in the analysis of PAHs in pore water (pg/ml or ppt). The method developed by the SCBA is EPA SW-846 Method SW-8272 and ASTM Method D-7363-07 (provisional), which utilizes solid-phase microextraction (SPME). This method has the advantage that direct pore water PAH concentrations can be compared directly to effects-based water concentrations to determine PAH impacts to benthic organisms.

In 2009, the EPA produced a white paper as an addendum to the 2003 EPA PAH ESB document (U.S. EPA, 2003), entitled *Evaluating Ecological Risk to Invertebrate Receptors from PAHs in Sediments at Hazardous Waste Sites* (EPA-600-R-06-162F)(U.S. EPA, 2009). This white paper addresses the failure of EqP to accurately predict pore water PAH concentrations in the presence of soot/black carbon and acknowledges that direct measurements of pore water PAHs values (i.e. solid-phase microextraction or SPME) is a more selective tool for assessing narcotic effects of PAHs on benthic organisms (see Figure 1-1). In this document, the EPA recognizes the direct measurement of pore water concentrations of PAHs as a viable tool for determining PAH exposures to benthic organisms. The Tiered approach presented in Figure 1-1 indicates that these measurements alone may be sufficient to predict potential effects to the benthic community (i.e., aquatic toxicity testing may not be necessary).

In total, the SCBA collected sediment samples from 18 sites (> 250 sediment samples), mostly from former manufactured gas plant sites owned by utility industries, or former and current aluminum smelters. A protocol was developed whereby sediment solid and pore water phases were analyzed for PAHs, and the sediment was subjected to benthic organism aquatic toxicity testing using an appropriate test species. A direct correlation was observed between the pore water PAH concentrations as analyzed using the SCBA method to effect-level concentrations for benthic organisms, thus providing two legs of the Sediment Quality Triad (SQT) where sediment chemistry is represented by pore water PAH concentrations. Note that these tests were conducted using the amphipod *Hyalella azteca*, and in some cases were also conducted using the midge *Chironomous dilutans*. In all cases, *H. azteca* was more sensitive to PAH impact than *C.*

[1] The SCBA was formed through an alliance of National Grid, ALCOA, the Northeast Gas Association, and RETEC/ENSR/AECOM. The SCBA is no longer operative, having met its goals (development of an EPA and ASTM test method); however, the SCBA database can be accessed through Mr. Nicholas Azzolina of Foth Infrastructure and Environment, LLD, at (920)496-6783 or: NAzzolina@foth.com .

dilutans. The purpose of conducting the ESTCP demonstration/validation was to observe the ability of this chemical method to accurately predict the acute and chronic toxicity of PAH-impacted sediments to benthic invertebrates organisms at a DoD site.

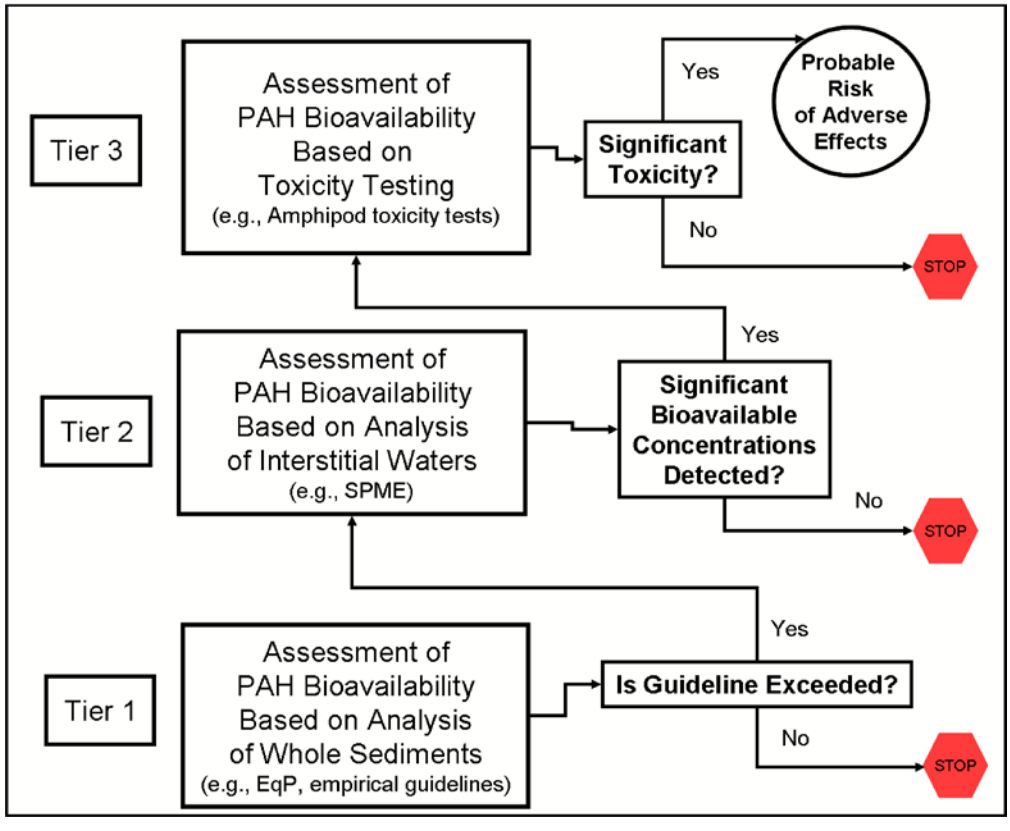


Figure 1-1. Conceptual Tiered Approach to Determine Risk of Adverse Effects Due to PAHs in Sediments (Source: U.S. EPA. 2009).

1.2 OBJECTIVE OF THE DEMONSTATION

The demonstration tests the applicability of a direct pore water analysis method for PAHs, the results of which were compared to toxic units (i.e., pore water concentrations divided by final chronic values) that result in hydrocarbon narcosis from PAHs to benthic organisms. As such, this method provides a site-specific estimate of PAH bioavailability in the lower Anacostia River sediments, and provides a direct comparison to aquatic toxicity test results utilizing *H. azteca* as a model benthic invertebrate species. This project served three purposes:

1. The project data supports the development of site-specific management strategies for sediments in the Anacostia River immediately adjacent to the Washington Navy Yard (WNY),
2. The project data supports a demonstration of this SPME analytical methodology that provides an important and cost effective sediment characterization tool for DoD site managers both at and beyond the WNY, and
3. The project data was combined with the existing SCBA database to support changes in the current regulatory paradigm for managing PAH-impacted sediments, which compares total PAH concentrations to published sediment quality guidelines.

Specific objectives of the demonstration project were to:

1. Use the SPME analyses of sediment pore water to predict the bioavailability of PAHs in freshwater sediments collected from the WNY site,
2. Compare the predicted bioavailability the impacted sediment PAHs to the actual measured toxicity of the sediment to the freshwater amphipod *Hyalella azteca*, and
3. Use these data to develop technical/regulatory guidance for the management of PAH-impacted sediments that incorporates the use of the site-specific estimates of PAH bioavailability for the purpose of predicting toxicity, assessing risk, and making more informed sediment management decisions.

1.3 REGULATORY DRIVERS

Remediation and management of contaminated sediments is often technically difficult and can be very expensive when large volumes of impacted sediments require treatment. The National Research Council (NRC) recently reviewed the implications and science regarding the bioavailability of contaminants in sediments, and determined that there is a need to improve risk-based assessments by including more explicit consideration of bioavailability processes (NRC, 2003). The SERDP and ESTCP Expert Panel Workshop on Research and Development Needs for the In-Situ Management of Contaminated Sediments (SERDP and ESTCP, 2004) also identified bioavailability as part of the following high priority research needs:

1. Develop and validate tools and techniques to assess site-specific bioavailability, and
2. Develop understanding of how sediment geochemical composition influences contaminant partitioning and bioavailability.

More recently, the SERDP-ESTCP Expert Panel Workshop on Research and Development Needs for Understanding and Assessing the Bioavailability of Contaminants in Soils and Sediments (SERDP and ESTCP, 2008) listed the need to better understand the nature, extent and behavior of PAHs in contaminated sediments at DoD sites.

The current approach to assessing the effect of PAH contamination in sediments is to implement one or more of the legs of the EPA Sediment Quality Triad (SQT), which consists of a weight-of-evidence evaluation of total chemical analysis, laboratory aquatic toxicity tests, and field benthic community surveys. For the total chemical analysis leg of the SQT, total PAH sediment concentrations are either directly compared to sediment quality guidelines/screening values or used to calculate equilibrium sediment benchmarks (ESBs) using the EPA equilibrium partitioning and hydrocarbon narcosis models. However, it has been shown that total PAH concentrations in bulk sediment may greatly overestimate the potential for sediments to be classified as toxic to aquatic organisms when using these methods. A more accurate method to predict toxicity to aquatic organisms is to use the bioavailable fraction, rather than the total concentration, of the contaminants in the sediments. PAHs in sediment pore water are closely related to the bioavailable fraction in sediments (U.S. EPA, 2003).

EPA has recommended that contaminant bioavailability be considered in the assessment of the residual risk that remains following the dredging, capping, or monitored natural recovery of

sediments (U.S. EPA, 2005; U.S. EPA, 2003). However, there is little guidance and no standard methods for determining the bioavailability of hydrophobic organic contaminants in sediments. This project demonstrates and validates a sediment characterization protocol for PAHs, one class of hydrophobic organic chemicals, that provides the analytical tools to generate site-specific bioavailability data at DoD sites. The demonstration also serves as technical guidance to use the results of direct pore water analysis and aquatic toxicity testing to make more informed and cost-effective sediment management decisions at DoD facilities impacted by PAHs.

The expected benefits of this technology are:

1. More accurate predictions of sediment toxicity and risk and the establishment of less conservative, site-specific remedial objectives, which will lead to reductions in the overall cost of sediment management (i.e., less volume of sediment requiring treatment),
2. Accurate assessments of PAH bioavailability, which will support improved evaluations of the efficacy of the remedial strategies that have been identified by the EPA in their recent guidance for sediment remediation at hazardous waste sites (U.S. EPA, 2005), i.e., monitored natural recovery (MNR), active/passive capping, and dredging,
3. Improved management of residual risk after the completion of MNR, active/passive capping and dredging of sediments and during the evaluation of beneficial reuse alternatives for dredged sediments, and

The results of this work will also provide the DoD with a technical protocol that will assist in formulating work plans and interpretation consistent with EPA guidance for sediment remediation at hazardous waste sites (U.S. EPA, 2005).

2.0 TECHNOLOGY

The concepts behind the direct pore water analysis approach used in the demonstration are presented in this section.

2.1 TECHNOLOGY DESCRIPTION

Studies of the bioavailability and toxicity of PAHs in sediments have shown that aqueous concentrations in pore water and aquatic toxicity are frequently much lower than expected based on predictions from total PAH concentrations. Consequently, sediments with high concentrations of PAHs often lack observable toxicity to sediment dwelling organisms (Bender et al., 1987; Hawthorne et al., 2007; Paine et al., 1996). For example, the toxicity of PAHs to the freshwater aquatic amphipod *H. azteca* was not related to the concentration of total PAHs determined using standard EPA extraction methods despite total PAH concentrations ranging from 4 to 5,700 mg/kg (Hawthorne et al., 2007) and no correlation could be shown between the concentration of total PAHs in sediments and toxicity to the marine amphipod *Rhepoxynius abronius* at an aluminum smelter in British Columbia, despite PAH concentrations up to 10,000 mg total PAH/kg sediment (Paine et al., 1996).

The observed lack of toxicity at high sediment PAH concentrations is attributed to the fact that these compounds are more strongly bound to sediment organic carbon than is assumed by the standard equilibrium partitioning model. PAHs sorbed to anthropogenic sources of "hard" or "black" organic carbon (e.g. charcoal, soot, coal or coke fines, or coal tar pitch) are more strongly sorbed and therefore less bioavailable to aquatic organisms than PAHs associated with natural sources of "soft", or natural organic carbon (Accardi-Dey and Gschwend, 2002; Ghosh et al., 2001; Talley et al., 2002). Although these particles generally account for a very small fraction of the sediment mass, they appear to control the release and bioavailability of PAHs. For example, detailed characterization of the sediment carbon, including petrographic identification and measurements of pitch content, showed that for sediment samples collected from MGP sites, light density particles dominated by "hard" carbon comprised 10-20% of the total mass of the sediment but contained 70-95% of the total PAHs (Talley et al., 2002).

Measured PAH partitioning coefficients between sediment organic carbon and water (Koc) were determined using 114 historically-contaminated and background sediments. The Koc values calculated from the measured pore water data ranged from 10 to 1000 times higher than literature Koc values that are commonly used for environmental modeling (i.e., the literature values overestimated the actual PAH concentrations in pore water) (Hawthorne et al., 2006; Figure 2-2). A mechanistic model for understanding the deviation from literature Koc values suggests differential PAH particle loading within the sediment carbon matrix, and the EPA has proposed using partitioning coefficients that are specific for "hard" soot-like and "soft" natural organic carbon to improve estimates of PAH bioavailability (U.S. EPA, 2000; U.S. EPA, 2003). However, these adjustments often do not improve the ability to predict the aquatic toxicity of the impacted sediment (Hawthorne, et al., 2007b; McDonough and Azzolina, 2010).

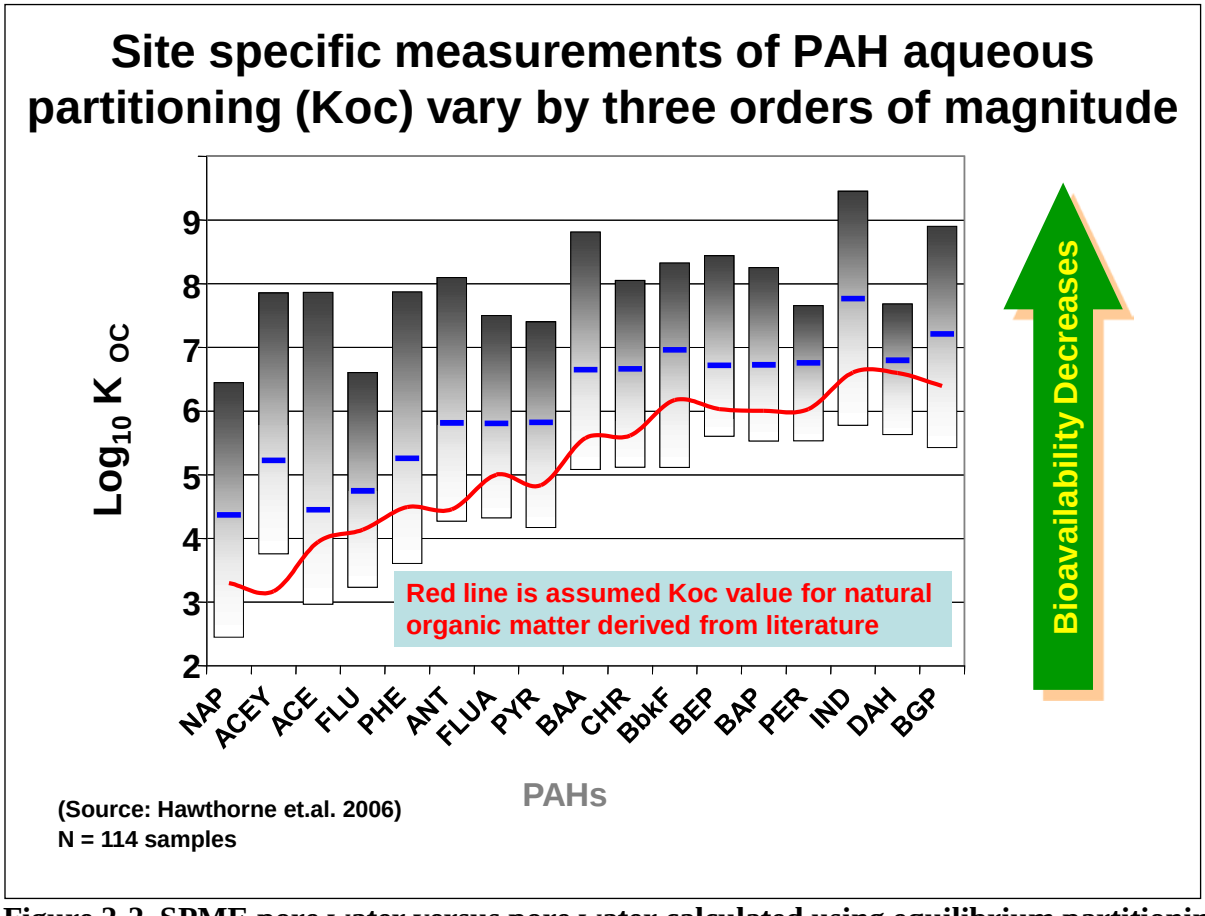


Figure 2-2. SPME pore water versus pore water calculated using equilibrium partitioning

To eliminate the uncertainty in pore water concentrations using published equilibrium partitioning coefficients, a method to directly analyze PAHs in pore water has been developed. An industry-led organization, the Sediment Contaminant Bioavailability Alliance (SCBA), was formed to develop an analytical method to 1) directly determine dissolved-phase PAH sediment pore water concentrations, thus alleviating the need to model or predict pore water PAH concentrations based on equilibrium partition from multiple phases of carbon that might be present, 2) enable the collection of a very small quantity of sediment (20 to 40 ml), and 3) provide low detection limits in the analysis of PAHs in pore water (pg/ml or ppt). The method developed by the SCBA is EPA SW-846 Method SW-8272 (see Appendix B) and ASTM Method D-7363-07 (provisional; see Appendix C), and utilizes solid-phase microextraction (SPME).

SPME has been used to directly estimate PAH bioavailability and predict the toxicity of PAHs in contaminated sediments with much greater accuracy than can be achieved by comparing total PAH concentrations to sediment screening guidelines (Hawthorne et al., 2007; Hawthorne, et al., 2002; Hawthorne, et al., 2005a; Hawthorne et al., 2005b). The pore water concentrations measured by SPME are expressed in terms of toxic units (TUs), as defined by the EPA procedures for the derivation of equilibrium partitioning sediment benchmarks (U.S. EPA,

2003)[1]. The TUs strongly correlate with survival of the freshwater amphipod *H. azteca*, the freshwater midge *Chironomous dilutus*, and the marine amphipod *Leptochirus plumulosus* (Hawthorne etal., 2007). The individual PAH TUs are summed over a sediment sample to arrive at a total PAH TU. A TPAH TU<1 indicates no probable impact to benthic organisms, while a TPAH TU>1 indicates a potential impact to benthic organisms (U.S. EPA, 2003).

It should be noted that the SPME that is utilized in this method is not the biomimetic SPME which has been extensively reported in the peer-reviewed literature (i.e., that which is being evaluated in ESTCP Project ER-0624: *Demonstration and Evaluation of Solid Phase Microextraction for the Assessment of Bioavailability and Contaminant Mobility*). Rather, the SPME of this method is being used strictly as an analytical tool to measure PAHs at very low detection limits (picograms/liter) in small samples (1.5 mL) of pore water (Hawthorne et al., 2005b). In fact, this SPME method is the only method available to achieve these detection limits using such small sample volumes. As an alternative to applying this SPME method to *ex situ* samples of sediment pore water to estimate the bioavailable PAHs in a sediment, other researchers are examining the field deployment and retrieval of passive SPME samplers (Hawthorne et al., 2005b). Ultimately, the method of choice for estimating bioavailability of PAHs, or any hydrophobic organic chemical in sediment, will be based upon the performance, ease of implementation, and costs of these various measurement techniques.

2.2 TECHNOLOGY DEVELOPMENT

A significant benefit of this dem/val project is the ability of the DoD to leverage the prior and on-going R&D investment being made by the SCBA. The SCBA has invested over $10 MM in the development of the SPME analytical methods and the characterization of over 250 sediment samples from 18 contaminated sites. The SCBA has initiated communications and technology review with various state regulatory agencies and the EPA, has conducted several technical workshops for regulatory agencies in 2007 and 2008, has presented results at symposia and conferences, and has obtained an EPA SW-846 method number and a provisional ASTM method number for the analytical method.

The addition of the ESTCP data will help establish the robustness of this pore water approach and will expedite regulatory acceptance and development of EPA guidance on its use for generating site-specific estimates of PAH bioavailability to predict toxicity and to characterize the risk of contaminated sediments. As such, this research will help attain state and federal agreement on the use of sediment bioavailability measurements for more accurate sediment characterization, development of less conservative risk–based cleanup objectives, and the more effective management of residual risks following the completion of sediment management using monitored natural recovery (MNR), active and passive caps, and dredging.

2.3 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

The expected benefits of this technology are more accurate assessments of sediment toxicity and risk and the establishment of less conservative, site-specific remedial objectives, which will lead

[1] A TU is the concentration of an individual PAH in sediment pore water divided by the published final chronic value (FCV) for that PAH. The FCV for individual PAHs is presented in EPA (2003), and is a water-based concentration derived from experimental results on benthic organisms.

to reductions in the overall cost of sediment management (i.e., less volume of sediment requiring treatment). The accurate bioavailability assessments obtained by using this technology will also support improved designs and efficacy evaluations of the remedial strategies that have been identified by the EPA in their recent guidance for sediment remediation at hazardous waste sites (U.S. EPA, 2005), i.e., MNR, active/passive capping, and dredging. This method can also be used for the improved management of residual risk after the completion of MNR, active/passive capping and dredging of sediments and during the evaluation of beneficial reuse alternatives for dredged sediments.

There is little risk associated with applying the sediment characterization assessment protocol at field sediment sites since similar studies have been conducted at 18 industrial sites, to date. A generic bioavailability assessment work plan was developed for the conduct of these field studies in New York. This work plan was reviewed by the state regulators prior to its use at utility and aluminum field sites.

The analytical method has received complete EPA and provisional ASTM approval (EPA SW-846 method 8272 and ASTM D-7363-07). Final ASTM approval is pending the results of inter-laboratory validation studies. Regulatory acceptance and approval of this analytical method are necessary for the broad application and use of this sediment characterization protocol at DoD facilities across the United States.

However, the estimation of bioavailable PAHs based on SPME analyses to predict sediment toxicity has not yet received regulatory acceptance at either the state or Federal level. The SCBA also recognized these risks and has focused much of its efforts of the last five years on conducting work to mitigate both of them. The addition of the data from this demonstration will contribute significantly to this ongoing effort.

The SPME method has been researched and verified for predicting PAH bioavailability in sediments at sites where these compounds were the predominant contaminant. However, the applicability of this approach to sites containing complex mixtures of contaminants has not yet been validated. The extension of the work to this application may require toxicity identification evaluations (TIEs) to resolve any confounding toxicity test results.

Costs associated with the SPME pore water methodology have not yet been standardized, although selected commercial U.S. laboratories have been conducting this method (e.g., Test America and Meta Environmental). As with all chemical methods, the cost of analysis needs to be balanced with overall expected benefits of conducting the analysis. A major component of the research used to develop the SPME pore water method has been to reduce uncertainty in cost/benefit site analysis.

3.0 PERFORMANCE OBJECTIVES

Performance objectives for this demonstration are provided in Table 3-1. The performance objectives were met using a technical task structure that has been developed for this project, and which is complimentary to the task structure that has been used within the SCBA case studies. A discussion of each of the performance objectives is provided.

Table 3-1: Performance Objectives.

Performance Objective	Data Requirements	Success Criteria	Results
Quantitative Performance Objectives			
Confirm the presence of impacted sediments at the site that are toxic to the aquatic test organisms	Standard sediment aquatic toxicity testing	Range of survival/growth of *H. azteca* from no impact to severe impact (i.e., acute toxicity)	Range in *H. azteca* response was obtained (no impact to acute toxicity)
Confirm the presence of sediment PAHs in the biologically-active zone of the site	Standard sediment PAH analysis	Range of total PAH concentrations above and below screening criteria (i.e., PEL of 22.8 mg/kg)	Sediment total PAH concentrations ranged from 14 to 600 mg/kg
Validate the use of the analytical method to estimate the bioavailability of sediment-bound PAHs by predicting aquatic toxicity	Aquatic toxicity results and toxic units (TUs derived from individual pore water PAHs and their respective final chronic values (FCV)	A quantitative correlation will exist between sediment pore water toxic units based on the concentration of 34 PAHs and the results of aquatic toxicity tests using the amphipod *H. azteca.*	A good correlation was observed for the data, consistent with the existing SCBA database
Qualitative Performance Objectives			
Develop a tiered, risk-based approach to management of PAH-impacted sediment based on direct chemical measures of PAH bioavailability and the hydrocarbon narcosis model	Aquatic toxicity and pore water PAH concentrations from the SCBA database as well as the ESTCP dem/val	A presentation of this risk-based approach will be made to representatives of State and Federal regulatory agencies.	Resulting data was used to comment on the EPA Draft White paper, resulting in substantial white paper revisions that includes pore water testing as a second tier analysis (prior to aquatic toxicity testing)
Assist the SCBA in securing final ASTM approval of the SPME pore water method	Conduct an inter-laboratory validation of the methodology to ASTM specifications	A final ASTM method number will be in place by the end of Calendar Year 2007	Final inter-laboratory analysis will be completed by the Fall of 2010 and the ASTM Standard method should be in place in Spring 2011

Performance Objective	Data Requirements	Success Criteria	Results
Quantitative Performance Objectives			
Transfer the technology to Navy RPMs and risk assessors	All ESTCP demonstration and SCBA results will be presented at various forums	Technical presentations will be made to the appropriate organizations. A generic work plan, QAPP, and technical /regulatory guidance for Navy sites will be generated. A web-based tool to facilitate access to the project results for Navy personnel will be developed.	Presentations were made to various organizations. The demonstration provided a generic work plan, QAPP, and technical/regulatory guidance for Navy sites. The web-based tool is under development, and will be finalized based on the accepted Final Report

3.1 PRESENCE OF IMPACTED SEDIMENTS

The presence of sediments containing PAHs at concentrations having impact on an aquatic test species was tested by conducting a standard 28-day toxicity evaluation using the amphipod *Hyalella azteca*. Aquatic toxicity text endpoints included mortality (i.e., percent survival) and growth. The goal was to obtain sediment samples from the area adjacent to the WNY which exhibited impacts to the aquatic test species ranging from no impact to acute/chronic impacts. Of the 15 collected sediment samples, one exhibited a reduced survival and one exhibited reduced growth. All other samples did not impact the test species. Therefore, the performance objective was successful in that a range of biological responses was obtained.

3.2 PRESENCE OF SEDIMENT PAHs

The WNY site was selected based on past characterization data which indicated 1) the presence of PAHs at a concentrations exceeding the probable effects concentration (PEL) of 22.8 mg/kg, and 2) the lack of other constituents which might cause a negative impact on the benthic community. A total of 36 sediments were field collected and pre-screened for total PAH concentrations. Fifteen of these sediments were selected for final study based on a range of PAH concentrations. The PAH concentrations ranged from 14 mg/kg to 600 mg/kg.

3.3 BIOAVAILABILITY ESTIMATION

Pore water PAHs were directly analyzed on the final 15 sediment samples. Pore water PAH concentrations were compared to Final Chronic Values (FCV) for each individual PAH, resulting in a toxic unit (TU) for that PAH in each sample (i.e., PAH/FCV = TU). A TU>1 indicates a potential for negative impact to benthos. The individual TUs were summed for each sediment sample (18 parent and 16 alkylated PAHs) to compute a PAH_{34} TU. PAH_{34} TUs were plotted against percent survival of the *H. azteca*. This plot was superimposed on an identical plot that included the entire SCBA database, which had been fit using probit analysis to depict the dose-response curve. This exercise demonstrated that the direct pore water analysis conducted at the

WNY was able to correctly predict the survival of *H. azteca* and was consistent with the previous data that had been generated at the 18 other field demonstration sites of the SCBA. Also, as was the case with the SCBA database, there was no correlation at the WNY between total sediment PAHs, pore water PAHs, and growth of *H. azteca*.

3.4 RISK-BASED APPROACH TO SEDIMENT MANAGEMENT

Several activities were completed related to the implementation of a risk-based approach to PAH sediment management using the direct pore water method. These were partially funded by ESTCP, and partially funded by the industry partners of the SCBA. These activities included discussion with state regulators on the potential acceptance of the SPME porewater method, as well as a response to a Federal Register Notice by the EPA on the evaluation of sediment PAHs with regards to their impact to benthic organisms. More detail on these activities is presented in Section 6.4 of this document.

3.5 ASTM METHOD APPROVAL

The provisional ASTM method is required to undergo a more exhaustive inter-laboratory evaluation that uses the analytical method to generate a minimum of seven independent data sets for a set of control samples prepared by a custodial laboratory. ESTCP provided partial funding for this ASTM approval process, which was used to set up the program and identify participating laboratories. More information on this activity is presented in Section 6.5 of this document.

3.6 TECHNOLOGY TRANSFER TO NAVY RPMs AND RISK ASSESSORS

The technology transfer performance objective is divided into three parts:

- Technical presentations
- Technical/regulatory guidance
- Development of a Web-based tool

The results of these activities are presented in Section 6.6 of this document.

4.0 SITE DESCRIPTION

A description of the WNY sediment site and lower Anacostia River is provided in this section.

4.1 SITE SELECTION CRITERIA

AECOM defined site selection criteria to serve as a basis for screening candidate sites for the bioavailability assessment case studies of the project. These criteria and their relative importance to the selection process are provided in Table 4-1. Using the information that was made available to AECOM by the site personnel and other interested parties, an evaluation of the site was completed. For comparison purposes, also shown in Table 4-1 are the final sampling results from the WNY that are relevant to each of the selection criteria.

Table 4-1. Site Selection Criteria and Final Sample Results.

Parameter	Preferred Value	Relative Importance (1-5 with 1 being highest)	Final Sampling Results
Contaminant of Concern – PAHs	100 – $1x10^6$ ug/kg (ppb)	1	Range of 14.4 to 600 mg/kg
Well-characterized Site	Yes	1	Yes
PAHs Identified as Risk Drivers	Yes	1	Yes
Interest of Site Manager	High	1	High
Anthropogenic Carbon in Sediment	Present	1	SOC was measured in all samples
Concentration of VOCs	Low (below toxicity levels, if possible)	2	Overall low levels from previous studies
Sediment Texture	Silt/Clay	2	Silt/Clay
Concentration of Metals	Low (below toxicity levels if possible.	2	Overall low levels from previous studies
Presence of Free-phase Hydrocarbons	None	3	None observed
Water Depth	< 50 feet	4	~ 10 ft
EPA CSTAG Site	Yes	4	WNY is currently on the NPL list but is not a CSTAG site
Located within ASTSWMO or ITRC State	Yes	4	Yes

Available site data which were reviewed included sediment concentrations and toxicity information from 35 databases that were compiled by NOAA (http://mapping.orr.noaa.gov/website/ portal/Anacostia River/); a 2003 U.S. Fish and Wildlife Service report entitled *Assessing the Bioavailability of Organic Contaminants in the Anacostia River Using Semi-permeable Membrane Devices and Filter-feeding Clams;* a 2001 report from the Academy of Natural Sciences entitled *Sediment Transport: Additional Chemical Analysis Study, Phase II*; documents from the Anacostia Watershed Toxics Alliance (AWTA), and data collected during the Phase I sediment characterization at the WNY in 2006.

Previous studies have shown that PAH concentrations in this part of the Anacostia River varied from 100 to 211,000 ppb (Figure 4-1). This was an appropriate range for the demonstration. The PAH concentrations are based on measuring the 16 parent PAHs (PAH_{16}), not the 34 NOAA PAHs that the SCBA program would normally use to screen a site (PAH_{34}). The fluoranthene to pyrene ratios for some of the historical sediment samples were greater than 1, suggesting a pyrogenic source of the PAHs such as coal carbonization. Work done previously by the SCBA (case studies at 18 different sediment sites) has shown that these types of samples generally have low bioavailability.

The freshwater PEC for PAH_{16} in sediment is 22,800 µg/kg. Based on the data from the SCBA database, this sediment screening value is only able to accurately predict toxicity to *H. azteca* 48% of the time because not all of the PAHs are bioavailable. In contrast, the measurement of pore water PAH_{34} concentrations is able to accurately predict toxicity to *H. azteca* 90% of the time (Hawthorne, et.al., 2007). The final PAH_{16} concentrations at the site ranged from 14 to 600 mg/kg.

Status of Site Characterization

The WNY sediments have been well characterized, with the last sampling occurring in 2006 as a Phase I site characterization. Several sediment samples have been analyzed from the areas associated with the Washington Gas and Light (WG&L) and General services Administration (GSA) sites; however, these areas have not been adequately characterized. It was expected that the highest concentrations of PAHs would be found in the area associated with the WG&L site, due to past manufactured gas plant operations.

PAHs Identified as Risk Driver

The Anacostia Watershed Toxics Alliance (AWTA) indicated that PAHs are a risk driver, in that fish tumors that have been observed in the river are being associated with the presence of the PAHs. Although the PAH concentrations are not particularly high, the lack of adequate sampling near the WG&L site suggests that higher concentrations of PAHs, typical of most MGP sites, may be present in this area.

Presence of Anthropogenic Carbon

TOC levels in the sediment samples were correlated with PAH concentrations, which suggests that anthropogenic carbon is present in the sediment. As noted previously, a site with anthropogenic carbon present is highly desirable. Given the correlation between TOC levels and PAH concentrations, the history of the WNY as a manufacturing facility, the history of the WG&L site as a manufactured gas plant site, and the urban runoff that continually enters the Anacostia River in this area through local outfalls and other upstream sources, there was ample reason to believe that anthropogenic carbon was present in the sediment.

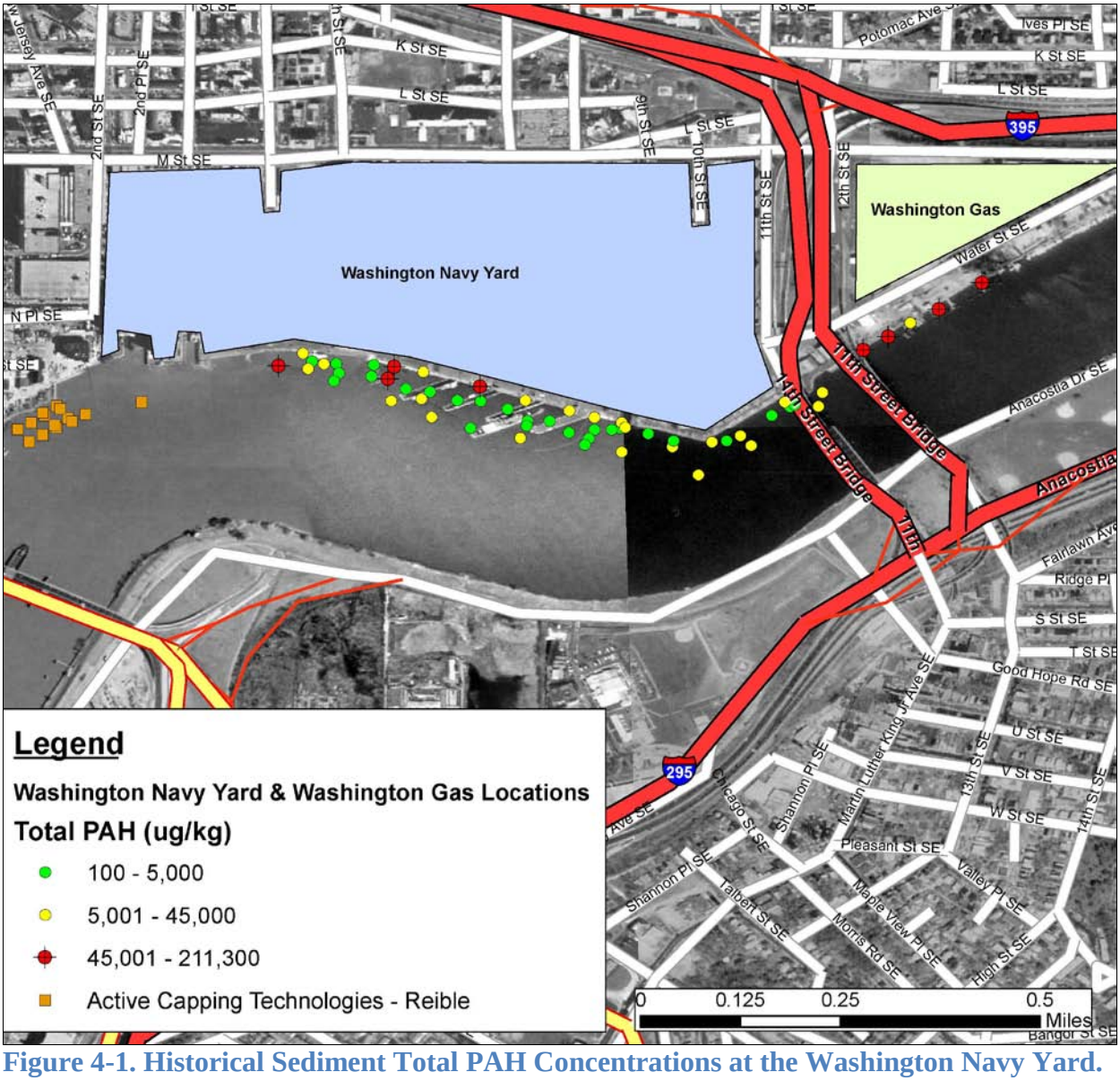


Figure 4-1. Historical Sediment Total PAH Concentrations at the Washington Navy Yard.

Sediment Texture

A silt/clay composition was preferred for the demonstration site although the technical approach can deal with the full range of sediment textures. The sediments in the WNY area of the Anacostia River generally consist of silt and clay.

Concentrations of Metals and VOCs

The Anacostia River does not have high levels of other contaminants of concern. Previous studies indicated that metal concentrations are generally very low. A few of the samples just barely exceeded the PEC for copper and one of the samples barely exceeded the PEC for nickel. Other than those few samples, the metals concentrations were below probable effects concentrations (PECs) except for lead. Lead concentrations exceeded the PEC in greater than 50% of the sediment samples collected and could be a possible cause of toxicity to *H. azteca*.

However, no acid volatile sulfides data was available and without these data it was not possible to determine whether or not lead is likely to be available for partitioning in the pore water. PCB concentrations were also fairly low in this area, with a maximum concentration of 12 ppm. There have not been any reported elevated levels of VOCs in the river sediments.

Free Phase Hydrocarbons
Based on preliminary conversations about the site and on the preliminary review of the sediment data, there did not appear to be significant amounts, if any, of free-phase hydrocarbons present in the sediment.

Water Depth
The water depth in the specified areas is generally less than 10 ft, which is within the range of the site selection criteria.

EPA or ASTSWMO/ITRC State Site
Environmental concerns in the Anacostia River are being addressed by the Anacostia Watershed Toxics Alliance (AWTA) which is a group of stakeholders led by EPA Region 3, and which includes NOAA, the U.S. Fish and Wildlife Service, the U.S. Park Service, the U.S. Corps of Engineers, and the District of Columbia Department of the Environment (DDOE). The DDOE has local jurisdiction over the river sediments and is a participant in the ITRC. The demonstration results were shared with the ITRC-SCBA regulatory advisory team, which consists of regulators from five ITRC states (WA, OR, NY, NJ, and MA).

4.2 TEST SITE HISTORY/CHARACTERISTICS

The demonstration site is located in the lower Anacostia River, which is tidal and also is a depositional area of the river. The area of the test includes sediments adjacent to WG&L, WNY, and the Southeast Federal Center (SEFC) operated by the GSA. Numerous sewer outfalls from the District of Columbia also occur in this area. Additional information about these areas, and the Anacostia River in general, can be found at NOAA's Anacostia River Watershed Database and Mapping website (http://mapping.orr.noaa.gov/website/portal/AnacostiaRiver/). In addition, the Anacostia Watershed Toxics Alliance (AWTA), a consortia of various stakeholders, provides information on contaminants and contaminant sources within the watershed (http://www.epa.gov/oswer/onecleanupprogram/anacostia.htm).

Washington Gas and Light Company
The WG&L site covers an area of approximately 19 acres, and includes property owned by Washington Gas, the National Park Service (NPS), the Washington DC Government, and the U.S. Army Corps of Engineers (USACE). The portion of the site owned by WG&L formerly contained the East Station MGP, which started operating in 1888. Operations continued until 1948, when WG&L converted to natural gas. Between 1948 and 1983, the plant was used only intermittently for periods of peak gas demand during winter months. Demolition of the MGP was completed in 1985 and the oil tanks were removed in 1997. In 1993, WG&L installed a two-story brick building containing offices and a groundwater treatment system.

The NPS property along the river is used principally by the D.C. Department of Public Works Street Maintenance Division to store and maintain heavy equipment. Small portions of the NPS property located under and east of the 11th Street Bridge are accessible to the public and mainly

used by a rowing club. The 18.8 acre parcel owned by the USACE is adjacent to the river and used as a staging area for the removal of floating debris from the Anacostia and Potomac rivers.

Since 1976, WG&L has been pumping and treating groundwater to remove dense non-aqueous phase liquid (DNAPL) and dissolved organic constituents of DNAPL. There have been limited, but incomplete, sediment investigations of PAHs associated with the WG&L site in the Anacostia River. Petroleum spills impacted the river from a Petroleum facility (Stuart Petroleum) located behind the WG&L site sometime in the 1960's and again in the early 1980's.

Washington Navy Yard

WNY is the oldest continuously operated Navy facility in the United States. It currently occupies 71.5 acres in the District of Columbia, and is bordered by the Anacostia River to the south, the SEFC to the west, public housing to the north, and WG&L East to the east. The facility was opened officially on October 2, 1799, and ship building and repair operations were ongoing by 1822. During the 1800's, ordnance production, research, and other industrial activities were prevalent at the yard. In 1886, the WNY was redesignated as the Naval Gun Factory. During the next 20 years, considerable expansion of the WNY occurred, and production of ordnance remained the primary operational activity at the facility during this time. Significant areas of adjacent marshlands were filled to accommodate the WNY.

In the 1940's, the primary role of the WNY shifted from production of ordnance to administrative activities. Although administrative activities became a large function of the WNY, all ordnance production was still monitored or tested at the facility. To accommodate the expanded activity, new administrative and research facilities were constructed on the eastern portion of the property. In 1961, the WNY officially became an administrative facility. Activities currently conducted at the WNY include administration, supply and storage, and training. An historic center that is open to the public is also currently located there.

Records documenting the wastes generated during the ordnance production or the various other industrial processes that occurred at the WNY have not been located, however, based on the description of the documented operations at the WNY, the typical wastes generated were determined. These wastes would include metals used in ordnance production and paint-spraying; solvents use in cleaning; cyanide and phenols use in the cooling process; creosote used in wood treatment, petroleum products and wastes; and PCB-containing oils in storage tanks and electrical equipment. Contamination also likely occurred during storage and handling of raw materials. The storm water system draining the facility is contaminated with metals and PCBs, which can be attributed to the industrial processes and ordnance production that historically occurred at the facility. The storm water system leads to nine outfalls that empty into the Anacostia River. Sediment sampling of the river shows metals, PAHs, and PCB contamination. In addition, volatile and semi-volatile contaminants have been found in soils throughout the facility. The WNY is on the EPA's National Priority List (NPL) as a hazardous waste site.

Southeast Federal Center (SEFC)

The SEFC is located to the west of the WNY, and comprises approximately half of the original 124-acre WNY site. PAHs, PCBs, and heavy metals have been detected on site and in the Anacostia River sediments. Remedial actions that are already underway or completed include: removal of contaminated sediments (heavy metals and PCBs) from four stormwater outfalls, including the primary storm sewer outfall onsite (December 1988), remediation or razing of 12

buildings contaminated with PCBs, heavy metals and asbestos; remediation of soil hot spots at 11 sites contaminated with heavy metals and PCBs; and renovation of the seawall along the Anacostia River.

PAHs in Sediments

PAHs in sediments have been reported from various studies within the test area of the Anacostia River. Previous studies show a range of 0.1 to 211 mg TPAH/kg sediment, while this ESTCP demonstration showed a range of 14 to 600 mg/kg.

The Freshwater Probable Effects Concentration (PEC) for TPAHs is 22.8 mg/kg. Based on previous studies, there are sediments containing TPAH concentrations in this area of the Anacostia River which exceed the regulatory standard. It should be noted that deposition of sediments from upriver is constantly occurring in this area. It has been estimated that approximately one inch of new sediment is deposited in this area every ten years (Kris Murthy, WG&L, personal communication), and that this area of the lower Anacostia River is often referred to as the depositional area of the river.

4.3 PRESENT OPERATIONS

Today the Navy Yard houses a variety of activities. It serves as headquarters, Naval District Washington, and houses numerous support activities for the fleet and aviation communities. There is currently a Phase II sediment investigation ongoing at the site.

5.0 TEST DESIGN

This section presents the planning, sampling, and data analysis requirements of the demonstration. These discrete technical elements of the demonstration are described below.

Historical Data Review, Identification of Sample Locations and Collection of Sediment Samples: The Project Team reviewed the historical sediment data from the area in front of and adjacent to the WNY site and identified preferred sampling locations for both impacted and reference sediment samples. A total of 36 sediment samples were collected as part of the demonstration.

Chemical Screening of Sediment Samples: The 36 sediment samples were rapidly analyzed (sonication) for total PAH_{34} concentrations to ensure that a broad range of PAH impacts are included in the demonstration. Based on these chemical screening results, 15 samples were selected for detailed chemical and biological testing.

Detailed Chemical Characterization of Sediment Samples: Total sediment and SPME pore water PAH concentrations were determined on each sediment sample. Basic characteristics of the sediment samples were also determined including grain size distribution, pH, and ammonia. TOC and SOC were determined for use in estimating pore water PAH concentrations using equilibrium partitioning.

Aquatic Toxicity Testing and Comparison with Toxicity Predictions Based on Estimates of Bioavailability: The acute (survival) and chronic (growth) toxicity of the selected sediment samples to the freshwater amphipod *H. azteca* were determined in a 28-day test. The SPME analysis results for the PAHs in the sediment pore water were converted to toxic units (TU), which were summed to produce a total toxic unit for the 34 PAHs of interest (i.e., TU_{34}). The toxicity test results were plotted against the pore water TU_{34} to investigate the existence of a dose-response relationship. As previously discussed, this data set was compared to the existing SCBA data set to show the similarity in the relationship between aquatic toxicity and pore water PAH concentrations.

Characterization of Sediment Carbon: The carbon types within a subset of four of the sediment samples were characterized to evaluate the potential for particle-scale binding of PAHs, which would provide support for a mechanistic interpretation of the observed bioavailability and toxicity data.

5.1 CONCEPTUAL EXPERIMENTAL DESIGN

The demonstration consisted of applying the SCBA protocol for evaluating PAH impacts to the benthic community in surficial sediments (biologically-active zone) at the Washington Navy Yard. This protocol consists of the following steps:

- Historical review of existing sediment PAH data
- Selection of sediment sampling locations, including background/reference sample locations

- Sampling of surficial sediments and preparation (e.g., debris removal, on-site screening and mixing) for shipment to the analytical laboratories
- Rapid screening of sediments samples for total PAHs (i.e., extraction by sonication)
- Selection of subset of sediments for detailed analysis:
    1. Total PAHs (soxhlet extraction)
    2. Pore water PAHs using SPME
    3. Total organic carbon (TOC)
    4. Soot organic carbon (SOC)
    5. Aquatic toxicity (survival and growth)
    6. Other physical and chemical parameters
- Selection of four sediments for detailed carbon analysis
- Creation of a total PAH toxic unit for each sediment sample
    1. Correlation of sediment PAH TUs to aquatic toxicity results
        - Total PAHs and survival of *H. azteca*;
        - Pore water PAH TU_{34} estimated using equilibrium partitioning and survival of *H. azteca*; and
        - Pore water PAH TU_{34} from direct SPME analysis and survival and growth of *H. azteca*
- PAH concentrations in total sediment and pore water, and the aquatic toxicity results, were evaluated in combination with default screening levels to make conclusions about potential impacts to the benthic community (weight-of-evidence approach)

5.2 BASELINE CHARACTERIZATION

Previous sediment total PAH concentrations, as well as site characteristics, were evaluated for suitability for the demonstration (see Section 4.0). Based on this information, twenty-five sediment sampling sites were selected based on past total PAH concentration data (Table 5-1). Note that 36 sediments were actually sampled due to the ease of sampling at this site (see Section 5.6.2 and Table 5-2). These sample locations included six original reference sites (AR20 through AR25).

5.3 TREATABILITY OR LABORATORY STUDY RESULTS

There was no pre-testing or analysis conducted as part of this demonstration, and therefore no treatability studies or laboratory studies were conducted prior to the field sampling effort.

5.4 DESIGN AND LAYOUT OF TECHNOLOGY COMPONENTS

No technology components were utilized for this demonstration.

5.5 FIELD TESTING

No field testing activities were undertaken for this demonstration.

5.6 SAMPLING METHODS

This section presents the procedures that were used to conduct field work to assess the bioavailability and toxicity of PAHs in aquatic sediments. The sediment sampling activities conformed to EPA and ASTM standard methods where appropriate (ASTM, 2000a; ASTM, 2000b; U.S. EPA, 2001).

Table 5-1. Sample Selection Locations Based on Past PAH Analysis.

AECOM ID	Study Name	Latitude	Longitude	Total PAH (ug/kg)
AR01	1996 WA Gas - East Station Project	38.873621	-76.988472	167700
AR02	1996 WA Gas - East Station Project	38.873131	-76.989395	211300
AR03	1996 WA Gas - East Station Project	38.872885	-76.989846	87100
AR04	1999 WA Navy Yard RI	38.871890	-76.991090	3710
AR05	2006 CH2M HILL	38.87115	-76.9918759	21519
AR06	2006 CH2M HILL	38.87062	-76.9928324	14950
AR07	2006 CH2M HILL	38.87104	-76.9942267	11980
AR08	1999 WA Navy Yard RI	38.871390	-76.995180	1670
AR09	2006 CH2M HILL	38.871783	-76.995172	10170
AR10	2006 CH2M HILL	38.87151	-76.9959437	645
AR11	2006 CH2M HILL	38.87197	-76.9959667	28710
AR12	1999 WA Navy Yard RI	38.871470	-76.996970	100
AR13	2006 CH2M HILL	38.87222	-76.9967964	48915
AR14	2006 CH2M HILL	38.872	-76.9978566	14568
AR15	2006 CH2M HILL	38.87249	-76.997822	23401
AR16	1999 WA Navy Yard RI	38.872360	-76.998470	122600
AR17	2006 CH2M HILL	38.87258	-76.9983521	68400
AR18	1999 WA Navy Yard RI	38.872320	-76.999440	2410
AR19	1995 Washington Navy Yard	38.872596	-77.000445	58210
AR20	1999 WA Navy Yard RI	38.881030	-76.971270	11790
AR21	1996 WA Gas - East Station Project	38.877126	-76.980504	12130
AR22	2006 CH2M HILL	38.87212	-76.9905851	10680
AR23	2006 CH2M HILL	38.87186	-76.9906543	8170
AR24	no data - across from SD60 (AR14)	38.869296	-76.996076	NA
AR25	no data - downstream reference	38.869877	-77.004342	NA

5.6.1 Sample Station Access

AECOM personnel collected surface sediment samples using a 21 ft boat equipped with a Ponar dredge, subcontracted with TG&B Marine Services, Inc. (TG&B). TG&B was responsible for mobilization and demobilization of the boat. Three AECOM personnel were on site for sediment sampling and processing. No utilities were needed to process the samples.

All sediment sampling and sample processing was conducted by AECOM. TG&B provided the sampling boat and crew to launch and operate the boat. The sampling boat was provided by Spencer Oceanographic Services, Inc. All personnel performing the fieldwork were certified for hazardous materials operations (OSHA 40-hour Hazwoper training, with up-to-date 8-hour refresher classes) and worked under the Site-specific Health and Safety Plan (HASP) provided in Appendix D.

An AECOM sediment sampling crew accessed the sediment sample locations using the boat and secured the sediment samples using procedures that have been used at other sites during previous sediment sampling events. The boat was anchored while the sample was collected. The vessel was mobilized in such a way as to minimize the potential for disturbance of the sediment and surface water via wave or propeller action.

5.6.2 Sample Station Locations

The original demonstration work plan indicated that 25 samples would be collected. However, sample collection was rapid at this site, thereby allowing the collection of 36 samples at no extra cost to the project. The 36 sediment sample locations are listed in Table 5-2. All sediment samples were surface grab samples. Sample locations were selected based on historical data (see Section 5.1 Baseline Characterization). Sample locations included those locations identified during previous sampling efforts as having the highest PAH concentrations, as well as locations expected to have lower and intermediate PAH concentrations. Field reference samples were collected as samples AR20 through AR25. The field reference samples were chosen in order to establish background levels in the river. Because the river is heavily industrialized it is not clear if the field reference samples represented true clean reference samples, but at a minimum they illustrated the PAH background concentrations present in the river surrounding the site.

Sample locations were designated coordinates of latitude and longitude. A differential GPS unit was used to position the sampling personnel at the sample station coordinates provided in Table 5-2, and daily station checks were conducted to confirm the unit was reporting accurately. No obstructions, such as boulders or cobbles, were encountered during sampling, and therefore no sample location changes were made in the field.

Table 5-2. Sediment Samples and Those Selected for Further Analysis.

Sample ID	Selected for Toxicity Testing	GPS Latitude[1]	GPS Longitude[1]	Water Depth (ft)	Field Description
AR01		38 52.4180	76 59.3067	8.6	Brown soft SILT, thick organic layer, leaves, twigs, snail shells
AR02	√	38 52.3876	76 59.3627	9.5	Dark brown silty CLAY, trace very fine grain sand, suspected black product, some gravel, moderate hydrocarbon-like odor, moderate sheen
AR03	√	38 52.3748	76 59.3851	4	Dark brown medium grained GRAVEL, little sand, trace large pebbles, visibly suspected product, slight hydrocarbon-like odor, trace sheen
AR04		38 52.3140	76 59.4671	8.4	Brown SILT, trace organics, slight hydrocarbon-like odor
AR05	√	38 52.2674	76 59.5119	11.5	Brown SILT, organic layer, leaves, twigs
AR06		38 52.2365	76 59.5727	13	Brown SILT, some organics, leaves, twigs, slight hydrocarbon-like odor, trace sheen
AR07	√	38 52.2632	76 59.6514	13.6	Brown SILT, organic layer, moderate hydrocarbon-like odor
AR08	√	38 52.2834	76 59.7122	15.4	Brown SILT, trace organics, snail shells, slight hydrocarbon-like odor
AR09		38 52.2961	76 59.7036	16	Brown SILT, dense organic layer, leaves, sticks, twigs, visible black product/staining, slight hydrocarbon-like odor
AR10	√	38 52.2905	76 59.7567	17.5	Brown SILT, organic layer-leaves, sticks, visibly black staining, slight hydrocarbon-like odor
AR11		38 52.3183	76 59.7587	13.7	Brown SILT, organic layer-leaves, sticks
AR12		38 52.2905	76 59.8303	19.8	Brown SILT, organic material-leaves, sticks, slight hydrocarbon-like odor
AR13	√	38 52.3336	76 59.8076	13.4	Dark brown SILT, heavy organic debris, twigs, leaves, shells
AR14	√	38 52.3201	76 59.8713	17.6	Dark brown SILT, some organic debris, twigs, leaves, shells, trace suspected black product by org material, slight hydrocarbon-like odor
AR15		38 52.3486	76 59.8691	13.8	Dark brown silty CLAY, moderate organic matter-sticks, some trash-plastic, trace suspected product, slight hydrocarbon-like odor
AR16	√	38 52.3316	76 59.9360	14	Dark brown silty CLAY, heavy organic matter-sticks, leaves, some suspected product
AR17	√	38 52.3636	76 59.9417	15.1	Brown SILT, some organic matter-leaves, twigs, snail shells, plastic, suspected trace product/black staining, slight hydrocarbon-like odor
AR18		38 52.3411	76 59.9679	19.3	Brown SILT and CLAY, trace organics, slight hydrocarbon-like odor
AR19		38 52.3566	77 00.0281	19.5	Dark brown SILT, some suspected product
AR20		38 52.8016	76 58.2947	9.2	Brown SILT, dense organic layer-leaves, twigs
AR21	√	38 52.6276	76 58.8315	8.7	Brown SILT, dense organic layer-peat, leaves, twigs, shells, trace black staining, slight hydrocarbon-like odor
AR22		38 52.3260	76 59.4362	10	Brown very fine SILT, many organics-sticks, leaves, some trash
AR23		38 52.3102	76 59.4329	12.5	Brown SILT, trace organics-leaves, roots
AR24		38 52.1553	76 59.7653	18.7	Brown SILT, trace organics-leaves, sticks, black staining, slight hydrocarbon-like odor
AR25	√	38 52.1930	77 00.2612	22.2	Brown SILT, trace organic material, some suspected black product, slight hydrocarbon-like odor
AR26	√	38 51.3340	76 59.9135	16.8	Brown coarse SAND, some silt, fine pebbles, dense organics sticks, leaves, cobbles, suspected black product, slight hydrocarbon-like odor, trace sheen
AR27	√	38 52.6513	76 59.5209	5.5	Brown SILT, little fine grain sand, some organic material-leaves, sticks
AR28		38 52.4841	76 59.1255	10.9	Brown SILT, organic layer-leaves, twigs
AR29		38 52.4515	76 59.2358	11.6	Brown SILT, dense organic matter-leaves, sticks, shells
AR30		38 52.3579	76 59.2817	9.4	Brown silty CLAY, trace organics-leaves, twigs, slight hydrocarbon-like odor
AR31		38 52.3467	76 59.3343	13.7	Brown SILT, trace very fine grain sand
AR32		38 52.2429	76 59.8043	17.5	Brown SILT, trace black staining/suspected product, slight hydrocarbon-like odor
AR33		38 52.2709	76 59.8772	18	Brown SILT, organic layer-leaves, twigs, slight hydrocarbon-like odor
AR34		38 52.2773	76 59.9542	18.1	Brown SILT, slight hydrocarbon-like odor
AR35		38 52.3484	77 00.0015	18.8	Brown SILT
AR36	√	38 52.3416	77 00.0925	18	Brown SILT, trace organics, slight hydrocarbon-like odor

[1] GPS latitude and longitude are in NAD 1983 degrees, decimal-minutes

5.6.3 Field Measurements

Upon arrival at each sampling station, field measurements of water quality and water depth were collected. Water depth was recorded by measuring the depth-to-sediment from the surface of the water, with an accuracy of 0.1 feet. Two sets of field measurements of water quality collected at each station. One measurement was taken near the water surface, approximately 1 foot below the water surface, and a second measurement was taken within 3 feet of the top of the sediment surface. The water quality parameters measured in the field included the following:

- Temperature (degrees Celsius)
- Dissolved Oxygen (mg/L)
- pH
- Salinity (ppt)
- Turbidity (NTU)
- Conductivity (µmhos/cm)

5.6.4 Sediment Sample Collection for Chemistry and Toxicity Testing

Approximately 12 liters of surface sediment, obtained from 0 to 10 centimeters below the sediment surface, were collected from each sample station. A ponar grab sampler (standard size) was used for collecting sediments. The ponar grab sampler is suitable for collecting soft sediments and also for harder sediments containing significant quantities of sand, gravel, and firm clay. The sample depth for this device is from 0 to 10 centimeters. The ponar grab sampler was lowered and raised from the sediment surface via a hand-winch and cable. The ponar size was 0.093 m^2.

Ponar samples were visually inspected to ensure that the following conditions were satisfied:

- The sampler was not overfilled so that the sediment surface was touching the top of the sampler
- Overlying water was present (indicates minimal leakage)
- The overlying water was clear or not excessively turbid
- The sediment-water interface was intact and relatively flat, with no sign of channeling or sample washout
- The desired depth of penetration was achieved
- There was no evidence of sediment loss (incomplete closure of the sampler, penetration at an angle, or tilting upon retrieval)

Care was exercised during sample collection to avoid problems such as loss of fine-grained surface sediment from sample washout upon ascent of the sampler. After the sample was collected, the sampling device was lifted slowly off the bottom, then steadily raised to the surface at a speed of approximately not more than 1 foot per second.

5.6.5 Field Observations

Field personnel recorded the sample station designation as well as observations of the physical characteristics of the sediment that were encountered at each sampling station, and the physical characteristics of the study area. Information that was recorded included:

- Presence of fill material, coal or coke, or asphalt- or tar-like materials at the shoreline
- Presence or absence of aquatic vegetation

- Sediment color, texture, and particle size
- Odor and presence of sheens or non-aqueous phase liquids (NAPL)

5.6.6 Sample Location Mapping

After each sample was collected, the station was located so that the site could be re-sampled in the future, if necessary. The mapping included the following:

- Recording of the latitude and longitude identified using a GPS receiver
- A hand-plotted estimate of the location on a field map of the study area
- Photo documentation of the significant features on the shore as location references
- Addition of notes to the field map showing any important site features that had a bearing on the sediment condition (e.g. pipe outfalls or subsurface debris)

5.6.7 Sample Processing Procedures

Sediments collected at each station were placed into a clean, 5-gallon plastic bucket. The bucket was labeled with the sample station designation and transported to shore. The sample was homogenized in the field by screening and mixing so that there was consistency in the sub-samples that were sent to each laboratory.

Sample homogenization was conducted as follows:

- Sediments in the bucket were first mixed by hand using a chemically inert, stainless steel spoon or spatula;
- The sediment sample was screened to remove oversized material. Materials such as twigs, shells, leaves, stones, pieces of wood, and vegetation were removed by hand and the sediment was press sieved through a #5 mesh sieve (4 mm openings) with a spatula;, and
- The sediment sample was then homogenized using an electric drill mounted mixing paddle. The paddle was used to thoroughly mix the sediment in the bucket.

Following homogenization, samples were split and placed into glass jars having Teflon-lined lids for submittal to the appropriate laboratories. Each jar was properly labeled with the name of the study site, the sample station designation, the time of collection, the date of collection, and name of collector. Following sample homogenization, glass jars were kept at 4ºC. Sample handling requirements for each testing laboratory are presented in Table 5-3.

Table 5-3. Sample Handling Requirements.

Sample Type	Matrix	Parameter	Qty	Container Type[1]	Minimum Volume	Preservation[3]	Holding Time from Sample Date	Laboratory[4]
Toxicity Tests	Sediment	Amphipod Toxicity (*H. azteca*) 28-day chronic test	TBD	glass	2 L	Cool to 4° C	28 days[2]	ERDC
Chemical/ Physical Characterization	Pore Water	Dissolved pore water PAHs (parent compounds and estimate of alkylated compounds)	TBD	(5)	(5)	Cool to 4° C	28 days[2]	EERC
	Sediment	Total Organic Carbon	TBD	glass	8 oz.		28 days[2]	
		Soot Carbon	TBD				28 days[2]	
		Total sediment PAHs: Parent & alkylated (34 compounds)	TBD				28 days[2]	
		Percent Solids	TBD	glass	2 oz.	Cool to 4° C	28 days[2]	STL
		Grain Size	TBD	glass	16 oz.		NA	
		pH	TBD	glass	8 oz.	Cool to 4° C	ASAP	
		Ammonia	TBD				28 days[2]	
Surface Water	Surface Water	pH, Temperature, Conductivity, Salinity, DO, Turbidity	TBD	field	field	field	15 min.	Field

Notes: (1) All glass jars had Teflon-lined lids
(2) Test were initiated within 28 days of sample collection
(3) Samples requiring thermal preservation were maintained at 2° - 6° C.
(4) EERC-Energy & Environmental Research Center
(5) ERDC – Engineering Research & Development Center
(6) STL Severn Trent Laboratories (now Test America)
(7) Pore water samples were generated in the EERC laboratory from the sediment samples

5.6.8 Field and Sample Documentation

This section presents the documentation procedures that were used during sample collection and handling, including field documentation, a description of the designations that were used for the samples, chain-of-custody (COC) procedures, and equipment documentation.

Field Documentation. All measurements and observations discussed above for both the field sampling and field processing of the sediments were recorded on a field form. Copies of the field forms are included in the final project report as Appendix E.

Sample Designations. Individual sediment samples were designated as follows:

- **Sediment Samples.** Sediment samples were numbered according to the numbering system outlined in Table 5-2; and
- **Background Sediment Samples.** Background sediment samples were distinguished only by their sampling location.

Sample COC (Chain of Custody). To establish the documentation necessary to trace the sample possession from the time of sample collection to final analysis, a COC record was completed for each sample, and is included as Appendix F. The COC record contains the following information:

- Sampling station identification
- Sampling date and time
- Identification of sample collector
- Sample identification
- Sample description (type and quantity)
- Analyses to be performed
- Signatures of persons involved in the chain of possession
- Date and times of possession

Field Quality Assessment and Quality Control (QA/QC) Procedures. All QA/QC procedures are defined in the project-specific Quality Assurance Project Plan (QAPP), which is attached as Appendix G.

Equipment Decontamination

Sediment processing and decontamination of equipment was performed on shore, at the Buzzard's Point Marina, with permission of the U.S. Park Service. All equipment used to collect or process the sediment samples was decontaminated between uses. The decontamination consisted of brushing/wiping sediment from the equipment, cleaning with Alconox, followed by a site-water rinse. Since NAPLs were not encountered, an acetone cleaning was not required.

5.6.9 Sediment Sample Analysis

Table 5-4 provides a summary of the chemical, physical, and bioavailability tests that were performed as part of the initial sample screening and detailed characterization. Table 5-5 provides method references for the tests and the laboratories that conducting the analyses. Chemical/physical tests that were completed for the sediment samples are described in the remainder of this section. A project-specific QAPP, which describes QA/QC procedures for the laboratory analyses, is included as Appendix G. All analytical/testing laboratories which were

used in the demonstration were included as performers in the project.

Table 5-4. Analytical Testing Program.

Sample Type	Matrix	Parameter	Purpose	Laboratory
Bioavailability Characterization	Sediment	Amphipod toxicity (*H. azteca*) 28-day chronic test	Direct Measurement of PAH toxicity	ERDC
Chemical/Physical Characterization	Sediment	Total and Soot Organic Carbon (SOC)	Characterization of sediment organic matter	EERC
		Total Solids	Potential confounding factors affecting interpretation of toxicity data	STL
		Grain size		STL
		pH		STL
		Ammonia		STL
		Characterization of Sediment Carbon		UMBC
	Pore Water	SPME total dissolved PAHs (parent compounds and estimate of alkylated)	Chemical test for estimating PAH bioavailability	EERC
Field Measurement	Surface Water	Temperature	General environmental quality parameters	Field YSI 6820 Field Meter or equivalent
		pH		
		Dissolved oxygen		
		Conductivity		
		Salinity		
		Turbidity		
EERC - Energy & Environmental Research Center - University of North Dakota ERDC - Engineering Research and Development Center STL - Severn Trent Laboratories, Inc., Burlington, VT (now Test America) UMBC -University of Maryland – Baltimore County				

Table 5-5. Laboratory Methods.

Parameter	Method	Method Reference(s)	Laboratory
Total PAH extraction and analysis – screening level	Sonication for 4 hrs. in 1:1 methylene chloride:acetone mixture followed by GC/MS analysis.	[Hawthorne, 2005b], [U.S. EPA EPA/600/R02/013, 2003] , [NOAA, 1998]	EERC
Total PAHs parent & alkylated (34 compounds)	Sohxlet extraction in methylene chloride: followed by GC/MS using Selected Ion Monitoring (SIM) for measuring parent and alkylated PAHs	[Hawthorne, 2005b], [U.S. EPA EPA/600/R02/013, 2003] , [[NOAA, 1998]	EERC
PAH extraction – pore water	Centrifugation and flocculation followed by solid phase microextraction (SPME) and GC/MS analysis for measuring parent and alkylated PAHs	[Hawthorne et al., 2005a], [Hawthorne, 2005b], [NOAA, 1998]	EERC
Total Organic Carbon (TOC)	Sample acidified to remove carbonates, followed by analysis using a Leeman CE44 Elemental Analyzer modified for sediment analysis. (triplicate analyses on sediments)	[Hawthorne, 2001]	EERC
Soot Organic Carbon (SOC)	TOC following pretreatment at 375º C for 24 hrs. [i.e., heat stable TOC]	[Gustafsson et al., 1997]	EERC
Percent Solids	STL SOP IN623	[U.S. EPA, 1983], [U.S. EPA, CLP SOW OLM04.2/4.3], [U.S. EPA, CLP SOW ILM05.2]	STL
Grain Size	ASTM Methods D422-63 and D421-85	[ASTM, 1990]	STL
pH in Sediment	U.S. EPA SW-846 Method 9045C	[U.S. EPA, 1996]	STL
Ammonia	U.S. EPA Method 350.2	[U.S. EPA, 1983]	STL
Sediment toxicity test	Amphipod (*H. azteca*) toxicity 28-day chronic test	[U.S. EPA, 2000]	ERDC

EERC – Energy & Environmental Research Center, ND
STL – Severn Trent Laboratories, Inc., Burlington, VT (now Test America)
ERDC – Engineering Research and Development Center, Vicksburg, MS

5.6.10 Sediment Characterization

Fifteen of the original 36 sediment samples were selected for further characterization based on

preliminary total PAH analysis. Details regarding the specific methods that were used to generate these data are and are discussed below.

Carbon and Grain Size Analysis of Sediments. Total organic carbon (TOC) and soot carbon (SOC) were determined on air-dried sediment samples using a Leeman Labs model CE440 15 elemental analyzer as described in the literature (Gustafsson et al., 1997; Accardi-Dey and Gschwend, 2002). Total solids and grain size were determined using EPA Method 160.3 and ASTM Method D-422-63/D421-85, respectively (U.S. EPA, 1983; ASTM, 2005).

Analysis of 18 Parent and 16 Alkylated PAHs (NOAA 34 PAHs) in Sediment and Sediment Pore water. Preliminary estimates of PAH concentrations on each of the original 36 sediment samples were performed by mixing 2 g of the wet sediment with 2 g of sodium sulfate and extracting with 20 mL of 1:1 acetone/methylene chloride for 18 h in a bath sonicator, and analyzing the extracts for PAH_{34}. Total PAHs (PAH_{34}), consisting of 34 parent and alkylated compounds, were analyzed in all sediment and pore water samples using GC/MS with selected ion monitoring (SIM). The analysis of parent and alkylated PAHs was conducted using the method originally developed by the National Oceanographic and Atmospheric Administration (NOAA) [NOAA, 1998] with additional response factors developed for quantification of alkylated PAHs [Hawthorne et al., 2005b]. All standard and sample peak areas were normalized to the d-PAH internal standards ranging in size from naphthalene-d8 to benzo[ghi]perylene-d12. When no deuterated analog of a PAH was available, the d-PAH with the closest molecular structure was used (e.g., benzo[a]pyrene-d12 was used as the internal standard for benzo[e] pyrene). The parent d-PAH was used for the related alkyl PAHs (except for methylnaphthalene-d10). Quantification was based on the peak areas of the molecular ions (compared to those of the relevant d-PAH internal standards) and the relative response factors as previously reported (Hawthorne et al., 2005; Hawthorne et al., 2006). Peak identities were routinely verified by analyzing representative extracts in the full scan GC/MS mode.

Pore Water PAHs in Sediment. Pore water samples were prepared fresh daily by transferring a 20 ml to 40 mL of the sediment/water slurry to a "certified clean" 40 mL glass "VOA" vial and centrifuging for 30 min at 1000*g*. This typically resulted in 10-15 mL of pore water that could be
gently collected with a pipette. Flocculation of the water samples was performed twice with a 10 wt % solution of alum (aluminum potassium sulfate) added to the water at a 1:40 ratio. A few drops of 1 M NaOH was added to the vials, after which it was mixed to induce flocculation. The vial was centrifuged again for 30 min and the supernatant water was collected with a pipette. The pore water sample was then split into four 1.5 mL aliquots that were placed into new 2-mL silanized glass autosampler vials (Agilent, Wilmington, DE) containing a precleaned (sonicated overnight in acetone) 7-mm Teflon coated stir bar. The *d*-PAH internal standards were immediately added to the sample. The samples were then subjected to SPME analysis within a few minutes of preparation to ca. 4 h after preparation (for the fourth replicate sample). Daily blank and calibration water samples were prepared in the same manner with 1.5 mL of HPLC-grade water (Fisher Scientific, Pittsburgh, PA).

PAH concentrations in pore water samples were determined following solid-phase microextraction (SPME) using an Agilent model 5973 GC/MS equipped with a 60-m Agilent HP-5 MS column (0.25 µm film thickness, 250 µm i.d.) operated in the selected ion mode for the molecular ions of the target PAHs and d-PAHs which were added to the pore water samples as

internal standards (Hawthorne et al., 2005).

Aquatic Toxicity Testing

PAH toxicity was determined directly by measuring survival and growth of the freshwater amphipod *H. azteca*[1]. Amphipod survival and growth was assessed using the standard 28-day chronic toxicity test. Following the 28-d sediment exposure period, the surviving amphipods were characterized. Comparisons of survival and dry weights among treatments were conducted using statistical techniques, i.e., arc sine (square root) transformed values subjected to either the parametric Dunnett's Test or non-parametric Many-One Rank Test procedures of Steel. All statistical comparisons were made at a 95% confidence level ($p < 0.05$).

Characterization of Sediment Carbon

Four sediment samples were selected to perform a detailed particle-scale assessment of PAH location and binding. These samples were selected to span the range of low to high contaminant availability as measured by the SPME analysis of the sediment pore water. The detailed assessment of contaminant association included size and density separation of particulate organic matter in sediments, particle-scale PAH measurements, and petrographic characterizations to identify the nature of the organic matter responsible for PAH binding. Identification of the geochemical nature of the sediment carbon, specifically the presence of coal, coke, lampblack, pitch, and tar help to explain any differences in site-specific values of PAH partitioning from what would have been estimated using standard empirical correlations. The proposed methods to perform this assessment were conducted as part of other ESTCP ([ESTCP ER-0510] Sediment project at Hunter's Point) and SERDP ([SERDP CU-1095] – Assessment and Prediction of Biostabilization of Polycyclic Aromatic Hydrocarbons (PAHs) in Sediments) projects and are described in the literature (Ghosh et al., 2000).

General Chemistry, Physical Analysis Methods, and Field Measurements

Analysis of sediment grain size, pH, and ammonia was conducted on samples subjected to toxicity testing. Field measurements of surface water parameters were also be made at the time of sediment sampling. These parameters include temperature, pH, dissolved oxygen, conductivity, salinity and turbidity. These data were collected using a Field YSI 6280 field meter or its equivalent. These data provide measures of general environmental quality, which can be useful when interpreting the chemistry and toxicology test results.

5.6.11 Data Interpretation

Toxic units (TU) were calculated for each individual PAH in the sediment pore water according to the method presented in EPA (2003). The individual TUs for each PAH were summed to form a total PAH TU.

Probit and Logistic Regression Analysis. One of the performance objects of the demonstration was the ability of the PAH TU_{34}s, as calculated from the measured SPME pore water concentrations, to predict toxicity to the amphipod *H. azteca*. A dose-response relationship relating the survival of *H. azteca* versus the sample total PAH TU was evaluated on the existing SCBA database using probit and logistic regression analysis. The Probit model goodness-of-fit was evaluated on the existing SCBA database using the p-values for the Pearson and likelihood ratio chi-square at the 95% confidence level. The lower 95th tolerance level for 90% survival and

[1] Reproduction was not examined as a bioassay endpoint because of the degree of variability associated with this test and the inability to effectively use the data to make any sediment management decisions.

upper 95th tolerance level for 10% survival were calculated to quantify predictions of toxicity (i.e., no toxicity, uncertain or potential toxicity, and likely toxicity). The relationship between the PAH pore water concentrations and growth from the SCBA database was also analyzed using regression analysis. The survival data for the WNY sediments was plotted against the following parameters:

- Bulk sediment PAH_{16} concentration
- Estimated PAH_{34} pore water concentration (expressed at TU_{34}) using equilibrium partitioning theory and adjustment for total organic carbon (TOC)
- Estimated PAH pore water concentration (expressed as TU_{34}) using equilibrium partitioning theory and adjustment for total organic carbon (TOC) and soot organic carbon (SOC)
- Pore water PAH_{34} concentrations (expressed at TU_{34}) determined by SPME analysis

In addition, *H. azteca* growth data were plotted against whole sediment PAH_{16} concentrations and SPME pore water PAH_{34} concentrations (expressed as TU_{34}). To aid these data comparison, they were superimposed on the historical SCBA data set to investigate conformity to the dose-response relationship that had been previously determined using the entire industry field database.

5.6.12 Experimental Controls

Experimental controls consisted of the collection of six field reference site sediment samples, of which two were used in the final data analysis (i.e., final 15 samples). The field reference areas were selected based on the results of historical sediment sampling and location upstream, cross stream, or downstream from the site did not contain significant quantities of target analytes.

5.7 SAMPLING RESULTS

This section of the document presents the findings of the demonstration. These results are discussed in the following order:

- General sediment physical and chemical properties: grain size, TOC, SOC, ammonia, and pH
- Bulk sediment PAH chemistry
- Pore water PAH chemistry
- Aquatic toxicity results: Survival and growth of *Hyalella azteca* (28-day test)
- Total organic carbon (TOC) and soot organic carbon (SOC)
- Carbon chemistry: detailed carbon chemistry of selected sediments

The measures of PAHs in sediments were correlated with the aquatic toxicity results to provide a weight-of-evidence analysis of the effects of sediment PAHs on the benthic community. The detailed carbon analysis explored the effect of sediment carbon type on pore water PAH chemistry (i.e., bioavailability).

Thirty-six samples locations were originally sampled. These samples were subjected to a rapid total PAH analysis (extraction by 16 hour sonication). The results of that analysis were used to select 15 sediment samples (including 2 reference samples) containing a range of total PAH concentrations (Table 5-2). The following sections discuss the chemical and physical analysis of the 15 final sediment samples selected for study.

5.7.1 General Sediment Physical and Chemical Properties

Sediment grain size was generally silty-clay, with the exception of samples AR03, AR16, and AR26, which were predominantly sandy (Table 5-6). pH values ranged from6.9 to 7.2 standard units, and ammonia ranged from 37.1 to 595 mg/kg. Sample AR03 had a relatively low ammonia concentration (37.1 mg/kg) which reflects the sandier nature of this sediment sample. The complete laboratory report for the general sediment physical and chemical properties is provided in Appendix H.

Table 5-6. Sediment Grain Size and Basic Chemistry.

Sample	Grain Size Data							Basic Chemistry	
	Gravel (%)	Coarse Sand (%)	Medium Sand (%)	Fine Sand (%)	Silt (%)	Clay (%)	Solids (%)	Ammonia (mg/Kg)	pH
AR02	4.9	6.5	9.6	12.5	22.9	43.7	48.5	225	7.1
AR03	7.7	17.4	47.0	21.2	5.2	1.4	80.8	37.1	7.2
AR05	0.1	0.5	1.7	30.3	49.6	17.9	47.6	303	6.9
AR07	0.0	0.0	0.2	4.2	69.6	26.0	39.6	309	6.9
AR08	0.0	0.0	0.1	0.4	66.9	32.6	37.6	315	6.9
AR10	0.0	0.1	0.1	0.7	59.1	40.0	38.7	335	6.9
AR13	20.2	6.7	12.0	14.5	31.2	15.5	33.8	595	6.5
AR14	0.3	0.3	0.3	1.9	51.1	46.0	35.4	271	6.8
AR16	3.5	6.1	13.4	44.7	21.4	10.9	43.1	136	6.9
AR17	19.7	9.0	8.4	14.3	25.8	22.8	34.4	161	7.1
AR21	3.6	1.2	1.3	6.0	60.3	27.6	27.2	431	6.9
AR25	0.0	0.0	0.4	1.8	61.9	35.9	38.4	408	6.9
AR26	14.9	9.6	19.2	42.3	8.1	6.0	48.7	123	7.1
AR27	0.1	0.4	1.2	27.6	52.3	18.4	48.1	182	6.9
AR36	0.0	0.0	0.1	2.7	71.0	26.2	36.9	325	7.0

5.7.2 PAH Chemistry

The 15 samples selected for detailed study were analyzed by EERC for the total extractable and pore water concentrations of the 34 National Oceanographic and Atmosphere Administration (NOAA) PAHs, which include 18 parent and 16 groups of alkylated PAHs, subsequently referred to as PAH_{34}. The 16 U.S. EPA priority pollutant PAHs (PAH_{16}) are a subset of the PAH_{34}. The complete laboratory report from EERC is provided in Appendix I, and the PAH chemistry is discussed in the following sub-sections. A summary of the total bulk sediment PAH concentrations as well as the SPME PAH pore water concentrations is provided in Table 5-9, along with the results of the aquatic toxicity testing.

5.7.2.1 Bulk PAH Chemistry

Total PAH_{16} and PAH_{34} concentrations are presented in Table 5-7. $TPAH_{16}$ concentrations ranged from 14.4 to 600 mg/kg in these samples. The two field reference samples (AR21 and AR25) had PAH_{16} concentrations of 38.5 and 21.9 mg/kg, respectively. Total PAH_{34}

concentrations in the bulk sediment samples ranged from 38.9 to 2009 mg/kg. All of these sediment samples exceed the freshwater threshold effects level of 1.6 mg PAH/kg sediment. The DC Department of the Environment (DDOE) currently uses the TEL as a TPAH screening concentration. According to that criteria, the area covered by these sediment samples would have to be remediated or subjected to further (i.e., higher tier) analysis. Many other U.S. states use the freshwater probable effects level (PEL) of 22.8 mg PAH/kg sediment as a screening level. However, it can be seen that almost all of the sediments within the areas of the WNY in this study also exceed that screening level.

5.7.2.2 Pore water PAH Chemistry

Pore water PAH_{34} concentrations are presented in Table 5-8. Concentrations of PAH_{16} in pore water ranged from 0.078 (AR16) to 114 ug/L (AR02). Concentrations of PAH_{34} in pore water ranged from 0.078 (AR16) to 677 ug/L (AR02). Pore water PAH concentrations are related to observed effects on benthic organisms through a final chronic value (FCV), which is derived from an Ambient Water Quality Criteria (AWQC) value (EPA, 2003). To make this comparison, pore water PAH concentrations are converted to toxic units (TU), where the TU is the individual PAH concentration/individual PAH FCV. The individual PAH TUs are then summed to provide a total PAH TU (i.e., PAH_{16} TU or PAH_{34} TU). The EPA recognized the contribution of alkylated PAHs to toxicity to benthic organisms, and recently provided evidence that TU PAH_{34} should be used rather than TU PAH_{16} (U.S. EPA, 2009) due to the low FCV (final chronic value) associated with the alkylated PAHs..

Table 5-7. Sediment Total PAH Concentrations and TUs Based on Equilibrium Partitioning.

	AR02			AR03			AR05			AR07			AR08		
Sediment PAHs (mg/kg)	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**
naphthalene	12.4	0.680	4	0.726	0.535	4	0.221	0.017	4	0.125	0.014	4	0.142	0.015	4
2-methylnaphthalene	11.6	0.784	4	0.935	0.741	4	0.201	0.007	4	0.135	0.005	4	0.132	0.010	4
1-methylnaphthalene	5.58	0.156	4	1.66	0.808	4	0.109	0.023	4	0.071	0.008	4	0.066	0.005	4
C2 naphthalenes	137	1.84	4	3.29	1.15	4	1.38	0.268	4	1.07	0.067	4	1.10	0.062	4
C3 naphthalenes	98.2	2.36	4	1.60	0.491	4	1.19	0.045	4	1.46	0.063	4	0.933	0.073	4
C4 naphthalenes	57.0	1.72	4	0.743	0.286	4	1.39	0.171	4	1.84	0.096	4	1.09	0.125	4
acenaphthylene	17.1	1.67	4	0.275	0.220	4	0.317	0.059	4	0.268	0.034	4	0.329	0.035	4
acenaphthene	48.5	2.21	4	1.10	0.333	4	0.266	0.078	4	0.168	0.011	4	0.146	0.013	4
fluorene	23.5	0.688	4	0.550	0.079	4	0.322	0.069	4	0.211	0.014	4	0.169	0.007	4
C1 fluorenes	48.7	1.08	4	0.823	0.343	4	0.674	0.110	4	0.555	0.011	4	0.525	0.024	4
C2 fluorenes	72.2	4.21	4	1.50	0.713	4	2.87	0.279	4	2.65	0.183	4	2.46	0.172	4
C3 fluorenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
phenanthrene	99.7	1.31	4	2.22	0.952	4	2.71	0.908	4	1.84	0.152	4	1.51	0.059	4
anthracene	58.1	2.25	4	1.66	0.705	4	1.61	0.539	4	0.904	0.071	4	0.863	0.041	4
C1 phenanthrenes/anthracenes	148	5.95	4	2.52	1.09	4	2.36	0.489	4	1.74	0.087	4	1.36	0.047	4
C2 phenanthrenes/anthracenes	299	17.3	4	3.02	1.66	4	5.02	0.530	4	7.01	0.397	4	5.29	0.139	4
C3 phenanthrenes/anthracenes	152	8.78	4	2.75	1.45	4	7.61	0.835	4	7.30	0.322	4	5.97	0.054	4
C4 phenanthrenes/anthracenes	28.8	4.65	4	0.595	0.321	4	2.42	0.324	4	2.00	0.050	4	1.93	0.252	4
fluoranthene	57.0	0.825	4	1.68	1.58	4	5.15	1.83	4	3.61	0.218	4	2.95	0.207	4
pyrene	92.9	1.77	4	1.77	1.21	4	4.27	1.42	4	3.13	0.185	4	2.62	0.153	4
C1 fluoranthenes/pyrenes	121	4.76	4	1.66	0.852	4	3.82	0.912	4	2.56	0.113	4	2.32	0.051	4
benz[a]anthracene	29.8	0.506	4	0.663	0.522	4	1.91	0.637	4	1.31	0.085	4	1.16	0.076	4
chrysene	34.2	1.13	4	1.19	0.765	3	3.74	0.933	4	2.50	0.102	4	2.48	0.329	4
C1 chrysenes	98.2	3.00	4	2.61	1.18	4	6.84	1.30	4	4.23	0.183	4	4.01	0.315	4
C2 chrysenes	80.6	4.42	4	ND	ND	ND	6.78	0.688	4	4.26	0.114	4	4.46	0.262	4
C3 chrysenes	31.4	3.78	4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C4 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[b+k]fluoranthene	27.8	0.925	4	1.25	0.872	4	6.45	1.41	4	3.00	0.084	4	3.03	0.342	4
benzo[e]pyrene	13.1	0.693	4	0.617	0.337	4	2.18	0.438	4	1.14	0.036	4	1.18	0.156	4
benzo[a]pyrene	37.7	1.92	4	0.872	0.568	4	3.97	1.08	4	1.91	0.093	4	1.82	0.140	4
perylene	7.66	0.610	4	0.208	0.140	4	0.954	0.232	4	0.843	0.045	4	0.857	0.080	4
indeno[1,2,3-cd]pyrene	29.9	0.876	4	0.295	0.246	4	2.53	0.647	4	2.60	0.076	4	2.76	0.113	4
dibenz[ah]anthracene	7.16	0.425	4	ND	ND	ND	ND	ND	ND	0.508	ND	1	ND	ND	ND
benzo[ghi]perylene	24.1	0.852	4	0.424	0.265	4	2.36	0.547	4	1.74	0.050	4	1.80	0.125	4
Total PAH_{16} (mg/kg)	600	9.56	4	14.4	7.42	4	35.8	10.0	4	23.4	1.03	4	21.8	1.40	4
Total PAH_{34} (mg/kg)	2009	55.1	4	38.9	12.9	4	81.6	15.0	4	62.3	2.19	4	55.5	2.50	4
Ratio PAH_{16}/PAH_{34}	0.30	0.01	4	0.37	0.13	4	0.43	0.05	4	0.38	0.01	4	0.39	0.01	4
Toxic Units (TU_{34})	31	0.8	4	6.8	2.2	4	3.2	0.6	4	2.1	0.1	4	2.0	0.09	4

ND - Non detected

Table 5-7. Sediment Total PAH Concentrations and TUs Based on Equilibrium Partitioning (continued).

	AR10			AR13			AR14			AR16			AR17		
Sediment PAHs (mg/kg)	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**
naphthalene	0.176	0.007	4	5.47	10.7	4	0.177	0.022	4	0.289	0.101	4	0.196	0.023	4
2-methylnaphthalene	0.154	0.007	4	2.45	4.67	4	0.151	0.006	4	0.236	0.075	4	0.172	0.009	4
1-methylnaphthalene	0.083	0.007	4	1.42	2.72	4	0.073	0.010	4	0.169	0.083	4	0.100	0.003	4
C2 naphthalenes	1.55	0.145	4	4.90	7.64	4	1.51	0.045	4	1.72	0.365	4	1.56	0.139	4
C3 naphthalenes	0.751	0.091	4	2.71	3.93	4	0.664	0.047	4	1.05	0.324	4	1.24	0.314	4
C4 naphthalenes	1.25	0.117	4	2.05	1.78	4	1.01	0.079	4	1.50	0.479	4	1.49	0.625	4
acenaphthylene	0.400	0.030	4	0.657	0.607	4	0.539	0.193	4	0.348	0.042	4	0.281	0.022	4
acenaphthene	0.163	0.009	4	15.1	29.7	4	0.162	0.008	4	0.252	0.081	4	0.289	0.099	4
fluorene	0.240	0.007	4	8.63	16.5	4	0.233	0.009	4	0.400	0.120	4	0.345	0.115	4
C1 fluorenes	0.451	0.038	4	6.61	12.0	4	0.476	0.037	4	0.535	0.093	4	0.596	0.099	4
C2 fluorenes	ND	ND	ND	8.84	12.7	4	ND	ND	ND	ND	ND	ND	2.50	0.452	4
C3 fluorenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
phenanthrene	1.81	0.042	4	50.2	93.3	4	1.84	0.239	4	2.45	0.707	4	3.18	1.25	4
anthracene	1.20	0.093	4	32.1	60.3	4	1.66	0.450	4	1.63	0.413	4	1.75	0.699	4
C1 phenanthrenes/anthracenes	1.75	0.075	4	25.3	46.3	4	1.92	0.188	4	2.39	0.712	4	2.14	0.471	4
C2 phenanthrenes/anthracenes	4.22	0.369	4	24.3	40.1	4	4.60	0.580	4	5.68	1.42	4	6.83	0.958	4
C3 phenanthrenes/anthracenes	6.42	0.249	4	19.8	23.5	4	6.80	0.825	4	8.19	1.80	4	7.42	1.46	4
C4 phenanthrenes/anthracenes	2.35	0.358	4	3.85	3.89	4	1.93	0.251	4	2.65	0.588	4	2.53	0.623	4
fluoranthene	3.55	0.086	4	51.3	87.5	4	4.00	0.282	4	5.03	1.56	4	5.85	2.31	4
pyrene	3.20	0.063	4	42.7	73.4	4	4.56	1.96	4	4.46	1.30	4	5.20	1.89	4
C1 fluoranthenes/pyrenes	2.70	0.155	4	37.7	66.3	4	4.00	0.880	4	3.68	0.889	4	4.22	1.64	4
benz[a]anthracene	1.46	0.080	4	23.7	42.0	4	2.24	0.513	4	2.04	0.515	4	2.88	1.19	4
chrysene	2.93	0.153	4	29.4	48.6	4	4.22	0.778	4	3.61	0.754	4	4.47	1.52	4
C1 chrysenes	4.74	0.143	4	48.4	79.2	4	6.95	1.64	4	6.22	1.14	4	8.10	2.43	4
C2 chrysenes	4.89	0.202	4	33.3	50.2	4	6.44	0.865	4	6.38	1.41	4	8.12	2.48	4
C3 chrysenes	ND	ND	ND	38.7	ND	1	ND	ND	ND	ND	ND	ND	ND	ND	ND
C4 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[b+k]fluoranthene	3.86	0.036	4	42.5	67.0	4	5.26	0.953	4	4.60	0.825	4	5.58	1.46	4
benzo[e]pyrene	1.61	0.035	4	11.4	16.8	4	2.08	0.302	4	1.86	0.337	4	2.22	0.620	4
benzo[a]pyrene	2.45	0.046	4	33.0	54.6	4	3.50	0.676	4	3.02	0.634	4	3.77	0.966	4
perylene	1.20	0.031	4	6.70	10.9	4	1.43	0.177	4	1.22	0.186	4	1.42	0.292	4
indeno[1,2,3-cd]pyrene	2.88	0.194	4	28.1	47.9	4	3.86	0.399	4	3.22	0.804	4	3.61	0.875	4
dibenz[ah]anthracene	0.552	0.084	4	5.68	10.0	4	0.761	0.160	4	0.589	0.100	4	ND	ND	ND
benzo[ghi]perylene	2.06	0.065	4	16.8	26.5	4	2.54	0.230	4	2.29	0.450	4	2.55	0.637	4
Total PAH_{16} (mg/kg)	26.9	0.525	4	385	669	4	35.6	5.63	4	34.2	8.06	4	40.0	12.1	4
Total PAH_{34} (mg/kg)	61.0	1.37	4	635	1070	4	75.6	11.1	4	77.7	17.6	4	90.6	21.6	4
Ratio PAH_{16}/PAH_{34}	0.44	<0.1	4	0.54	0.06	4	0.47	0.01	4	0.44	0.01	4	0.44	0.04	4
Toxic Units (TU_{34})	2.2	0.05	4	18	30	4	2.5	0.35	4	1.5	0.35	4	3.4	0.79	4

ND - Non detected

Table 5-7. Sediment Total PAH Concentrations Based on Equilibrium Partitioning (continued).

	AR21			AR25			AR26			AR27			AR36		
Sediment PAHs (mg/kg)	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**	**Mean**	**Std Dev.**	**N**
naphthalene	0.186	0.014	4	0.153	0.010	4	0.585	0.169	4	0.113	0.006	4	0.209	0.033	4
2-methylnaphthalene	0.178	0.011	4	0.136	0.002	4	0.548	0.175	4	0.108	0.021	4	0.188	0.016	4
1-methylnaphthalene	0.102	0.010	4	0.087	0.005	4	0.455	0.164	4	0.063	0.009	4	0.093	0.006	4
C2 naphthalenes	1.52	0.174	4	1.34	0.156	4	2.56	0.655	4	0.882	0.138	4	1.57	0.121	4
C3 naphthalenes	1.72	0.133	4	0.714	0.069	4	2.13	0.429	4	0.716	0.109	4	1.09	0.137	4
C4 naphthalenes	1.94	0.097	4	0.979	0.147	4	1.97	0.393	4	0.826	0.141	4	1.23	0.046	4
acenaphthylene	0.351	0.048	4	0.293	0.033	4	0.477	0.192	4	0.308	0.117	4	0.312	0.009	4
acenaphthene	0.204	0.009	4	0.130	0.010	4	0.610	0.122	4	0.110	0.010	4	0.163	0.008	4
fluorene	0.315	0.004	4	0.176	0.007	4	0.989	0.135	4	0.168	0.015	4	0.238	0.012	4
C1 fluorenes	0.754	0.028	4	0.373	0.048	4	1.55	0.774	4	0.430	0.055	4	0.613	0.030	4
C2 fluorenes	3.37	0.435	4	ND	ND	ND	4.21	1.86	4	1.95	0.186	4	2.86	0.227	4
C3 fluorenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
phenanthrene	2.43	0.154	4	1.49	0.080	4	7.83	3.89	4	1.55	0.210	4	1.92	0.113	4
anthracene	1.46	0.090	4	0.873	0.058	4	2.93	1.29	4	0.901	0.057	4	1.20	0.041	4
C1 phenanthrenes/anthracenes	2.50	0.209	4	1.46	0.133	4	4.34	1.31	4	1.44	0.098	4	1.93	0.085	4
C2 phenanthrenes/anthracenes	6.26	0.403	4	3.67	0.257	4	11.3	2.78	4	3.32	0.496	4	4.57	0.316	4
C3 phenanthrenes/anthracenes	9.09	0.401	4	5.53	0.307	4	10.8	3.93	4	5.17	0.808	4	7.66	0.330	4
C4 phenanthrenes/anthracenes	2.69	0.353	4	1.98	0.453	4	2.67	0.638	4	1.59	0.348	4	2.73	0.385	4
fluoranthene	5.12	0.343	4	2.99	0.129	4	11.8	5.89	4	3.46	0.499	4	3.74	0.223	4
pyrene	4.41	0.323	4	2.69	0.103	4	9.32	4.56	4	2.95	0.351	4	3.35	0.156	4
C1 fluoranthenes/pyrenes	4.43	0.297	4	2.13	0.074	4	7.09	2.55	4	2.89	0.539	4	3.68	0.184	4
benz[a]anthracene	1.93	0.068	4	1.20	0.041	4	3.95	2.45	4	1.34	0.133	4	1.55	0.099	4
chrysene	3.94	0.127	4	2.26	0.036	4	8.21	4.46	4	2.61	0.198	4	3.00	0.161	4
C1 chrysenes	7.39	0.183	4	3.85	0.156	4	10.3	5.31	4	4.72	0.992	4	5.54	0.299	4
C2 chrysenes	7.41	0.222	4	4.31	0.281	4	8.31	3.10	4	4.77	1.26	4	6.48	0.278	4
C3 chrysenes	ND	ND	ND	ND	ND	ND	4.05	1.22	4	ND	ND	ND	ND	ND	ND
C4 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[b+k]fluoranthene	6.87	0.443	4	3.15	0.121	4	7.24	4.38	4	4.58	0.233	4	5.50	0.367	4
benzo[e]pyrene	2.10	0.129	4	1.28	0.050	4	2.56	1.46	4	1.42	0.069	4	1.63	0.106	4
benzo[a]pyrene	4.21	0.343	4	2.05	0.090	4	4.06	2.57	4	2.95	0.260	4	3.41	0.261	4
perylene	1.04	0.054	4	1.15	0.054	4	1.12	0.649	4	0.644	0.033	4	1.01	0.034	4
indeno[1,2,3-cd]pyrene	3.73	0.240	4	2.40	0.066	4	3.90	2.35	4	2.32	0.194	4	3.11	0.151	4
dibenz[ah]anthracene	0.624	0.093	4	0.408	0.024	4	0.968	0.618	4	0.431	0.048	4	0.528	0.067	4
benzo[ghi]perylene	2.76	0.114	4	1.68	0.088	4	2.54	1.50	4	1.92	0.232	4	2.23	0.131	4
Total PAH_{16} (mg/kg)	38.5	2.19	4	21.9	0.775	4	65.4	33.6	4	25.7	1.78	4	30.5	1.68	4
Total PAH_{34} (mg/kg)	91.0	4.68	4	50.9	2.37	4	141	57.2	4	56.7	5.95	4	73.4	3.65	4
Ratio PAH_{16}/PAH_{34}	0.42	0.00	4	0.43	0.01	4	0.45	0.07	4	0.46	0.03	4	0.42	0.00	4
Toxic Units (TU_{34})	2.3	0.12	4	1.7	0.08	4	1.7	0.66	4	2.4	0.25	4	2.1	0.10	4

ND - Non detected

Table 5-8. Sediment SPME Pore Water PAH Concentrations and TUs.

	AR02			AR03			AR05			AR07			AR08		
Sediment SPME Pore Water PAHs (µg/L)	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**
naphthalene	1.24	0.176	8	0.087	0.012	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
2-methylnaphthalene	0.439	0.075	8	0.055	ND	1	ND	ND	ND	ND	ND	ND	ND	ND	ND
1-methylnaphthalene	1.74	0.117	8	0.301	0.020	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
C2 naphthalenes	82.1	15.8	8	1.31	0.073	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
C3 naphthalenes	61.3	16.9	8	1.38	0.164	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
C4 naphthalenes	25.3	4.89	8	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
acenaphthylene	8.24	1.20	8	0.148	0.021	4	ND	ND	ND	0.043	0.025	4	ND	ND	ND
acenaphthene	45.1	2.92	8	0.619	0.037	4	ND	ND	ND	0.072	0.018	4	0.080	0.046	4
fluorene	14.6	1.99	8	0.216	0.028	4	ND	ND	ND	0.076	0.020	4	0.064	0.039	4
C1 fluorenes	12.0	1.71	8	0.638	0.074	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
C2 fluorenes	6.51	1.12	8	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C3 fluorenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
phenanthrene	27.7	5.96	8	0.194	0.011	4	0.108	0.018	4	0.134	0.020	4	0.068	0.040	4
anthracene	3.05	0.487	8	0.076	0.013	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
C1 phenanthrenes/anthracenes	20.5	5.96	8	0.439	0.050	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
C2 phenanthrenes/anthracenes	8.07	4.40	8	0.305	0.039	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
C3 phenanthrenes/anthracenes	2.72	0.206	4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C4 phenanthrenes/anthracenes	2.22	0.302	4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
fluoranthene	4.73	1.99	8	0.086	0.001	4	0.031	0.006	4	0.060	0.008	4	ND	ND	ND
pyrene	7.90	3.54	8	0.124	0.006	4	ND	ND	ND	0.049	0.005	2	ND	ND	ND
C1 fluoranthenes/pyrenes	4.19	2.29	8	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benz[a]anthracene	0.367	0.160	8	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
chrysene	0.343	0.131	8	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C1 chrysenes	0.155	0.023	4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C2 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C3 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C4 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[b+k]fluoranthene	0.237	0.050	4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[e]pyrene	0.262	0.058	4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[a]pyrene	0.450	0.099	4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
perylene	0.024	0.004	4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
indeno[1,2,3-cd]pyrene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
dibenz[ah]anthracene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[ghi]perylene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
SPME PAH_{16} (µg/L)	114	18.7	8	1.55	0.070	4	0.139	0.015	4	0.409	0.086	4	0.212	0.101	4
SPME PAH_{34} (µg/L)	677	51.0	4	5.94	0.228	4	0.139	0.015	4	0.409	0.086	4	0.212	0.101	4
SPME Toxic Units (TU_{34})	62	5.7	4	0.43	0.03	4	0.01	0.00	4	0.02	0.00	4	0.01	0.00	4

ND - Non detected

Table 5-8. Sediment SPME Pore Water Concentrations and TUs (continued).

	AR10			AR13			AR14			AR16			AR17		
Sediment SPME Pore Water PAHs (µg/L)	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**
naphthalene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2-methylnaphthalene	ND	ND	ND	ND	ND	ND	0.024	0.004	4	ND	ND	ND	ND	ND	ND
1-methylnaphthalene	ND	ND	ND	0.043	0.010	4	0.028	0.005	4	ND	ND	ND	ND	ND	ND
C2 naphthalenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.475	0.093	4
C3 naphthalenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.389	0.071	4
C4 naphthalenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
acenaphthylene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
acenaphthene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
fluorene	ND	ND	ND	0.066	0.017	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
C1 fluorenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C2 fluorenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C3 fluorenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
phenanthrene	0.058	0.012	4	0.103	0.008	4	0.081	0.011	4	0.029	0.005	4	0.146	0.051	4
anthracene	0.018	0.012	3	0.021	0.006	4	0.017	0.004	4	0.011	0.002	4	ND	ND	ND
C1 phenanthrenes/anthracenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C2 phenanthrenes/anthracenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C3 phenanthrenes/anthracenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C4 phenanthrenes/anthracenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
fluoranthene	0.042	0.003	4	0.049	0.005	4	0.050	0.005	4	0.023	0.007	4	0.052	0.011	4
pyrene	0.036	0.007	4	0.041	0.008	4	0.041	0.005	4	0.015	0.002	4	ND	ND	ND
C1 fluoranthenes/pyrenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benz[a]anthracene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
chrysene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C1 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C2 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C3 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C4 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[b+k]fluoranthene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[e]pyrene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[a]pyrene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
perylene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
indeno[1,2,3-cd]pyrene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
dibenz[ah]anthracene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[ghi]perylene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
SPME PAH_{16} (µg/L)	0.151	0.035	4	0.281	0.016	4	0.190	0.015	4	0.078	0.012	4	0.197	0.061	4
SPME PAH_{34} (µg/L)	0.151	0.035	4	0.324	0.020	4	0.241	0.011	4	0.078	0.012	4	1.06	0.175	4
SPME Toxic Units (TU_{34})	0.01	0.00	4	0.02	0.00	4	0.02	0.00	4	0.01	0.00	4	0.07	0.01	4

ND - Non detected

Table 5-8. Sediment SPME Pore Water Concentrations and TUs (continued).

	AR21			AR25			AR26			AR27			AR36		
Sediment SPME Pore Water PAHs (µg/L)	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**	**Mean**	**Std Dev**	**N**
naphthalene	ND	ND	ND	ND	ND	ND	0.079	0.013	4	ND	ND	ND	0.082	0.023	4
2-methylnaphthalene	ND	ND	ND	ND	ND	ND	0.015	0.001	4	ND	ND	ND	ND	ND	ND
1-methylnaphthalene	ND	ND	ND	ND	ND	ND	0.031	0.005	4	ND	ND	ND	ND	ND	ND
C2 naphthalenes	ND	ND	ND	ND	ND	ND	0.412	0.044	4	ND	ND	ND	ND	ND	ND
C3 naphthalenes	ND	ND	ND	ND	ND	ND	0.293	0.038	4	ND	ND	ND	ND	ND	ND
C4 naphthalenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
acenaphthylene	ND	ND	ND	0.031	0.008	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
acenaphthene	ND	ND	ND	0.044	0.038	4	ND	ND	ND	0.052	0.016	4	ND	ND	ND
fluorene	ND	ND	ND	ND	ND	ND	0.058	0.009	4	ND	ND	ND	0.032	0.012	4
C1 fluorenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C2 fluorenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C3 fluorenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
phenanthrene	0.060	0.010	4	0.045	0.013	4	0.080	0.003	4	ND	ND	ND	0.049	0.006	4
anthracene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.011	0.004	4
C1 phenanthrenes/anthracenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C2 phenanthrenes/anthracenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C3 phenanthrenes/anthracenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C4 phenanthrenes/anthracenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
fluoranthene	0.03	0.005	4	0.030	0.007	4	0.074	0.004	4	0.025	0.012	4	0.038	0.001	4
pyrene	0.02	0.005	4	0.026	0.004	4	0.049	0.005	4	0.023	0.011	4	0.025	0.004	4
C1 fluoranthenes/pyrenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benz[a]anthracene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
chrysene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C1 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C2 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C3 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
C4 chrysenes	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[b+k]fluoranthene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[e]pyrene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[a]pyrene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
perylene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
indeno[1,2,3-cd]pyrene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
dibenz[ah]anthracene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
benzo[ghi]perylene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
SPME PAH_{16} (µg/L)	0.114	0.016	4	0.176	0.019	4	0.340	0.019	4	0.100	0.015	4	0.236	0.034	4
SPME PAH_{34} (µg/L)	0.114	0.016	4	0.176	0.019	4	1.09	0.094	4	0.100	0.015	4	0.236	0.034	4
SPME Toxic Units (TU_{34})	0.01	0.00	4	0.01	0.00	4	0.06	0.00	4	0.01	0.00	4	0.01	0.00	4

ND - Non detected

5.7.3 Aquatic Toxicity Testing

The aquatic toxicity tests used the amphipod *Hyalella azteca* in a 28-day testing regime. Both acute and chronic endpoints were determined (i.e., survival as well as growth). The results of the *H. azteca* toxicity tests conducted on the sediments adjacent to the WNY were statistically compared to the results from the reference samples (Table 5-9). These results indicate a reduced survival of one sediment sample (AR02 at 18.3 +/- 16%) and a reduced growth of one sediment sample (AR03 at 0.20 +/- 0.05%). Note that growth was not measured on the sample with reduced survival. Both of these samples are located at the northern end of the WNY, at the boundary of the Washington Gas former MGP site. This site is also the location of the M street combined sewer outfall (CSO). It is not certain if the reduced survival/growth in this area is due to impacts from the CSO, MGP site, operations at the WNY, or a result of deposition of PAH-contaminated suspended particulates from upstream sources. Note that PAHs associated with suspended particulates originating from upstream sources in the Anacostia River have been previously quantified. Since the WNY sediments are in a depositional region of the Anacostia river, many of the PAHs analyzed in the surficial sediments at the WNY may have been transported from off-site source areas. Aquatic toxicity test results and statistical analysis are provided in the ERDC report as Appendix J.

Table 5-9. Aquatic Toxicity Test Results with Summarized Total and Pore Water Concentrations.

Sample Type	Sample	Total PAH_{16} (mg/kg)	SPME Pore water TU_{34}	*Hyalella azteca* (28 day)							
				Survival (%)				Growth (mg dry wt./organism)			
				Sig	Mean[a]	S.D.[b]	N[c]	Sig	Mean[a]	S.D.[b]	N[c]
CONTROL	AR_Control	NA	NA		100	0.0	6		0.26	0.05	6
REFERENCE	AR21	38.5	0.010		88	7.5	6		0.44	0.09	6
REFERENCE	AR25	21.9	0.010		95	5.5	6		0.26	0.04	6
TEST	AR02	600	62.1	*#	8.3	16	6	+	0.29	0.13	6
TEST	AR03	14.4	0.427		88	10	6	*	0.20	0.05	6
TEST	AR05	35.8	0.010		95	5.5	6		0.26	0.06	6
TEST	AR07	23.4	0.021		88	16	6		0.32	0.06	6
TEST	AR08	21.8	0.007		93	10	6		0.34	0.13	6
TEST	AR10	26.9	0.013		92	10	6		0.36	0.06	6
TEST	AR13	385	0.020		95	5.5	6		0.40	0.05	6
CONTROL	AR_Control	NA	NA		88	12	6		0.30	0.03	6
REFERENCE	AR21	38.5	0.010		77	14	6		0.42	0.07	6
REFERENCE	AR25	21.9	0.010		88	9.8	6		0.33	0.03	6
TEST	AR14	35.6	0.017		93	5.2	6		0.30	0.04	6
TEST	AR16	34.2	0.007		93	5.2	6		0.38	0.03	6
TEST	AR17	40.0	0.066		95	5.5	6		0.31	0.06	6
TEST	AR26	65.4	0.062		73	37	6		0.46	0.06	6
TEST	AR27	25.7	0.007		63	50	6		0.37	0.06	6
TEST	AR36	30.5	0.012		90	15	6		0.36	0.08	6

[a] Arithmetic mean

[b] Standard deviation

[c] Number of replicate beakers per treatment (sample)

* Statistically different from the pooled field reference samples, AR21 and AR25 ($p<0.05$)

[#] Statistically different from the laboratory performance control ($p<0.05$)

[+] Significant for survival and therefore not included in statistical analysis of growth or reproduction

Comparisons of survival and dry weights among treatments were conducted with the Wilcoxon/Bonferroni and Bonferroni Adj. t procedures (U.S. EPA, 2000). Statistical tests for comparison of survival and growth compared to the pooled field reference samples and laboratory control were conducted using CETIS™

5.7.4 Organic Carbon

TOC ranged from 0.9 to 11.1% and SOC ranged from 0.2 to 6.4%, with a SOC/TOC ratio ranging from 0.1 to 0.6, indicating varying amount of anthropogenic carbon in the sediments (Table 5-10). Sediment sample AR03 showed a reduced TOC concentration relative to the other 14 sediment samples, reflecting its sandier constitution. Organic carbon chemistry results are provided in Appendix I.

Table 5-10. Sediment TOC and SOC.

Sample	Organic Carbon			
	Sediment TOC (wt. %)	Sediment Soot C (wt. %)	Dissolved Organic Carbon (mg/L)	Ratio SOC/TOC
AR02	9.4	3.7	15.5	0.4
AR03	0.9	0.2	4.8	0.2
AR05	3.3	0.3	4.1	0.1
AR07	3.9	0.4	6.9	0.1
AR08	3.7	0.4	4.5	0.1
AR10	3.7	0.3	3.4	0.1
AR13	4.9	0.7	3.3	0.1
AR14	3.9	0.4	4.1	0.1
AR16	6.6	1.6	2.8	0.2
AR17	3.5	0.6	4.0	0.2
AR21	5.0	0.3	12.8	0.1
AR25	3.9	0.5	4.2	0.1
AR26	11.1	6.4	3.5	0.6
AR27	3.0	0.3	7.9	0.1
AR36	4.5	0.5	5.0	0.1

5.7.5 Detailed Carbon Chemistry

Four of the 15 sampled sediments were selected for a more detailed analysis of the type of carbon present, based on total bulk PAH concentrations, pore water PAH concentrations, SOC concentrations, and *H. azteca* survival, to provide a range of characteristics found at the WNY. The properties of the selected samples are listed in Table 5-11, and are summarized as:

- AR02: high bulk sediment PAH_{16} and TU PAH_{34}, high SOC, and low H. azteca survival
- AR03: low bulk sediment PAH_{16}, a relatively high pore water TU PAH_{34}, a reduced growth of *H. azteca*, and low TOC and SOC
- AR13: high bulk sediment PAH_{16} and low pore water TU PAH_{34} and high percent survival of H. azteca

- AR16: moderate bulk sediment PAH_{16} and low pore water TU PAH_{34}, and high SOC.

A summary of the type of organic carbon associated with each of the four sediment samples is presented in Table 5-12. The full suite of analytical results is provided as the UMBC report in Appendix K.

Table 5-11. Properties of the Four Sediment Samples Used for Detailed Carbon Analysis.

Sample ID	Total PAH16 (mg/kg) UMBC	Total PAH16 (mg/kg) (UND)	SPME pore water TU34	TOC Wt % dry	SOC Wt % dry	Hyalella Azteca (28 day)			
						Survival		Growth	
						mean	n	mean	n
AR02	886±73	800	62.2	9.4± 0.12	3.70± 0.13	8.3± 16	6	0.29± 0.13	6
AR03	15±2	19.2	0.427	0.88± 0.14	0.18± 0.03	88± 10	6	0.20± 0.05	6
AR13	44±4	514	0.020	4.89± 0.26	0.67± 0.30	95± 5.5	6	0.40± 0.05	6
AR16	45± 8	45.6	0.007	6.64± 0.32	1.64± 0.28	93± 5.2	6	0.38± 0.03	6

Table 5-12. Petrographic Compositional Analysis of the Sediment Organic Particle Fraction in the Size Range of 0.25 – 1.0 mm.

SAMPLE NAME	AR-02	AR-03	AR-13	AR-16
	Volume %	*Volume %*	*Volume %*	*Volume %*
Carbons				
Softened Coal	2.2	2.2	---	---
High Vol. Bituminous Coal	14.8	0.4	---	4.6
Medium Vol. Bituminous Coal	7.8	0.8	---	0.6
Low Vol. Bituminous Coal	4.8	1.0	0.4	1.4
Anthracite Coal	9.4	14.0	---	1.8
Oxidized Coal	2.2	4.6	0.6	1.2
Coal Inerts	0.6	0.2	---	1.6
Total Coal	41.8	23.2	1.0	11.2
Metallurgical Coke	9.2	5.4	---	6.0
Depositional Carbon on Coke	0.4	0.4	---	---
Burnt Coke	0.4	---	---	---
Coked Inert	1.8	0.2	---	2.0
Carbon Black	1.2	---	---	---
Charcoal	1.0	---	---	1.2
Graphite	---	0.8	---	0.2
Total Coke/Carbon	14.0	6.8	0.0	9.4
Coal Tar Pitch Low to Medium QI	3.6	---	1.0	---
Coal Tar Pitch High QI	3.0	1.0	0.8	---
Coal Tar Pitch Coating/Mixed	2.6	---	---	---
Cenosphere	1.0	---	0.2	0.8
Gum or Tar	---	1.6	0.6	1.0
Total Byproduct Related	10.2	2.6	2.6	1.8
Woody Plant Material (cellular structure)	2.8	5.4	12.2	11.8
Green Plant Material (cellular structure)	6.2	17.0	47.8	32.6
Total Plant Material	9.0	22.4	60.0	44.4
Mineral Matter				
Groundmass Minerals	8.4	20.8	18.8	8.2
Groundmass Mineral Coating Plant/Carbon	5.0	1.4	2.2	1.2
Groundmass Mineral Mixed - Plant/Carbon	3.4	1.2	1.2	0.2
Glassy Slag	3.2	---	---	8.4
Slag with Metallics	2.2	---	---	---
Quartz	0.4	---	1.2	---
Transparent Mineral	---	1.4	1.0	2.8
White Grainy Mineral w/ red & green Mineral Inclusions	---	---	5.4	---
White Mineral with Milky to Grainy Texture	---	5.8	---	10.2
Pyrite	0.8	---	0.2	---
Iron Oxide	---	0.8	1.8	0.4
Plant Like Structure w/Mineral Matter Walls	---	6.6	---	---
Diatom	1.6	7.0	4.6	1.8
Total Mineral Matter	25.0	45.0	36.4	33.2
Grand Total	100.0	100.0	100.0	100.0

5.7.6 Interpretation of analytical results

The sediment PAH chemistry and aquatic toxicity test results were compared in order to provide a weight-of-evidence approach for indicating whether surficial sediments at the WNY could have a potential impact on the benthic community. The interpretation consisted of the following comparisons:

- Bulk sediment PAH_{16} to the aquatic toxicity results
- Bulk sediment PAH_{34} to the aquatic toxicity results
- SPME pore water PAH_{34} to the aquatic toxicity results
- EqP-estimated pore water PAH_{34} to the aquatic toxicity results

5.7.6.1 Bulk sediment PAH comparison to survival of H. azteca

A comparison of the bulk sediment PAH concentration to the survival of *H. azteca* is presented in Figure 5-2 for PAH_{16} and Figure 5-3 for PAH_{34}. Note that the gray squares indicate data previously collected in the SCBA program. It can be seen from this comparison that although there is a difference in PAH_{16} between the sample with reduced survival (AR02 at 600 mg/kg) and the non-toxic sample with highest total PAH concentration (sample AR13 at 385 mg/kg) both of these concentrations are well above generic screening levels. Also note that the use of the TEL and PEL screening values (1.6 and 22.8 mg/kg, respectively) would result in almost all of the PAH_{16} samples exceeding the screening values, and all of the PAH_{34} samples exceeding the screening values.

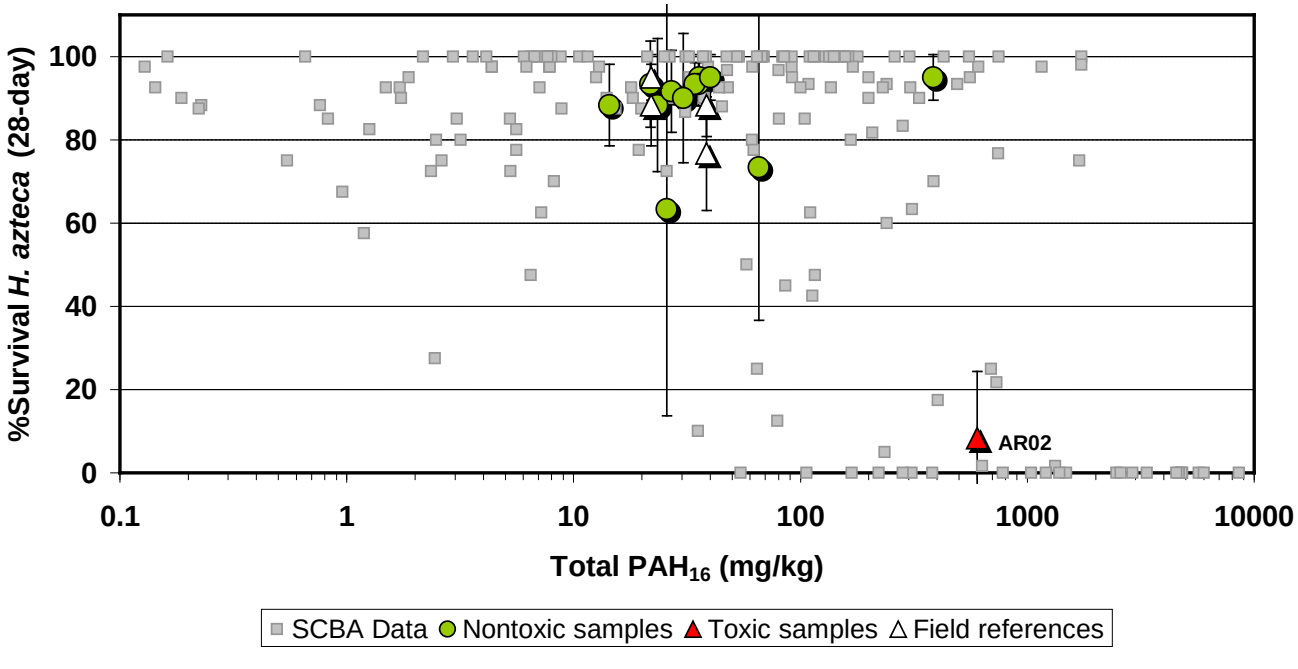


Figure 5-2. Bulk Sediment PAH_{16} Compared to *H. azteca* Survival.

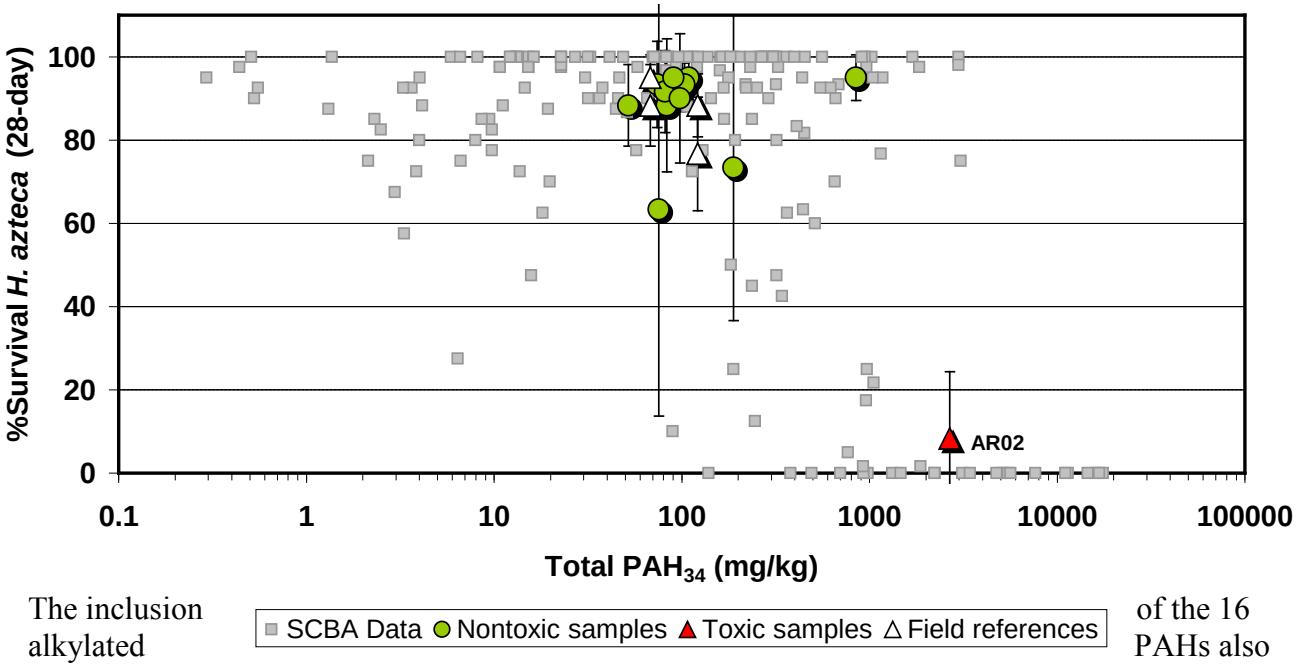


Figure 5-3. Bulk Sediment PAH_{34} Compared to *H. azteca* Survival.

The inclusion of the 16 alkylated PAHs also increases the total PAH concentration, so that the use of this summation value will result in a greater probability of exceeding the TEL/PEL screening values.

5.7.6.2 Pore water PAH comparison to survival of H. azteca

The relationship between SPME pore water PAH_{34} expressed on a TU basis and survival of *H. azteca* is presented in Figure 5-4. Note that the expression of pore water concentrations in terms of TUs creates a larger spread between the non-toxic samples and the toxic sample. In addition, all non-toxic samples occur at a TU $PAH_{34} < 1$, while the toxic sample (AR02) occurs at a TU $PAH_{34} > 1$. The previous SCBA data (gray squares) follow a pattern which was modeled using probit analysis (see the solid line in Figure 5-4). The WNY SPME pore water TU PAH_{34} and survival data conform to this model.

The comparison between the SPME pore water PAH concentrations and the survival of *H. azteca* shows that sediments with moderate amounts of PAHs are generally not toxic to benthic invertebrates in the lower Anacostia River. The U.S. Fish and Wildlife Service conducted a sediment triad study in the lower Anacostia River, consisting of 10-day survival and growth test using *H. azteca* and *C. dilutus*, bulk sediment PAH concentrations, and benthic community surveys collected at 20 sampling stations. The study concluded that only one of the 20 stations had a high probability of benthic degradation due to sediment-associated contaminants. This sample was located just south of the WNY, near the Southeast Federal Center (SEFC). One of the sediment samples located near AR02 did not show any reduced survival or growth of the test species, but scored low on a benthic index of biotic integrity (B-IBI), indicating that the sample area was in a degraded condition. However, this benthic community assessment did not correlate with chemical or toxicological results (McGee, et.al., 2009).

Traditionally, when bulk sediment PAH concentrations exceed a tier 1 screening level (i.e., TEL or PEL), the next tier analysis is to compare pore water chemistry to FCVs. In this case, pore water TUs are calculated using equilibrium partitioning, whereby the bulk sediment

concentrations are adjusted to TOC, and then multiplied by the published equilibrium partitioning coefficient to estimate pore water PAH concentrations. These estimated pore water concentrations are then converted to TUs, and summed to provide a total PAH TU. The relationship between estimated pore water PAH TUs and survival of *H. azteca* is presented in Figure 5-5. Two important insights are evidenced on this figure: 1) All of the sediment samples have TUs > 1, although only one was found to be toxic to *H. azteca*, and 2) there is still considerable scatter in the data (both the WNY data and the SCBA database) which makes any sort of competent prediction of aquatic toxicity from EqP-estimated pore water analysis not practical - although there is some separation between samples in the WNY data, the separation is not sufficient to be of much predictive use. Figure 5-6 shows the relationship between the published EPA equilibrium partitioning coefficients (LKoc) and the site-specific equilibrium partitioning coefficients for sample AR02 (the sample showing toxicity to *H. azteca*). The EPA values overestimate the LKoc for the alkylated PAHs (a higher LKoc indicates a lower bioavailability). These results differs from the majority of samples within the SCBA database, where the EPA Koc values are generally much lower than site-specific Koc values. This might be related to the observation that an oil phase is present in some of the samples, where the partitioning coefficient from an oil phase will be lower than that from a soil carbon. It has been shown previously that the alkylated PAHs provide much of the toxicity of these compounds to benthic organisms (U.S. EPA, 2009).

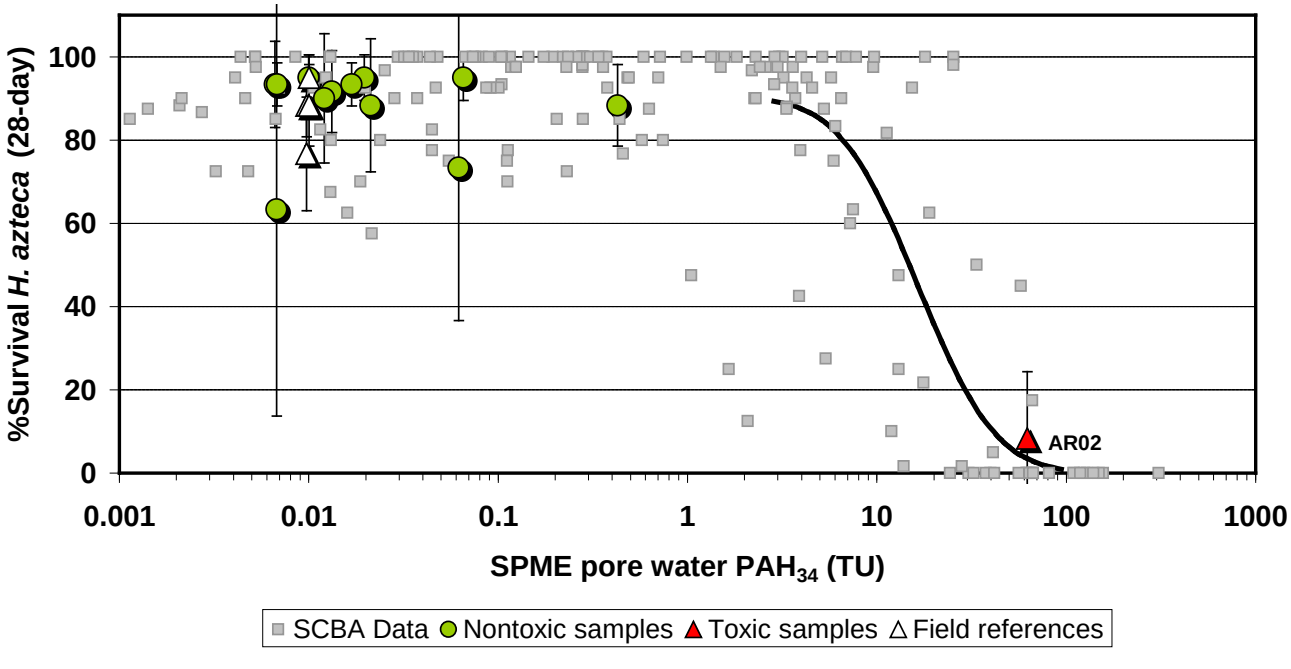


Figure 5-4. SPME Pore Water TU PAH_{34} Compared to *H. azteca* Survival.

Attempts have been made to incorporate the soot organic carbon (SOC) fraction into the estimation of PAH pore water concentrations (see EPRI, 2005). Although this additional step may better represent the relationship with aquatic toxicity tests, the comparison often does not add any additional information when there are multiple types of SOC, which occurs in most industrial/urban waterways. For the WNY data, McDonough and Azzolina (2010) estimated

PAH pore water concentrations using published equilibrium partitioning coefficients based on soot organic carbon (EPRI, 2005). The estimated PAH TUs do not match well with the actual SPME PAH TUs, and fail to recognize the increased TUs found in the only toxic sample (AR02)(Figure 5-7).

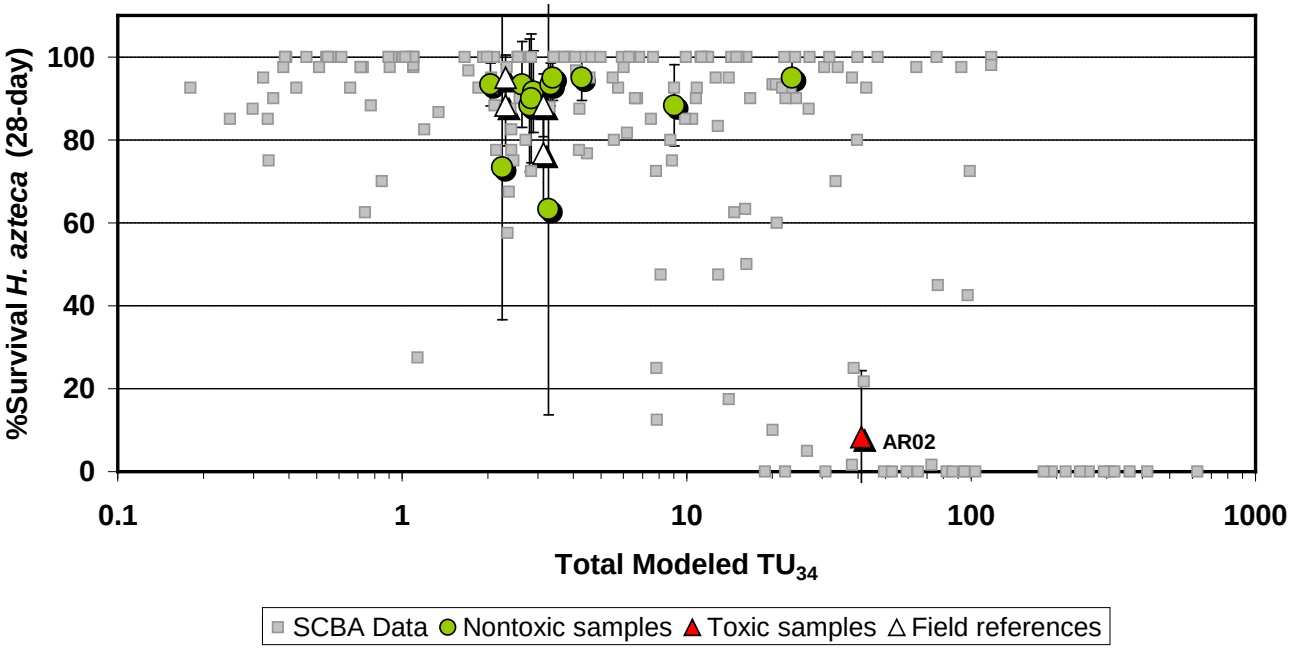


Figure 5-5. EqP-estimated Pore Water TU PAH_{34} Compared to *H. azteca* Survival.

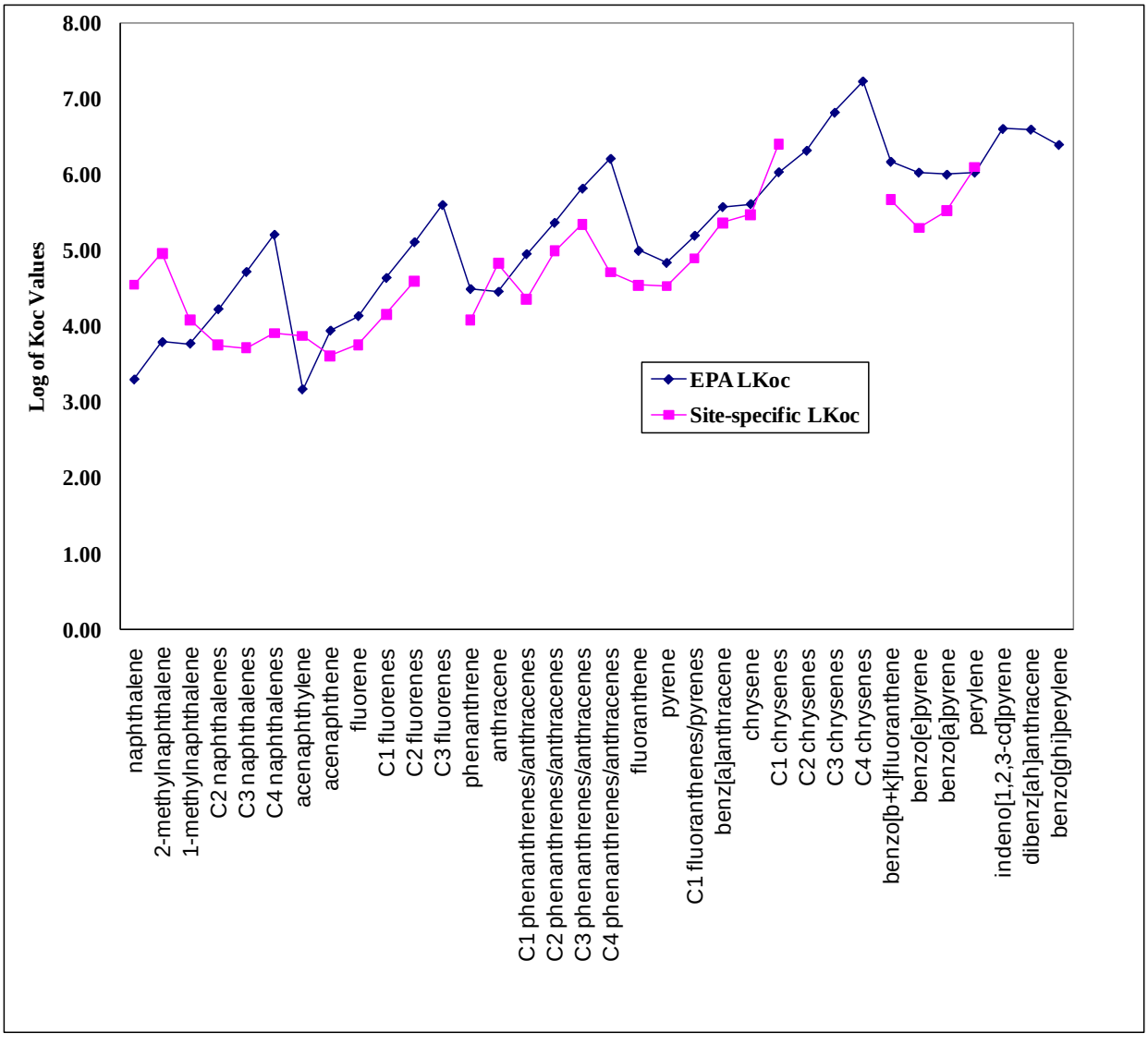


Figure 5-6. Relationship Between Published Koc (EPA LKoc) and WNY Koc (Site-specific LKoc) values.

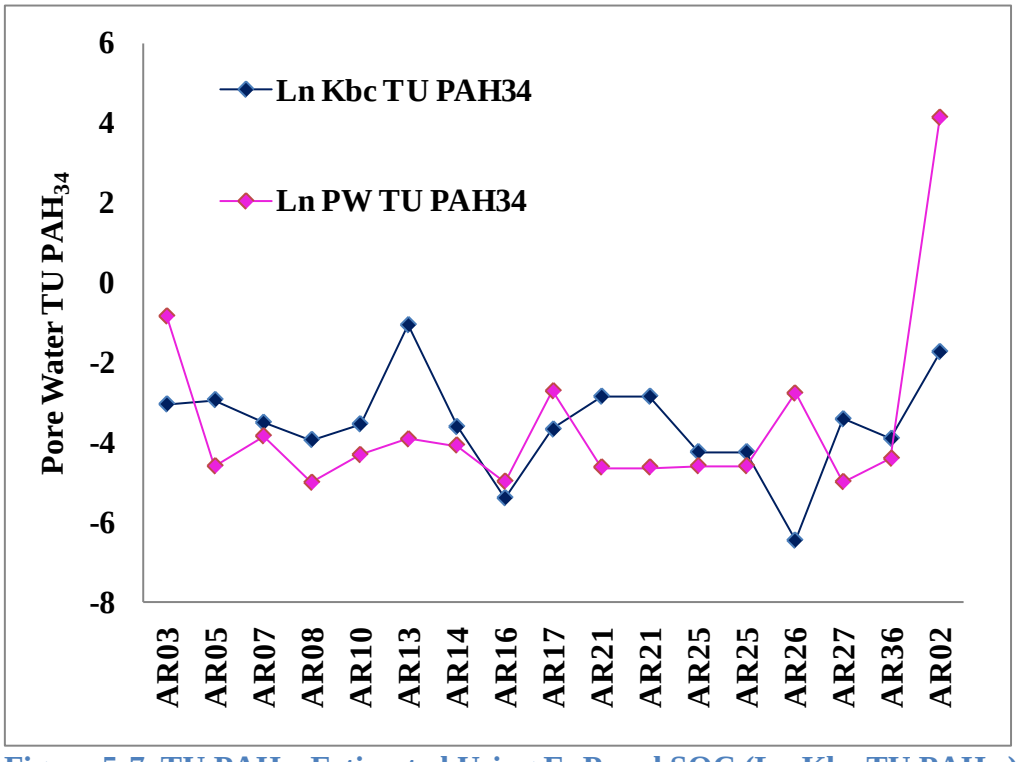


Figure 5-7. TU PAH_{34} Estimated Using EqP and SOC (Ln Kbc TU PAH_{34}) and from SPME Pore Water Analysis (Ln PW TU PAH_{34}).

5.7.6.2 Pore water PAH comparison to growth of H. azteca

The growth of *H. Azteca* was compared to the total PAH_{16} and the TU PAH_{34} concentrations for all samples, and is presented in Figures 5-8 and Figure 5-9. The SCBA dataset indicates no correlation between growth of *H. azteca* and either total PAH_{16} or TU PAH_{34} over the range of samples included in the program (gray squares). There was also no correlation between these variables in the WNY dataset. However, one sample (AR03) was found to have statistically reduced growth. This sample had the lowest total PAH_{16} concentrations, but the highest TU PAH_{34} concentration, although it was slightly less than a TU of 1 (note that sample AR02 was not included in the analysis since it showed statistically reduced survival). Sample AR03 is located proximate to sample AR02, which was the only sample observed to induce toxicity in *H. azteca*. Factors in the low growth of H. azteca in sample AR03 are the sandy nature of the sediment, as well as low TOC and SOC.

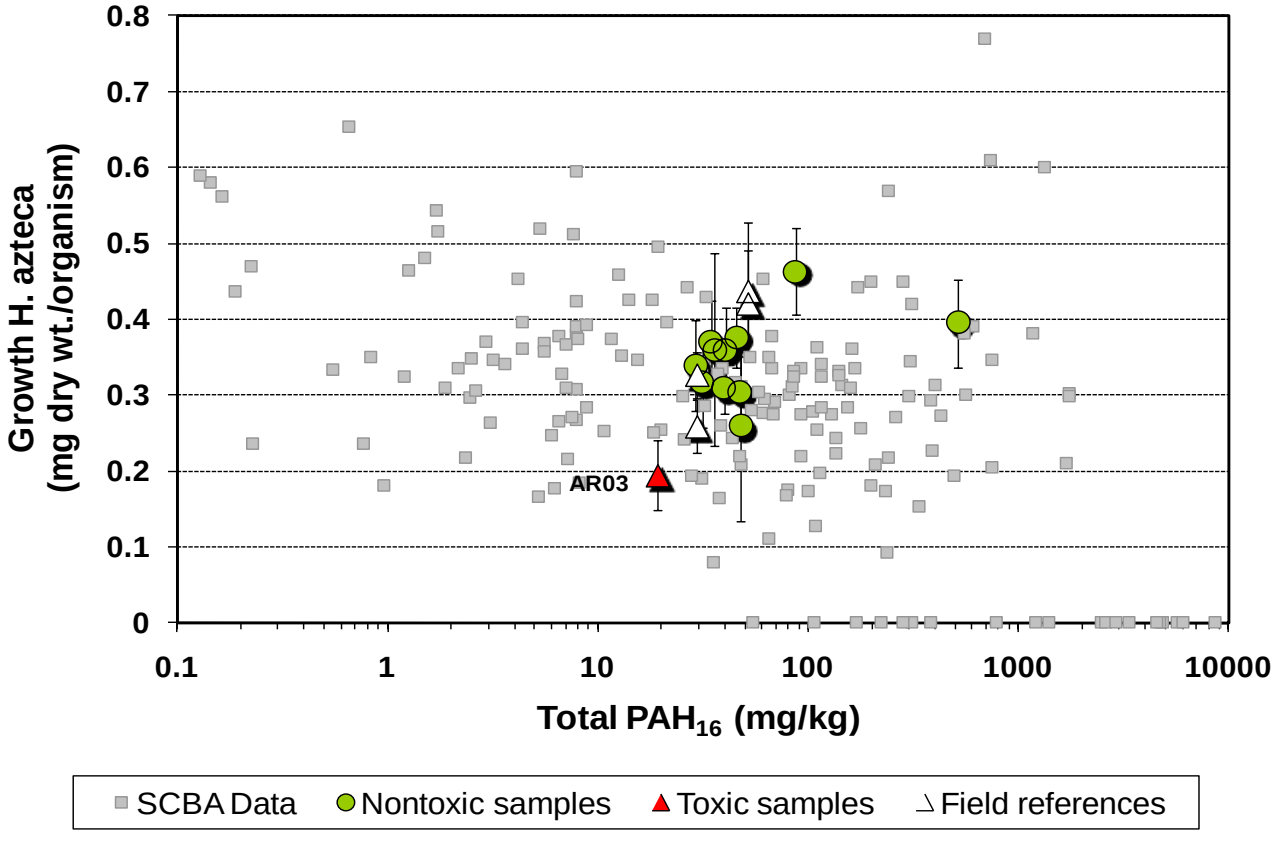


Figure 5-8. Bulk Sediment PAH_{16} Concentrations Compared to Growth of *H. azteca*.

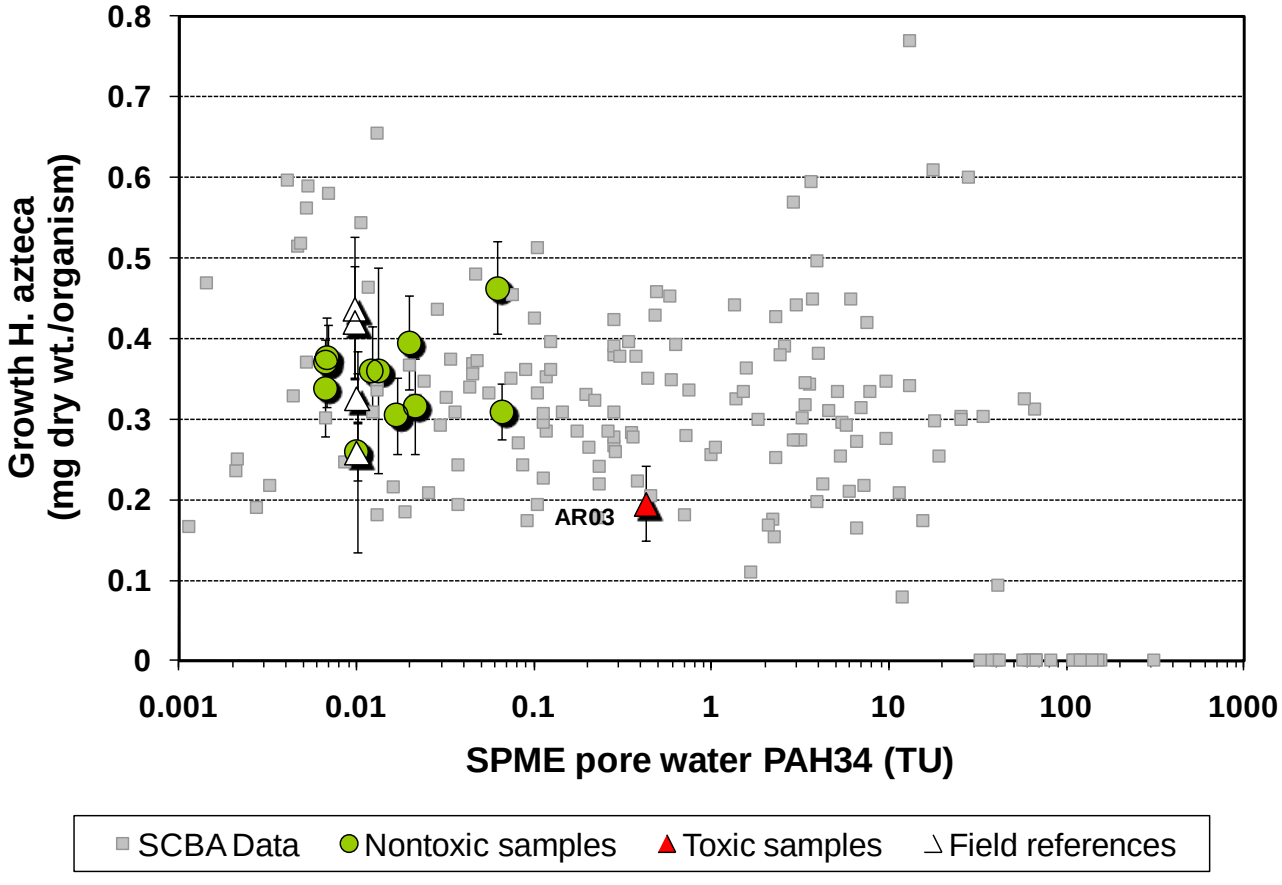


Figure 5-9. SPME Pore Water TU PAH_{34} Compared to Growth of *H. azteca*.

5.7.6.3 Detailed Carbon Analysis

The four sediments selected for detailed carbon analysis differed considerably in the distribution of PAHs within the carbon types present. Sample AR02 (high bulk sediment PAH_{16} and TU PAH_{34}, high SOC, and low *H. azteca* survival) PAHs were predominantly in the coal and wood fraction, with a smaller amount in the coke fraction. Sample AR03 (low bulk sediment PAH_{16} and high pore water TU PAH_{34}, and a reduced growth of *H. azteca*) PAHs were predominantly in the coal fraction, with a smaller amount in the coke fraction, although the majority of the sample was comprised of heavy density mineral particles. Sample AR13 (high bulk sediment PAH_{16} and low pore water TU PAH_{34} and high *H. azteca* survival) PAHs were distributed between the coal and pitch fractions. Sample AR16 (moderate bulk sediment PAH_{16}, and low pore water TU PAH_{34}, and high SOC) PAHs were distributed between the coal, coke and wood fractions, with a small amount in the sand fraction (Figure 5-10).

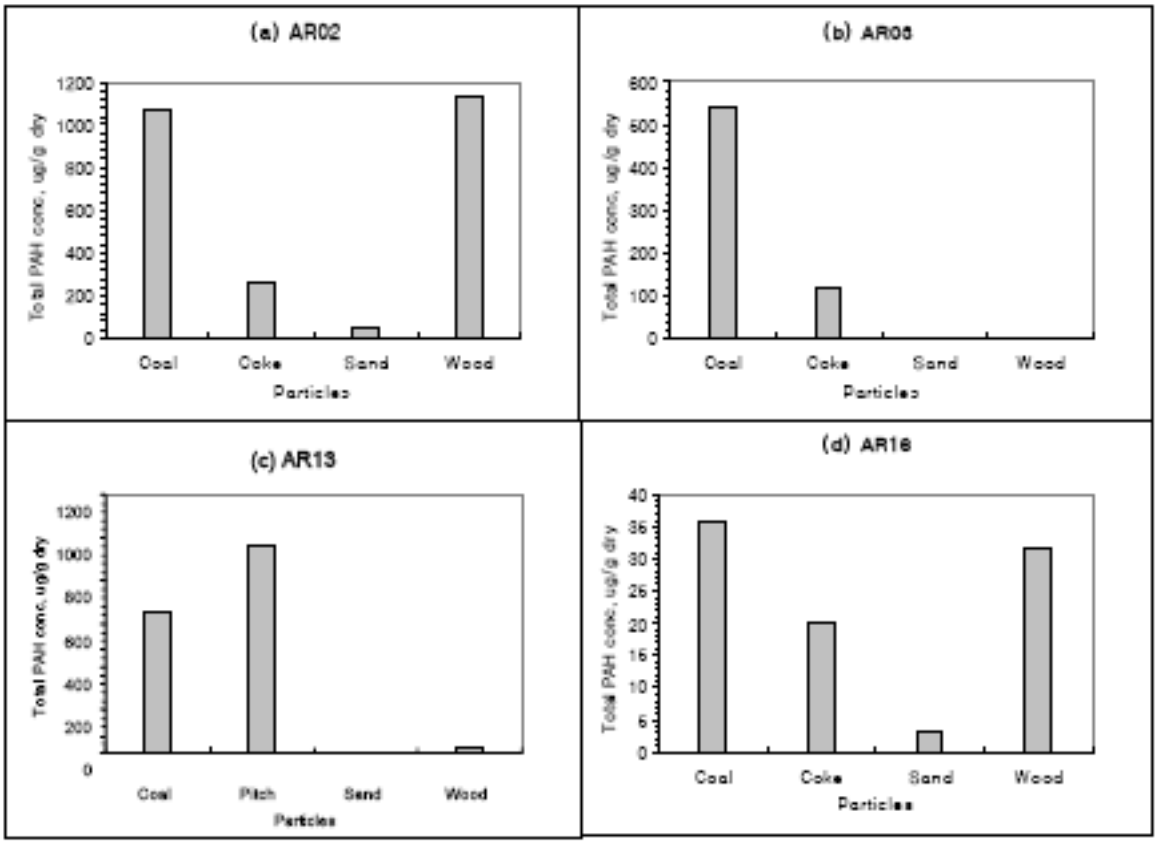


Figure 5-10. Total PAH Concentrations in the Particulate Fraction of Four Sediment Samples.

A detailed description of the carbon type in each of the four sediments is as follows:

Sample AR02: Sample AR02 was the only sample to show reduced survival of *H. azteca*, and the only sample to have a SPME pore water TU PAH_{34} >1. The total PAH_{34} concentration in this sample is 886 mg/kg. This sediment sample is odorous and a sheen of oil was visible on the top of the overlying water indicating that an excess free phase of tar/oil was present that causes high pore water toxic units and *H. azteca* mortality. Clays and silt are the most abundant component of the sediment and a majority of the PAHs are associated with this fine fraction as expected for a sediment with a free oil phase that can coat particle surfaces and be enriched in the finer size mineral fraction. It is important to note that this sediment also had the highest TOC and SOC of all the sediment samples. Petrography analysis revealed an abundance of coal and coke derived particles in the organic fraction. However, in the presence of a free oil phase, the black carbon particles are inactivated and behave no different from wood particles as evidenced by similar PAH concentrations on wood and coal particles in this sediment. The interpretation of the role of black carbon on PAH partitioning in sediment needs to be interpreted carefully when there is free oil phase present as discussed in detail by Hong et al. (2003). In summary, the toxicity of sediment PAHs to *H. azteca* in this sample is a function of the free oil phase coating which negates the effect of SOC on sequestering PAHs. This result explains why the TU PAH_{34}

modeled using EqP with the addition of SOC was unable to predict the toxic effect (see discussion in Section 5.7.6.2)

Sample AR03: This sediment was the only sample which showed reduced growth of *H. azteca*, although SPME pore water TU PAH_{34} was less than 1.0. This sediment is made up of coarse sandy particles and contains low PAHs (15 mg/kg) compared to other samples tested in this report. More than 59% of the sediment constitutes heavy and larger than 1 mm particles and the sum of light sediment mass is less than 1%. A majority of the PAHs in this sediment is associated with the heavy density mineral particles which may explain why, in this sediment with the lowest PAH concentration, the pore water TUs (0.427) is higher than some of the other sediments with higher PAH concentrations. Although this sample contains some coal and coke particles in the organic fraction, the overall abundance of organic particles is small as indicated by the low TOC value of 0.88%. It is highly likely that the low growth of H. azteca in this sample is a function of the particle-size distribution influence on the ability of the sediment to effectively sequester PAHs.

Sample AR13: The subsample used for organic characterization at UMBC contained 44 mg/kg PAHs, compared to 504 mg/kg measured in the subsample used by EERC for pore water analysis. Thus, the results of the various characterization studies for this sample will need to be interpreted with caution. This sediment was a mixture of various sized particles with a large fraction of wood debris. More than 50 % of total PAH in this sediment is associated with the light density 0.25-1.0 mm sized particles which comprises only 4 % of total sediment mass. Within this size fraction, coal and weathered pitch particles had the highest PAH concentration (600-1000 mg/kg) which was about 2 orders of magnitude higher than the PAH concentration on wood particles. This sediment sample exemplifies a case where the coal and pitch particles exhibit high sorption capacity for PAHs, as expected, and which are the primary reservoirs of PAHs in the sediment. As expected, this sediment has a low pore water PAH concentration and TU, and was non-toxic to *H. azteca* (95% survival).

Sample AR16: This sediment is similar to AR13 in PAH concentration and distribution among particle classes. A majority of the PAHs in this sediment are associated with the light density 0.25-1.0 mm sized particles which comprises only 5% of total sediment mass. However, the total PAH concentration on individual particle types in this sediment is much lower compared to that in sample AR13. Wood particles in this sediment appear to have similar PAH concentration compared to coal and coke particles. However, petrography analysis reveals that some of the wood particles are partially charred to form charcoal that can have a high affinity for PAHs. As expected based on the low PAH concentration and abundance of strongly sorbing coal, coke, and charcoal particles, this sediment has a low pore water PAH concentration and TU, and was non-toxic to *H. azteca* (93% survival).

The results from particle separation and organic characterization of the WNY sediment samples support the observations of strong sorption of PAHs to sediment and reduced toxicity to *H azteca*. The strong sorption is explained by the association of PAHs with coal, coke, charcoal, and weathered pitch particles found in the sediment when the sorption capacity of the geosorbents is not attenuated by the presence of a free oil phase. Presence of an excess oil phase in sediment can complicate interpretations using black carbon analysis as the sole tool to assess PAH bioavailability and toxicity in sediments. However, because a mechanistic understanding of the contaminant partitioning phenomena is not required, pore water PAH analysis and toxic unit

calculation provides an accurate assessment of PAH availability in sediments even within the complex interactions between different geosorbent forms and a free oil phase in sediment.

5.8 Practical Application of the Demonstration Results

The demonstration results showed that the direct analysis of pore water PAH concentrations using SPME , in conjunction with the tiered approach presented in the EPA's White Paper entitled *Evaluating Ecological Risk to Invertebrate Receptors from PAHs in Sediments at Hazardous Waste Sites* (EPA-600-R-06-162F) (U.S. EPA, 2009), was able to accurately quantify the effect of sediment-bound PAHs to a benthic test species. Other methods to estimate this effect, such as the use of generic screening levels and pore water concentrations derived from bulk sediment concentrations based on equilibrium partitioning theory (with and without the inclusion of a black carbon phase) were not able to predict the actual effect on the survival of *H. azteca*. The practical application of these results are summarized in Figures 5-11 through 5-13, which present an aerial plan view of the effects of using the suggested tiered approach to characterizing PAH impacts to these sediments:

1. Tier 1a (Figure 5-11): Compare bulk sediment PAH concentrations to the threshold effects levels (TEL) or probable effects levels (PEL). Using this approach suggests that all but two of the sediment samples near the WNY would result in adverse effects to the benthic community;
2. Tier 1b (Figure 5-12): Estimate pore water PAH concentrations using equilibrium partitioning, and compare pore water PAHs with final chronic values (FCV) to derive toxic units (TU) and predict toxicity using the hydrocarbon narcosis model – This approach suggest that all of the sediment samples near the WNY are potentially toxic to benthic organisms;
3. Tier 2 (Figure 5-13): For samples exceeding a screening level or a TU of 1.0 in the Tier 1 analysis, conduct a direct analysis pore water PAH concentrations, and compare the pore water concentrations with the FCV to derive TUs – This analysis provides results that are totally counter to the Tier 1 results, suggesting that only one of the sediment samples would be toxic to *H. azteca*; and
4. Tier 3 (Figure 5-13): For samples exceeding a critical TU threshold (as determined based on the development of a site-specific dose-response curve (i.e., toxicity versus pore water TUs)), this tier involves conducting aquatic toxicity tests using an appropriate test species. At the WNY, the aquatic toxicity testing yielded results that were identical to the results of the Tier 2 analysis prediction.

The result of the Tier 2 and Tier 3 analysis above indicated that, based on the proposed EPA approach, the Tier 3 analysis would never have been required at an actual site since none of the pore water samples exceeded the threshold TU value.

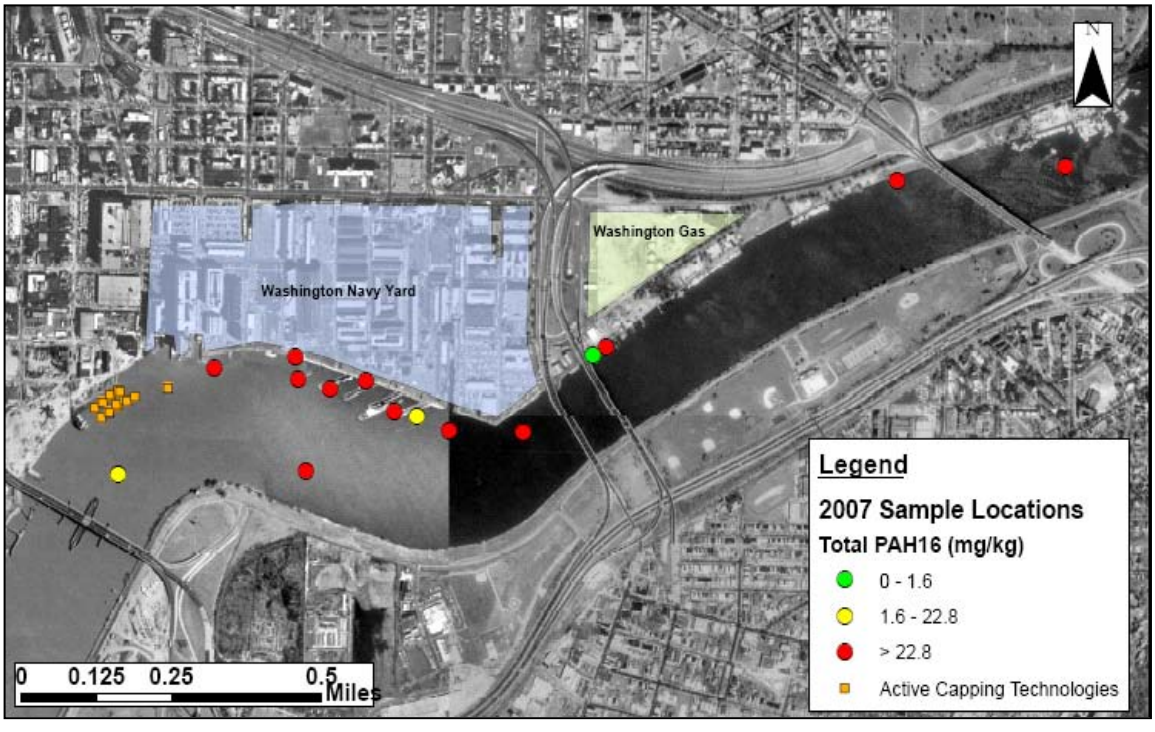


Figure 5-11. Bulk Sediment Total PAH_{16} Concentrations Compared to the Threshold Effects level (TEL) and 1.6 mg/kg and the Probable Effects Level (PEL) of 22.8 mg/kg.

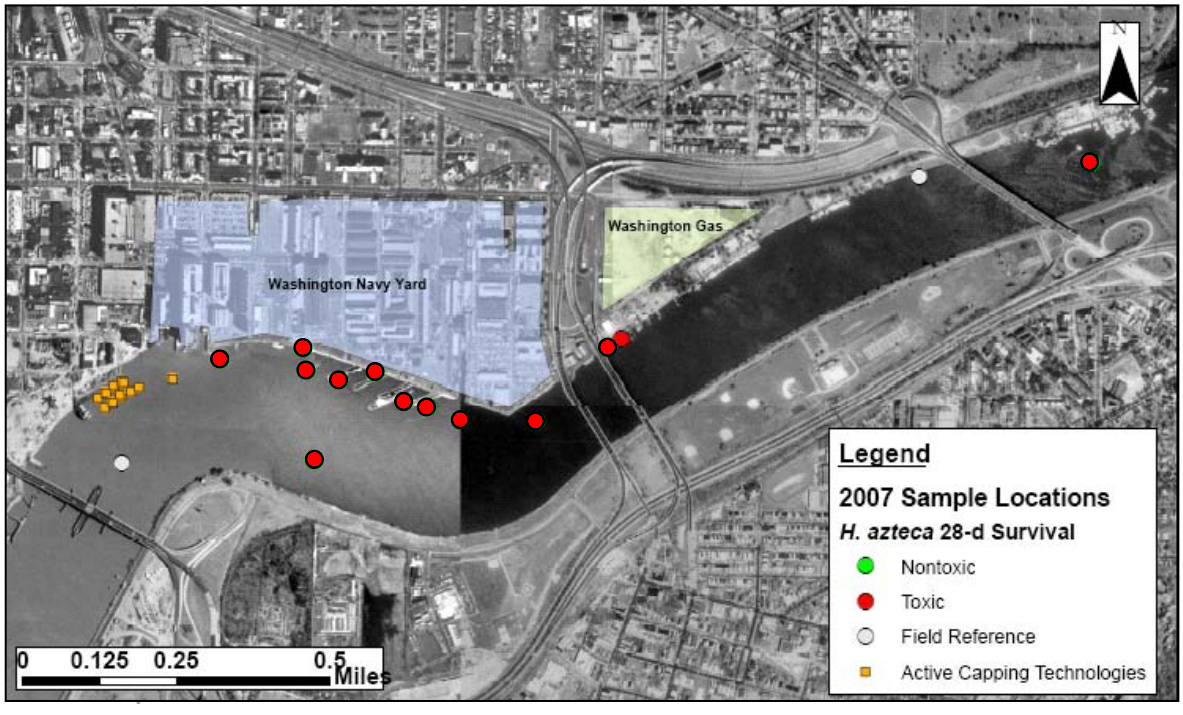


Figure 5-12. Toxicity of PAHs Estimated Using Equilibrium Partitioning.

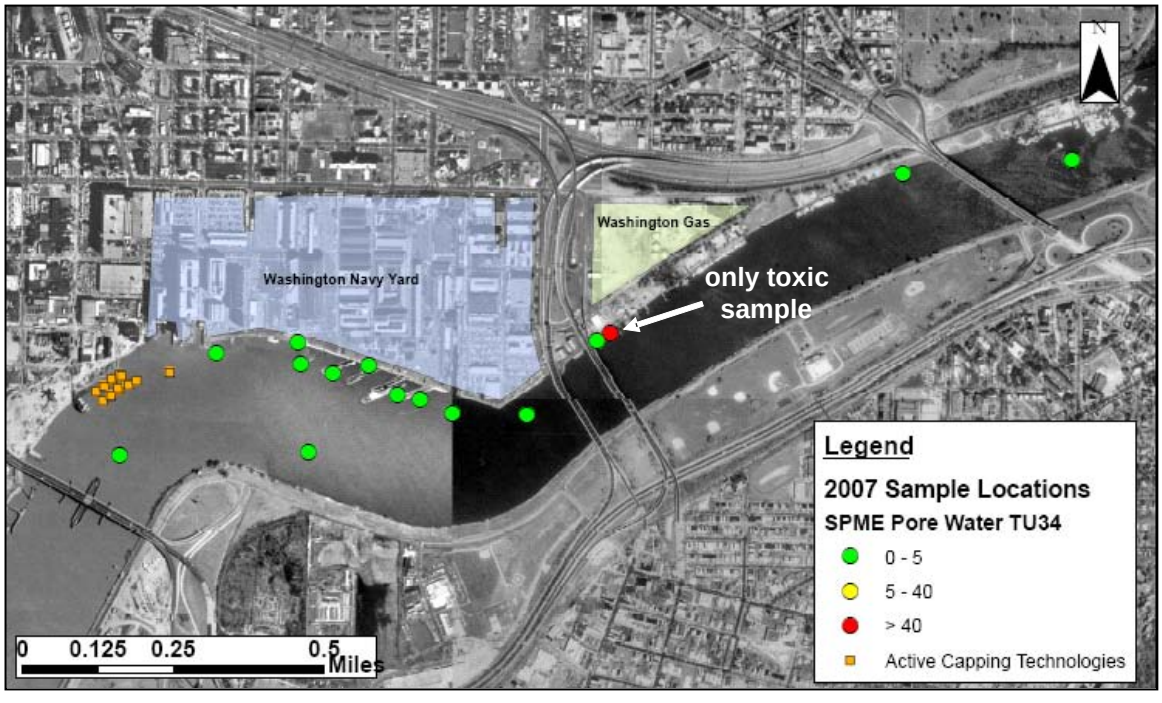


Figure 5-13. Prediction of Toxicity Based on the *H. azteca* 28-day Test for Survival and the Toxicity Predicted Using the Direct Analysis of Pore Water PAHs Using SPME.

6.0 PERFORMANCE ASSESSMENT

This section presents the results of the study and how they relate to the original performance objectives. The original performance objectives are presented and discussed in Section 3.0. The following performance objectives were presented in the demonstration:

1. Quantitative Performance Objectives
    a. Confirm the presence of impacted sediments at the site that are toxic to the aquatic test organisms
    b. Confirm the presence of sediment PAHs in the biologically-active zone of the site
    c. Validate the use of the SPME pore water analytical method to estimate the bioavailability of sediment-bound PAHs by predicting aquatic toxicity
2. Qualitative Performance Objectives
    a. Develop a tiered, risk-based approach to management of PAH-impacted sediment based on direct chemical measures of PAH bioavailability and the hydrocarbon narcosis model
    b. Assist the SCBA in securing final ASTM approval of the SPME pore water method
    c. Transfer the technology to Navy RPMs and risk assessors

6.1 Confirm the presence of impacted sediments at the site that are toxic to the aquatic test organisms

The presence of sediments containing PAHs at concentrations having impact on an aquatic test species was tested by conducting a standard 28-day toxicity evaluation using the amphipod *Hyalella azteca*. Aquatic toxicity text endpoints included chronic (survival) and acute (growth) endpoints. The goal was to obtain sediment samples from the area adjacent to the WNY which exhibited a range of impacts to the aquatic test species. Of the fifteen studied sediment samples, one (AR02) exhibited a reduced survival and one (AR03) exhibited reduced growth. These data were used to construct dose-response (i.e., toxicity versus PAH concentration [on a TU basis]) figures. The relationship between *H. azteca* survival and TU PAH_{34} is shown in Figure 5-4. The data are consistent with the probit fit (dose-response relationship) that was been developed using the sediment samples collected during the SCBA project.

6.2 Confirm the presence of sediment PAHs in the biologically-active zone of the site

The WNY site was selected based on past characterization data which indicated 1) the presence of PAHs at a concentrations exceeding the probable effects concentration (PEL) of 22.8 mg/kg, and 2) the lack of other constituents which might cause a negative impact on the benthic community in surficial sediment samples. The bulk sediment PAH_{16} concentrations ranged from 14 mg/kg to 600 mg/kg in the 15 samples used for this study (Table 5-7). The range of PAH values above and below the PEL allowed for an analysis of whether sediments containing PAHs exceeding the PEL are truly toxic to freshwater amphipods.

6.3 Validate the use of the analytical method to estimate the bioavailability of sediment-bound PAHs by predicting aquatic toxicity

Pore water PAHs were directly analyzed on the final 15 sediment samples. Individual PAH pore water concentrations were divided by their Final Chronic Values (FCV) to generate a toxic unit (TU) for that PAH in each sample (i.e., PAH concentration/FCV = TU). The TUs for individual PAHs were summed for each sediment sample (18 parent and 16 alkylated PAHs) to arrive at a total PAH TU (i.e., TU_{34}). Total PAH TUs were plotted against percent survival of the *H. azteca*. The resulting relationship was consistent with the previously collected data from other PAH-contaminated sites, and conformed to a probit dose-response relationship, with the result that the direct pore water analysis was able to correctly identify toxicity to *H. azteca* (Figure 5-4). The WNY data presented in this report follow the expected outcomes from the U.S. EPA publications concerning equilibrium sediment benchmarks for PAHs (U.S. EPA, 2003; U.S. EPA, 2009), in that direct measures of pore water PAHs are more representative of biological effects than bulk sediment concentrations and/or predictions of pore water concentrations using the equilibrium partitioning model.

This study confirms that the analytical protocol developed by the SCBA program, and used in this demonstration, is a valuable tool for evaluating protection to benthic resources.

6.4 Develop a tiered, risk-based approach to management of PAH-impacted sediment based on direct chemical measures of PAH bioavailability and the hydrocarbon narcosis model

ESTCP provided partial funding, in conjunction with funding provided by the SCBA industry partners, for several activities related to the implementation of a risk-based approach to PAH sediment management using the direct pore water method.

First, the Interstate Technology Regulatory Council (ITRC) was funded to identify five state regulators who formed a Regulatory Advisory Team to provide insights from the regulatory community concerning the acceptance of the SPME method. The regulators were:

- Gary Buchanan — New Jersey DEP
- Paul Craffey — Massachusetts DEP
- Gardiner Cross — New York DEC
- Brad Helland — Washington DOE
- Jennifer Peterson/Angie Obery — Oregon DEQ

Two face-to-face meetings were held with the ITRC regulators, as well as two conference calls. No written output was expected, in order to minimize the regulator's time commitment. The results of the discussions indicated a general willingness by the regulators to accept a tiered risk-based approach for evaluating PAH impacts in sediments, where the direct pore water method could be used to predict aquatic toxicity after a site-specific correlation of pore water PAHs and aquatic toxicity test results had been determined. This site-specific correlation is developed by simultaneously characterizing pore water concentrations and toxicity of 20 to 30 sediments and using these results to generate a dose-response curve for the site. In addition, the ITRC regulators indicated that that they would like to see both acute and chronic aquatic toxicity data (i.e., survival and growth), as well as the use of more than one benthic species used for the aquatic toxicity test.

Second, in the Fall of 2007, the EPA listed a Federal Register Notice [Federal Register Volume 72, No. 41, Friday March 2, 2007 pp. 9522-9523] with a 30-day public comment period on the draft document titled *Evaluating Ecological Risk to Invertebrate Receptors from PAHs in Sediments at Hazardous Waste Sites* [EPA/600/R-06/162]. This document was intended as an addendum to the EPA ESB PAH document (U.S. EPA, 2003). The draft document did not include any relevant information on the effect of anthropogenic carbon on the disconnect between pore water estimated using equilibrium partitioning and a direct analysis of pore water. The members of SCBA submitted a 100 page response which presented the results of the SPME pore water method and its ability to accurately predict effects to benthic organisms in aquatic toxicity tests, as well as literature to support the claim that equilibrium partitioning using default partitioning coefficients is not able to accurately estimate PAH pore water concentrations in the presence of anthropogenic carbon. The U.S. EPA completed a total revision of the document, which was published in 2009 as *Evaluating Ecological Risk to Invertebrate Receptors from PAHs in Sediments at Hazardous Waste Sites* (EPA-600-R-06-162F). The recommendations of this publication include a tiered site-specific evaluation of sediment PAHs, where direct pore water analysis was specified one way to estimate bioavailability (see Figure 1-1 in Section 1.0 of this report).

6.5 Assist the SCBA in securing final ASTM approval of the SPME pore water method

The provisional ASTM method is required to undergo a more exhaustive inter-laboratory evaluation before it can be designated as an ASTM standard method. The interlaboratory study required the generation of a minimum of seven independent analytical datasets on a set of control samples that are prepared and managed by a custodial laboratory. These data are used to analyze the precision and bias of the method, which are used as metrics to determine if the method warrants designation as a standard method. ESTCP provided partial support for this ASTM approval process. This funding was used to set up the program and identify participating laboratories. As of the writing of this report, four laboratories (Meta Environmental, Inc., Test America, Inc., Alpha Woods Hole, and the University of Maryland – Baltimore County) have been selected and are conducting concurrent analyses of the study samples. Each lab is providing two independent operators, which will yield a total of eight analytical datasets. The current schedule has the sample analyses completed in May 2010 and the initial presentation of the data to ASTM in June 2010. ASTM will initiate the balloting of the method and it is anticipated that the method will become an ATM standard method as of January or June 2011.

6.6 Transfer the technology to Navy RPMs and risk assessors

This performance objective is divided into three parts:

- Technical presentations
- Technical/regulatory guidance
- Development of a Web-based tool

Technical presentations using the demonstration data were made to the Navy Risk Assessment Work Group (RAW) and the TriServices Environmental Risk Assessment Work Group (TSERAW). The demonstration was presented to the Washington Navy yard RPM (Armalia Berry-Washington) in two phases: 1) Phase 1: Demonstration workplan and 2) Phase 2: Formal presentation of project results. In addition, the workplan and project results were presented to the Anacostia Watershed Toxics Alliance (AWTA) on two separate occasions. The AWTA is an EPA-led organization of stakeholders concerned with the environmental health of the Anacostia River (http://www.epa.gov/oswer/onecleanupprogram/anacostia.htm).1)

The following presentations on this study were made:

Work Plan Presentations:

- Determination of Sediment Polycyclic Aromatic Hydrocarbon (PAH) Bioavailability Using Supercritical Fluid Extraction (SFE) and Ultra-Trace Pore water (UTP) Analysis. Tri-Service Environmental Risk Assessment Work Group. January 17, 2007. NAVBASE Ventura County – Port Hueneme, CA. Oral presentation by S. C. Geiger.
- Understanding and Using PAH Bioavailability Data for Effective Management of Impacted Sediments. Anacostia Watershed Toxics Alliance (AWTA) Annual Meeting. June 25, 2007. Washington, DC. Oral presentation by S.C. Geiger.
- The Bioavailability of PAHs in the Anacostia River. July 12, 2007. Washington Navy Yard Tier 1 Partnering Team Meeting. Washington DC. Oral presentation by S.C. Geiger.
- Determination of Sediment Polycyclic Aromatic Hydrocarbon (PAH) Bioavailability Using Supercritical Fluid Extraction (SFE) and Ultra-Trace Pore water (UTP) Analysis. SERDP-ESTCP Symposium. November 9, 2007. Poster presentation by D.V. Nakles.

Project Result Presentations:

- Determination of Sediment Polycyclic Aromatic Hydrocarbon (PAH) Bioavailability Using Supercritical Fluid Extraction (SFE) and Ultra-Trace Pore water Analysis. ESTCP Internal Project Review (IPR). Arlington, VA. February 3, 2008. Oral presentation by D.V. Nakles.
- The Bioavailability of PAH Compounds in Sediments of the Anacostia River. Washington Navy Yard Tier 1 Partnering Team Meeting. Washington DC. March 5, 2008. Oral presentation by S.C. Geiger.
- Enhanced SPME Pore water Method for Characterizing Actual PAH Sediment Bioavailability. Association of State and Territorial Solid Waste Management Organizations (ASTSWMO) State Superfund Managers Symposium. July 29, 2008. Phoenix, AZ. Oral presentation by S.C. Geiger.
- The Determination of Sediment Polycyclic Aromatic Hydrocarbon (PAH) Bioavailability using Direct Pore Water Analysis by Solid-Phase Microextraction (SPME) (ESTCP Project ER-0709). SERDP-ESTCP Bioavailability Workshop. Annapolis, MD. August 20, 2008. December 4, 2008. Poster presentation by D.V. Nakles.
- The Characterization of PAH Bioavailability in Sediments at the Washington Navy Yard Anacostia Watershed Toxics Alliance (AWTA) Annual Meeting. October 28, 2008. Washington, DC. Oral presentation by S.C. Geiger.
- Applying Pore-Water Measurements to Decision-Making for PAH-Impacted Sediments". Society for Environmental Toxicity and Chemistry (SETAC) North America 2008 Conference. November 19, 2008. Tampa, FL. Oral presentation by S.C. Geiger.
- The Determination of Sediment Polycyclic Aromatic Hydrocarbon (PAH) Bioavailability

using Direct Pore Water Analysis by Solid-Phase Microextraction (SPME) (ESTCP Project ER-0709). SERDP-ESTCP Symposium. Washington DC. December 4, 2008. Poster presentation by S.C. Geiger.

The final study report contains information that will serve as a generic work plan, QAPP, and technical / regulatory guidance that can be used as a template for applying the technology to for other PAH-contaminated Navy sites.

A web-based tool to facilitate access to the project results for Navy personnel will be developed by NFESC, using this final dem/val report as a guide. The web-based tool will undergo development pending final acceptance of this demonstration report by the ESTCP Program Office.

7.0 COST ASSESSMENT

Cost information for implementing the bioavailability assessment for sediment-bound PAHs is included in this section.

7.1 Cost Model

Because the demonstration concerned a higher-level phase of analysis, the cost assessment assumes that a Phase 1 site assessment has been conducted (i.e., bulk sediment chemistry has been determined, and a reasonable estimate that PAHs are the main driver of risk at the site is assumed). The evaluation of Phase 1 or historical data should be made to indicate that bulk sediment PAH concentrations exceed screening level values (i.e., sediment quality guidelines/screening levels and/or PAH ESBs calculated from bulk sediment concentrations). At that point, a decision needs to be made whether to continue to characterize PAH bioavailability at the site (i.e., conduct a Tier 2 and/or Tier 3 analysis).

The cost assessment follows the Tiered site characterization process presented in EPA 2009 (see Figure 1-1). The Tier 2 process (direct analysis of pore water PAHs) and Tier 3 process (aquatic toxicity testing) are included in the cost assessment. It is also assumed that the sediment samples for the Tier 2 and Tier 3 analyses will be collected at the same time (i.e., Tier 2 pore water data will be determined within the holding times for aquatic toxicity testing).

Because the demonstration validated a laboratory method, there were no capital costs for the project.

7.2 Cost Drivers

The cost model does not include costs for conducting a toxicity identity evaluation (TIE), as it assumes that a Tier 1 analysis has already identified the risk drivers at the site. However, under the following condition a TIE might be warranted after conducting a Tier 2/Tier 3 assessment: if the SPME pore water TUs < 1, but there is a significant decrease in survival/growth of one or more of the aquatic test species that cannot be attributed to a NAPL phase or adverse sediment conditions (i.e., gravelly or sandy textures).

The cost model assumes that only 24 sediment samples are necessary for a Tier 2/Tier 3 assessment. This number of samples may have to be adjusted upward or downward depending on the site. Costs for implementing the SPME pore water PAH analysis for MNR, or for capping design/integrity will vary depending on the number of samples and sampling frequency desired.

The cost model assumes that sampling depths, access to the site, and water/weather conditions are such that the costs quoted are applicable. Excess depths (i.e., > 50 to 100ft) requiring specialized sampling equipment, large distances from the docking area to the site that require extra time and fuel costs, and/or adverse weather conditions requiring an extended stay at the site will need to be factored into any final cost.

The cost model does not include labor hours spent on regulatory interaction, as this will be site, state, and EPA-region specific.

Table 7-1. Cost Model for a Tier 2 and Tier 3 PAH Bioavailability Study.

Cost Element	Task	Costs	
Project Set-up and Planning	Historical data review and project planning	Project manager $150/hr x 60 hr	$9,000
		Technician $100/hr x 40 hr	$4,000
Sediment Sample Collection (costs adjusted to 3-day effort)	Mob/Demob		$3,000
	Boat and Captain	$950/day x 3 days	$2,850
	Travel costs	$1,400/person x 3 persons	$4,200
	Equipment rental	Ponar dredge ($50/day)	$150
		Differential GPS ($50/day)	$150
	Field equipment		$2,300
	Labor	Field technicians (2) x 3 days x $100/hr	$4,800
	Shipping		$800
Porewater and Sediment Analysis	Bulk sediment PAHs TOC/SOC SPME porewater PAHs	24 samples x $1,600/sample	$38,400
Genraly Chemical and Physical Properties Analysis	Grain size, total solids, NH_3, pH	24 samples x $160/sample	$3,840
Aquatic toxicity testing	Survival and growth of two (2) benthic test species	24 samples x 2 species x $1,800/sample	$86,400
Project Management	Project management	PM $150/hr x 80 hrs	$12,000
	Data validation	Technician $100/hr x 16 hrs	$1,600
	Data analysis	Technician $100/hr x 80 hr	$8,000
Operating Costs (Indirect Environmental Costs)	Overhead	Office supplies and support	$1,400
		Total Project Cost	$182,890

Field equipment may include: glass sampling jars, 5-gallon pails with lids, estimated cell phone charges, health and safety supplies, ice, shipping tape, paper towels, plastic wrap, PID, sieve screens, and photographic record supplies.

Note that Aquatic toxicity test costs are for two species. Using only one test species would reduce overall costs by $43,200.

7.3 Cost Analysis

Since the demonstration represents a case study that would be conducted at any contaminated

sediment site, there is no increased scale of operations for applying this technology, other than increased costs for extra sample collection and analysis.

The SPME pore water PAH method is a laboratory-based site characterization tool, and therefore there are no associated lifecycle costs.

7.3.1 Ideal site description

The site conditions which are applicable to the SPME pore water PAH analysis are included in the site selection criteria table (see Table 4-1). Any gross deviations from the site selection criteria can be expected to either add significant costs, or to invalidate the method.

7.3.2 Cost analysis assumptions

The main assumption to be made when estimating the costs of implementing the SPME pore water PAH analysis approach is how much data is needed to fully evaluate the site. For example, if the site regulators will accept the direct pore water analysis concentrations (i.e. Tier 2 analysis) as the final arbiter of site conditions, there will be no need to conduct aquatic toxicity testing. However, if there is still some uncertainty as to whether PAHs are the main risk drivers at the site, an aquatic toxicity test will most likely need to be conducted with one or two test species.

7.3.3 Cost Comparison

Alternative technologies to which the SPME pore water PAH analysis can be compared are other methods for estimating PAH bioavailability in sediments, such as polyethylene strips (PE) and *in-situ* SPME. Analytical costs associated with these methods will be similar to the SPME direct pore water analysis method. However, if these methods are conducted in the field, then at least one extra mob/demob and sampling trip will need to be conducted. If these methods are conducted in the laboratory, then adequate time for equilibration of the PE or *in-situ* SPME fibers with the sediment will need to be accounted for.

8.0 IMPLEMENTATION ISSUES

Permits and permission to sample sediments in the Anacostia River were obtained for the site before the scheduled sampling event. A sampling permit was obtained from the U.S. Army Corp of Engineers. Once that permit was obtained, it was reviewed and certified by the Washington DC Department of Water Quality. Permission for sampling was also obtained from the U.S. National Park Service. The complexity of the permitting process required three months for final acceptance by the appropriate regulatory agencies, and therefore time must be budgeted for future sediment sampling projects on the Anacostia River.

The U.S. National Park Service operates a public marina at Buzzard's Point in Washington DC. The marina is located about one-half mile downstream of the WNY site. The U.S. National Park Service graciously allowed the use of the marina for docking the sampling boat as well as staging a sample process station.

The publication of the EPA white paper entitled *Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: PAH Mixtures* (EPA-600-R-02-013) established the effectiveness of using direct PAH pore water measurements when assessing the effects of sediment-bound PAHs to benthic organisms. However, many state and federal agencies still require aquatic toxicity tests be conducted alongside of pore water analysis before making site decisions. Therefore, the acceptability of the SPME direct PAH pore water method to regulatory agencies will need to be negotiated on a site-by-site basis. The upcoming Interstate Technology Regulatory Council (ITRC) document on contaminated sediment bioavailability should help regulators understand the use of bioavailability measurements for site assessment and characterization.

The SPME direct pore water method has been accepted as an EPA SW-846 method SW-8272. It has also been given an ASTM provisional method designation (D-7363-07). As of the writing of this report, the round-robin laboratory testing required for full ASTM method establishment is underway. The pore water analysis for this study was conducted in a combination research/commercial analytical laboratory (EERC). Other research laboratories have conducted this analysis (UMBC, University of New Hampshire). There are three commercial laboratories are participating in the ASTM method approval (Test America, Alpha Woods Hole, and META) and therefore are able to provide this analytical service. It is hoped that other commercial laboratories will provide analytical services on this method once the full ASTM method assignation is provided.

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Appendix A: Points of Contact

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Point of Contact	Organization	Phone/Fax/e-mail	Role in Project
Dr. Stephen Geiger	AECOM 675 N. Washington Street Suite 300 Alexandria, VA 22314	T: (703)297-9118 F: (703)706-9409 steve.geiger@aecom.com	Final PI; project management, data analysis, writing the final reports
Dr. David Nakles	Dept. of Civil and Environmental Engineering Carnegie Mellon University 5000 Forbes Avenue Pittsburgh, PA 15213-3890	T: (412) 268-5280 F: (412) 268-7813 dnakles@andrew.cmu.edu	Original PI; project management, sediment sample collection, and liaison with the SCBA
Ms. Amy Hawkins	Naval Facilities Engineering Service Center (NFESC) Consultation/Information Management Branch 1100 23rd Ave. Port Hueneme, CA 93030	T : (805) 982-4890 amy.hawkins@navy.mil	Co-PI; project management and transfer of the technology to the DoD
Dr. Todd Bridges	U.S. Army Engineer Research and Development Center (ERDC) Waterways Experiment Station (EP-R) 3909 Halls Ferry Rd. Vicksburg, MS 39180-6199	T: (601) 634-3626 F: (601) 634-3713 Todd.S.Bridges@erdc.us.	Aquatic toxicity testing of the sediment
Dr. Steven Hawthorne	Energy and Environmental Research Center (EERC) University of North Dakota PO Box 9018 Grand Forks, ND 58202-9018	T: (701) 777-5000 F: (701) 777-5181 shawthorne@undeerc.org	Total and SPME porewater PAH analysis and detailed chemical characterization of sediments
Dr. Upal Ghosh	Department of Civil and Environmental Engineering (UMBC) University of MD Baltimore County 5200 Westland Blvd. Baltimore, MD 21250	T: (410) 455-8665 F: (410) 544-6500 ughosh@umbc.edu	Technical lead on the characterization of carbon chemistry
Mr. David Thal	Environmental Standards, Inc. 1140 Valley Forge Road P.O. Box 810 Valley Forge, PA 19482	T : 610.935.5577 F : 610.935.5583 dthal@envstd.com	Standard physical and chemical analysis of sediments

Appendix B: EPA SW-8272 method

METHOD 8272

PARENT AND ALKYL POLYCYCLIC AROMATICS IN SEDIMENT PORE WATER BY SOLID-PHASE MICROEXTRACTION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY IN SELECTED ION MONITORING MODE

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generation its own detailed standard operating procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 The U.S. Environmental Protection Agency (USEPA) narcosis model for benthic organisms in sediments contaminated with polycyclic aromatic hydrocarbons (PAHs) is based on the concentrations of dissolved PAHs in the interstitial water or pore water in sediment. Method 8272 covers the separation of pore water from PAH-impacted sediment samples, the removal of colloids, and the subsequent measurement of dissolved concentrations of the 10-parent PAHs and two alkylated daughter PAHs in the pore water samples. This method directly determines the concentrations of dissolved PAHs in environmental sediment pore water, groundwater, and other water samples. The following polycyclic aromatic hydrocarbons (PAHs) have been determined by this method and other PAH compounds may also be amenable to analysis by this method:

Analyte	CAS No[a]
Naphthalene	91-20-3
2-Methylnaphthalene	91-57-6
1-Methylnaphthalene	90-12-0
Acenaphthylene	208-96-8
Acenaphthene	83-32-9
Fluorene	86-73-7
Phenanthrene	85-01-8
Anthracene	120-12-7
Fluoranthene	206-44-0
Pyrene	129-00-0
Benz(a)anthracene	56-55-3
Chrysene	218-01-9

[a]: Chemical Abstract Registry Number

NOTE: Method 8272 is specifically for the determination of dissolved PAHs in interstitial water or pore water in sediment samples only. If the heavy molecular weight PAHs that may be present in the particulates are of concern, additional determinative extraction and analysis methods are required to measure suspended and sediment-based (i.e., total) PAHs.

Regulatory methods using solvent extraction have not achieved the wide calibration ranges from nanograms to milligrams per liter and the necessary levels of detection in the nanogram per liter range. In addition, conventional solvent extraction methods require large aliquot volumes (liter or larger), the use of large volumes of organic solvents, and filtration to generate the pore water. Solvent extraction entails the storage and processing of large volumes of sediment samples and may result in the loss of low molecular weight PAHs in the filtration and solvent evaporation steps.

This method can be used to determine nanogram to milligram per liter PAH concentrations in pore water. Small volumes of pore water are needed for solid phase microextraction (SPME), only 1.5 mL per determination, and virtually no solvent extraction waste is generated.

1.2 Lower molecular weight PAHs are more water soluble than higher molecular weight PAHs. Therefore, PAH concentrations in pore water samples vary widely due to differing saturation water solubilities that range from 0.2 µg/L for indeno[1,2,3-cd]pyrene to 31,000 µg/L for naphthalene. This method can accommodate the measurement of milligram per liter concentrations for low molecular weight PAHs and nanogram per liter concentrations for high molecular weight PAHs, such as benz(a)anthracene and chrysene and any other four-five ring PAHs that can be determined by this method.

1.3 This method can achieve the necessary lower limits of quantitation, which range from approximately 0.06 µg/L for high molecular weight PAHs, to approximately 9 µg/L for low molecular weight PAHs.

1.4 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600 and 8000). For additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance, analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography/mass spectrometers and skilled in the

interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Pore water is separated from wet sediment samples by centrifugation and supernatant collection. The groundwater and tap water samples begin preparation with the colloid removal step. Colloids are removed from the separated pore water, groundwater, and tap water samples by flocculation with aluminum potassium sulfate (alum) and sodium hydroxide. A second flocculation and centrifugation step, followed by supernatant collection, completes the colloid removal.

2.2 The PAHs are determined using SPME followed by gas chromatography/mass spectrometry (GC/MS) analysis in selected ion monitoring (SIM) mode. Either the use of an autosampler, or a manual approach can be used to perform the SPME extraction and the subsequent injection of collected analytes into the GC/MS. Isotopically labeled analogs of the target compounds are introduced prior to the extraction, and are used as quantitation references.

2.3 The mass spectrometer is operated in the SIM mode for the molecular ions of the target PAHs and d-PAHs to achieve low limits of detection. Analyte concentrations are quantitated by either of two methods: (1) parent PAHs (i.e., unsubstituted PAHs) for which an exact deuterated analog is not included in the internal standard mix are quantitated by reference to a deuterated analog of a PAH with the same number of rings as the analyte, or (2) PAHs for which an exact deuterated analog is included in the internal standard mix are quantitated by isotope dilution.

2.4 Test Method Options: Either the use of an autosampler or a manual approach may be used to perform the SPME extraction and the subsequent injection of collected analytes into the GC/MS. An autosampler is much preferred over the manual method because: (1) the autosampler yields lower and more reproducible blanks, (2) the manual method requires the use of a stir bar that can cause sample cross-contamination, (3) the manual method is highly labor-intensive and requires multiple timed manipulations per analysis leading to operator fatigue and resultant errors, and (4) the autosampler reduces the technician time required to prepare samples for a 24-hour run sequence to approximately 3 hours, while the manual method requires 24 hour operator attendance. Therefore, the method procedures are written assuming the use of an autosampler, with modifications to the autosampler procedures listed for the manual method.

2.4.1 Autosampler Method

2.4.1.1 Pore Water Separation and Preparation: Pore water is separated from wet sediment samples by centrifugation and supernatant collection. The groundwater and tap water samples begin preparation with the colloid removal step. Colloids are removed from the separated pore water, groundwater, and tap water samples by flocculation with aluminum potassium sulfate (alum) and sodium hydroxide. A second flocculation and centrifugation, followed by

supernatant collection completes the colloid removal. The prepared water samples are then split into the number of replicate aliquots needed and placed into silanized glass autosampler vials. The 8 perdeuterated PAH internal standards (d-PAHs) are then added immediately. All of the water preparation steps beginning with the centrifugation and ending with the addition of d-PAH internal standards should be conducted continuously and in the minimum amount of time possible.

The SPME fiber should be cleaned at the beginning of each sampling set (and after very contaminated samples) while the water samples are being prepared.

2.4.1.2 Solid-Phase Microextraction: The SPME extraction of the water samples is performed using a commercially available polydimethylsiloxane (PDMS)-coated fused silica fiber while the water sample is mixed by the precession of the autosampler mixing chamber. The target PAHs and d-PAH internal standards adsorb to the nonpolar PDMS phase at equivalent rates. The use of the d-PAHs (i.e., isotopic dilution) to quantitate the target PAHs compensates for variations in equilibrium partitioning and kinetics.

2.4.1.3 GC/MS SIM Analysis: Following the sorption period, the SPME fiber is immediately desorbed to a GC/MS injection port in the splitless mode. Following the desorption period, the SPME fiber is inserted into the cleaning port and additionally cleaned. At the end of the cleaning period, sorption of the next water sample is begun.

2.4.2 Manual Method

Alternate Procedures for Manual Method: Samples are prepared as for the autosampler method, except that a small Teflon-coated stir bar is placed in the silanized autosampler vial prior to adding the water and d-PAH internal standard solution. A new stir bar should be used for each sample, calibration standard, and blank to avoid cross-contamination caused by carryover on the stir bar. To perform the SPME step, the vial is set on a stir plate and the stirring rate adjusted so that no large vortex is formed. The SPME fiber should be inserted into the water so that the entire-active length is exposed to the water sample, but not so low that the fiber comes into contact with the stir bar or that the metal needle sheath contacts the water. All time sequences should be the same as described for the autosampler method. A spare GC split/splitless injection port under helium flow can be used for the cleaning step between samples as well as for the initial cleaning step at the beginning of each working day.

2.5 This method includes specific calibration, sample analysis, and quality control steps that supersede the general requirements provided in Method 8000.

3.0 DEFINITIONS

Refer to the SW-846 Chapter One, Chapter Four and appendix of terms and definitions for potentially applicable definitions.

3.1 Data Acquisition Parameters – Parameters affecting the scanning operation and conversion of the analytical signal to digitized data files. These include the configuration of the ADC circuitry, the ion dwell time, the MID cycle time, and acquisition modes set up for the method. Examples of acquisition modes for the HP5973 include SIM mode and Low Mass Resolution Mode.

3.2 Lower Limit of Quantitation – The lower limit of quantitation (LLOQ) for each individual PAH is defined as the concentration of an individual PAH that would yield 1/34 of a toxic unit (see Sec. 3.4). Due to the differences in saturation solubilities for the PAH compounds recommended in this method the LLOQ will also vary for each compound. Ideally, the exact LLOQ should be at or below those recommended in Table 3 for optimum method performance. However, the actual LLOQs should be a project planning decision based on the desired project-specific data quality objectives. For example lower limits of quantitation of each individual PAH refer to Table 3. See Ref. 2 in Sec. 16.0 for additional details.

3.3 Solid Phase Microextraction (SPME): Solid phase microextraction has been used for the determination of PAHs in water samples. SPME utilizes a commercially available 7 micron thick polydimethylsiloxane (PDMS)-coated fused silica fiber. The target PAHs and d-PAH internal standards adsorb to the nonpolar PDMS phase at equivalent rates. PAHs are extracted onto the fiber from the sediment pore water for 30 minutes before they are desorbed into the GC/MS injection port.

3.4 Toxic Units - Using contaminated site sediment PAH concentrations and sediment organic carbon content, equilibrium partitioning (EqP) is used to predict the pore water concentrations of the PAHs, which are equivalent to the bioavailable concentrations for the hydrocarbon narcosis model. Alternatively, the pore water concentrations can be directly measured to more accurately determine the bioavailable concentrations than is possible using sediment concentrations and the EqP model. The analyte list in Sec. 1.1 can be expanded as described in Refs. 2 and 7 to include the alkyl PAHs necessary to calculate total toxic units based on hydrocarbon narcosis theory. For a more detailed discussion of toxic units and their application see Ref. 6 in Sec. 16.0.

4.0 INTERFERENCES

4.1 Non-target hydrocarbons can cause peaks on selected ion current profiles (SICPs) intended for other PAHs. Analysts should be familiar with both parent and alkyl PAH analyses in complex environmental samples.

4.2 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferences under the conditions of analysis by performing laboratory method blanks.

Analysts should avoid using PVC gloves, powdered gloves, or gloves with measurable levels of phthalates. The use of high purity reagents and solvents helps minimize interference problems.

4.3 For lower molecular weight PAHs, atmospheric contaminants can cause significant background peaks. This problem is most likely to be significant in urban areas impacted by atmospheric PAHs (e.g, from diesel exhaust), and with laboratories using manual techniques, rather than the SPME autosampler.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

This section does not list common laboratory glassware (e.g., beakers and flasks).

The mention of trade names or commercial products in this method is for illustrative purposes only, and does not constitute an EPA endorsement of exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance for the intended application has been demonstrated and documented.

6.1 Centrifuge capable of sustaining 1000 g with cups for securing 40-mL and 20-mL vials.

6.2 SPME fiber holder compatible with 7-µm SPME fiber and compatible with either the autosampler or the manual method.

6.3 SPME fused silica fibers coated with 7 µm film thickness polydimethylsiloxane (PDMS) from Sigma-Aldrich (formerly Supelco®) or equivalent.

6.4 PTFE coated stir bars (stir fleas) of a size effective for stirring 1.5 mL water without vortexing (for manual method only).

6.5 Magnetic stir plate (for manual method only).

6.6 SPME holder stand (for manual method only) or GC/MS autosampler capable of SPME extraction and injection (LEAP Technologies Combi-Pal or equivalent).

6.7 Cleaning port, capable of purging SPME fibers in a helium-swept atmosphere at 320 °C.

6.8 40-mL vials with Teflon-lined caps.

6.9 20-mL vials with Teflon-lined caps.

6.10 Silanized 2.0-mL autosampler vials.

6.11 GC/MS Analysis

6.11.1 Gas Chromatograph – Shall have split/splitless injection port for capillary column, temperature program with isothermal hold.

6.11.2 GC column – 60 m x 0.25 mm ID x 0.25 μm film thickness HP5-MS or equivalent. The column listed in this section was the column used in developing the method. The listing of this column in this method is not intended to exclude the use of other columns that are available or that may be developed. Laboratories may use this or another column provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

6.11.3 Inlet liner 2 mm i.d. silanized glass.

6.11.4 GC inlet 320 °C, splitless mode.

6.11.5 Oven program: Isothermal 5 minute hold at 40 °C. Ramp at 50 °C /minute to 110 °C, followed by a temperature ramp of 12 °C/minute to 320 °C (Hold for 10 min).

6.11.6 Mass Spectrometer – Electron impact ionization with the ionization energy optimized for best instrument sensitivity (typically 70 eV), stability and signal to noise ratio. Shall be capable of repetitively and selectively monitoring at least 12 separate m/zs during a period of approximately 1 second.

6.11.7 Data System – A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectral information obtained throughout the duration of the chromatographic program. The computer should have software that can search any GC/MS data file for the SIM ions collected during each time window and that can plot such ion abundances.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is sufficiently high purity to permit its use without lessening the accuracy

of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

7.2 Reagent water: Deionized water, free of the analytes of interest. Water that meets the purity specifications of HPLC-grade water, or equivalent.

7.3 Internal standard stock solution. A dichloromethane solution of d-PAH internal standards used for preparing spiking solutions by dilution into acetone (see Sec. 11.3).

7.4 Internal standard spiking solution. A dilution of the internal standard stock solution in acetone used to spike d-PAH internal standards into all sample, calibration, and blank water vials (see Sec. 11.3).

7.5 Calibration stock solution. A dichloromethane solution of PAHs used for preparing calibration standards (see Sec. 11.3).

7.6 Calibration Spiking Solutions. A series of solutions prepared by diluting the calibration stock solution with acetone (see Sec. 11.3).

7.7 Calibration Standards. Prepared by adding internal standard and calibration spiking solutions in reagent water (see Sec. 11.3).

7.8 Acetone (CH_3COCH_3)

7.9 Dichloromethane (DCM) (CH_2Cl_2)

7.10 Sodium Hydroxide (NaOH). 1 M NaOH - Slowly add 10 g NaOH pellets to 125 mL reagent water and stir until completely dissolved. Fill the flask to 250 mL. Wear goggles and be aware of heat of solution. Store in a plastic container.

7.11 Aluminum Potassium Sulfate Dodecahydrate ($AlK(SO_4)_2 \cdot 12H_2O$).

7.12 Alum Solution: Add 20g ($AlK(SO_4)_2 \cdot 12H_2O$) to 80 mL reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See the introductory material to Chapter Four, "Organic Analytes." Prior to shipment, the sediment samples should be mixed well. Sieve the slurry of sediment and site water through a 2-mm screen to remove debris. If the sieved slurry is to be stored or shipped before use, store in 250-mL to 1-L glass jars fitted with PTFE-lined lids. Great care must be taken to clean the lid of the jar before capping the jar with the lid to avoid leakage of the water during shipment. Groundwater and tap water samples should be stored in 250-mL to 1-L glass bottles fitted with PTFE-lined caps.

8.2 Ship samples in an ice chest with adequate ice to maintain 0-6 °C. Store the samples at the laboratory in the dark at 0-6 °C. Do not allow the samples to freeze.

8.3 Once the sample preparation process has begun, pore waters must be generated and flocculated as quickly as possible, but must be done within 28 days of

sediment sample collection. Pore water, groundwater, and tap water samples must then be immediately spiked with 10 µL of d-PAH solution following flocculation.

8.4 Solid phase micro-extraction must be completed within 24 hours of flocculation for pore water, groundwater, and tap water samples.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a quality assurance project plan (QAPP) or a sampling and analysis plan (SAP), which translates project objectives and directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference and inspection.

9.2 Refer to Method 8000 for specific determinative method QC procedures. Refer to Method 3500 for QC procedures to ensure the proper operation of the various sample preparation techniques. If an extract cleanup procedure is performed, refer to method 3600 for the appropriate QC procedures. Any more specific QC procedures provided in this method will supersede those noted in Methods 8000, 3500, or 3600.

9.3 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. Any more specific QC procedures provided in this method will supersede those noted in Method 8000.

9.4 Initial demonstration of proficiency

Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes by generating data of acceptable accuracy and precision for target analytes in a clean matrix. Analyze seven replicates, or any other suitable number based on the project planning objectives, of an initial demonstration of performance (IDP) solution. The IDP solution is a reagent water or field sample matrix solution fortified with the method analytes and internal standards at known concentrations. Ideally, the IDP solution should be prepared by an independent analyst. The mean and standard deviation of the seven values should then be calculated and compared to the test method accuracy and precision guidance values in Sec. 13.0.

If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions. The laboratory must also repeat the demonstration of proficiency whenever new staff is trained or significant changes in instrumentation are made. See

Method 8000 for further information on how to accomplish a demonstration of proficiency.

9.5 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are free from contaminants and interferences. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed in the retention time window of any analyte that would prevent the determination of the analyte, determine the source and eliminate it, if possible, before processing any samples. The blanks should be carried through all stages of sample preparation and analysis. When new chemicals or reagents are received, the laboratory should monitor the preparation and/or analysis blanks associated with the samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents. This initial and continuing monitoring is accomplished through the analysis of extraction and analytical method blanks analyzed between every calibration verification standard and sample to monitor the baseline. See Table 4. Target analytes must not be detected above 1/3 of the lower limits of quantitation or greater than 20% of the associated sample result(s).

Should the acceptance criteria not be met for any extraction and analytical blank, locate the source of the contamination and correct the problem. Re-extract and reanalyze the associated samples that are less than ten times the level of the contaminant(s) present in the method blank.

9.6 Sample quality control for preparation and analysis

The laboratory must also have procedures for documenting the effect of the matrix on method performance (i.e., precision, accuracy, method sensitivity). At a minimum, this should include the analysis of QC samples including a method blank, a matrix spike, a duplicate and a laboratory control sample (LCS) in each analytical batch. The use of deuterated analogs as internal standards makes the addition of surrogates unnecessary. Any method blanks, matrix spike samples, and replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples.

9.6.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, laboratories may use a matrix spike and duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike/matrix duplicate pair. See Method 3500 for instructions on preparing the matrix spike standard. The same standard may be used as the laboratory control standard (LCS) and the spiking solution should be the same source as used for the initial calibration standards to restrict the influence of standard accuracy on the determination of

recovery through preparation and analysis. Consult Method 8000 for information on developing acceptance criteria for the MS/MSD.

9.6.2 A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, where appropriate. As noted in Sec. 9.6.1, the LCS is ideally prepared from the same source stock standard that is used to prepare the calibration standards. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Consult Method 8000 for information on developing acceptance criteria for the LCS.

9.6.3 Also see Method 8000 for the details on carrying out sample quality control procedures for preparation and analysis. In-house method performance criteria for evaluating method performance should be developed using the guidance in Method 8000.

9.7 Initial Calibration. The following acceptance criteria must be used for initial calibration: (i) The signal to noise (S/N) ratio for the GC signals present in every SICP must be $\geq$ 10:1 for the labeled internal standards and calibration compounds; (ii) The percent relative standard deviation (RSD) for the mean area ratio/ng for labeled internal standards and the calibration compounds must be less than 30% for high molecular weight PAHs and less than 25% for low molecular weight PAHs, and the r^2 must be greater than 0.99. The calibration curve must not be forced through the origin; and (iii) the number of calibration standards may be reduced from four to three based on the criteria in Sec. 11.4.1 of this procedure.

There must be an initial calibration of the GC/MS system as described in Sec. 11.3. In addition, the initial calibration curve should be verified immediately after performing the standard analyses using a second source standard (prepared using standards different from the calibration standards if available). It is assumed after this method is formally published that various standard vendors will offer other sources than the current single standard mix source option. Should an independent source or possibly another lot number from the same standard source used to prepare the calibration standards not be available during planned sample analyses, the initial calibration should be verified using the continuing calibration standard. The suggested acceptance limits for this initial calibration verification analysis are 70 - 130%. Alternative acceptance limits may be appropriate based on the desired project-specific data quality objectives. Quantitative sample analyses should not proceed for those analytes that fail the second source standard initial calibration verification. However, analyses may continue for those analytes that fail the criteria with an understanding these results could be used for screening purposes and would be considered estimated values.

The initial calibration must be re-established if the RSD(s) exceed the limit(s). However, it is not necessary to re-establish the initial calibration in response to a nonconforming RSD if the reported sample result(s) are less than the lower limits of quantitation and the signal to noise ratio and r^2 criteria are met.

9.8 Continuing Calibration Verification. The following acceptance criteria must be used for the daily duplicate calibration verifications: (1) The S/N ratio for the GC signals present in every SICP must be $\geq$ 10:1 for the labeled internal standards and the calibration compounds; (2) The percent differences for the measured area ratio/ng of all analytes must be within ±25% for high molecular weight PAHs and within ±20% for low molecular weight PAHs of the mean values established during the initial calibration.

The calibration verification standard should be prepared from the same stock standard source as is used for the initial calibration curve standards.

Should the acceptance criteria for the daily duplicate calibration verifications not be met, a new initial calibration curve must be established before sample extracts can be analyzed.

9.9 The signal to noise (S/N) ratio for the GC signals present in every SICP must be $\geq$ 3:1 for any target analyte in environmental samples and $\geq$ 10:1 for the labeled internal standards.

Should the acceptance criteria for any sample and/or labeled internal standard signal to noise ratio not be met, the sample should be reanalyzed unless obvious matrix interference is present.

10.0 CALIBRATION AND STANDARDIZATION

See Sec. 11.0 for calibration and standardization information.

11.0 PROCEDURE

11.1 To prepare the apparatus, set up the GC system using the following parameters.

11.1.1 GC Column Agilent HP-5MS column (0.25 µm film thickness, 0.25 mm ID) or equivalent.

11.1.2 Inlet liner 2-mm i.d. silanized glass.

11.1.3 GC Inlet 320 °C, splitless mode.

11.1.4 Oven program: Isothermal 5 minute hold at 40 °C. Ramp at 50 °C/minute to 110 °C, followed by a temperature ramp of 12 °C/minute to 320 °C. (Hold for 10 min.)

MS Quad Temperature: 150 °C, maximum 200 °C

MS Source Temperature: 230 °C, maximum 250 °C

11.2 SIM Group Set Up

11.2.1 Set up a SIM program with the necessary ions to acquire all the PAHs using the ion groups shown in Table 1 and set a 25 msec dwell time per ion.

11.2.2 Update the expected retention times in the method section of the quantitation software using the d-PAH internal standards of previous runs as relative retention time markers.

11.3 Establish Initial Calibration

The following initial calibration guidance is based on data generated during the method development process. The recommended calibration concentrations listed in Table 2 were based on the PAH distributions previously determined in 120 sediment pore water samples and take into account the water solubilities of each individual PAH compound. These are the recommended calibration standard concentrations for optimum method performance. However, other concentrations may be used provided acceptable method performance can be attained.

11.3.1 Prepare stock solutions of PAHs and internal standard stock solutions of d-PAHs at approximately the concentrations shown in Table 2. Stocks are prepared in DCM. Spiking solutions are prepared by dilution of intermediate stocks in acetone. For calibration solutions, spiking solutions are added to reagent water.

11.3.1.1 Prepare calibration standard spiking solutions. These are prepared by adding acetone to the stock to give the calibration solution concentrations (CS1-CS4), as described below.

11.3.1.1.1 For CS1, take 5 µL stock to 100 mL in acetone.

11.3.1.1.2 For CS2 take 50 µL to 100 mL in acetone.

11.3.1.1.3 For CS3, take 25 µL to 10 mL in acetone.

11.3.1.1.4 For CS4, take 100 µL to 10 mL in acetone.

11.3.1.2 Spike 4 µL of each calibration solution into 1.5 mL of reagent water to give a calibration series with the low calibration limits (LCLs) and upper calibration limits (UCLs) shown in Table 2. Spike 10 µL of internal standard spiking solution at the concentrations shown in Table 2 into each vial.

11.3.1.3 Extract and analyze the calibration series.

11.3.1.3.1 Extract and analyze two method blank solutions.

11.3.1.3.2 Extract and analyze the water calibration solutions, as described in Secs. 11.4 and 11.5. Begin with the CS1-spiked sample, followed by sequentially more concentrated calibration standards. Follow by two water blanks.

11.3.1.4 Calculate the performance parameters for the calibration.

11.3.1.4.1 Generate ion chromatograms for the masses listed in Table 1 that encompass the expected retention windows of the target analytes. Integrate the selected ion current profiles of the quantitation ions shown in the table.

11.3.1.4.2 Calculate the area ratio (analyte peak area divided by internal standard peak area) per unit mass of analyte, using the area of the appropriate internal standard listed in Table 3.

Quantitative calculations are based on a comparison of the area ratio per ng from the calibration and sample waters. The area ratio per ng is calculated for calibration runs by dividing the calibration peak area by the peak area of its most closely associated d-PAH internal standard (the deuterated parent PAH, in most cases), and dividing this result by the ng of the calibration PAH present in the vial (i.e., its mass in the vial, not its concentration). Calibration standards are given in Table 2.

(area ratio/ng) = [(peak area cal. std)/(peak area d-PAH)]/(mass of std in cal vial)

11.3.1.4.3 Calculate the mean area ratio/ng. The mean relative response factor for these duplicate daily calibration standards should agree with those from the 4-point (or 3-point) standard curve within 20% for the two- and three-ring PAHs, and within 25% for the four-ring PAHs. No sample data will be reported if these calibration criteria are not met. Calculate the mean area ratio/ng and the standard deviation of the relative response factors for each calibration standard solution using the following equations:

$$\overline{\text{area ratio/ng}} = \frac{1}{\text{n}} \sum_{i=1}^{n} (\text{area ratio/ng})_{\text{i}}$$

Where:

$(\text{area ratio/ng})_i$ = area ratio/ng calculated for calibration solution "i" using the equation in Sec. 11.3.1.4.2.

n = The number of calibration points in the curve.

11.3.1.4.4 Calculate the percent relative standard deviation.

$$\%\text{RSD} = \frac{\text{SD}}{\overline{\text{area ratio/ng}}} \times 100$$

Where:

$\overline{\text{area ratio/ng}}$ = Mean area ratio/ng calculated above.

SD = The sample standard deviation of the replicate area ratio/ng values used to calculate the mean area ratio/ng.

11.4 Criteria for acceptable initial calibration. Prior to analyzing any samples, the standard curves are prepared using the identical analysis procedures as used for sample waters. To be acceptable, the linearity of each PAH standard curve should be r^2 greater than 0.99, and the relative response factor per ng for each concentration should show a relative standard deviation of less than 25% for two- to three-ring PAHs, and less than 30% for four-ring PAHs. If an acceptable initial calibration is not achieved, identify the root cause, perform corrective action, and repeat the initial calibration. If the root cause can be traced to an abnormal disruption of an individual acquisition (e.g., injector malfunction) repeat the individual analysis and recalculate the percent relative standard deviation. If the calibration is acceptable, document the problem and proceed; otherwise repeat the initial calibration. Additionally, prior to sample analyses the initial calibration should be verified using a check standard mix that is prepared from an independent source as the calibration standards. Should an independent source or possibly another lot number from the same standard source used to prepare the calibration standards not be available during planned sample analyses, the initial calibration should be verified using the continuing calibration check standard.

11.4.1 Because of the large range of calibration concentrations required, the wide range of water solubilities of the individual PAHs, and the desire to require only one stock calibration solution, some PAHs may only have a three point linear calibration curve that meets the above criteria. This is most likely to occur for the higher molecular weight PAHs, because the dilution of lowest calibration standard is likely to be below the lower limit of quantitation required for the method, so it does not negatively impact the analysis. In such

cases, the lowest calibration standard is ignored. Less frequently, the highest concentrations of the lowest molecular weight PAHs may exceed the linear dynamic range of the GC/MS response. In such cases the laboratory should investigate lowering the MS multiplier voltage to autotune voltage or slightly below and rerun the calibration curve. If the highest calibration standard still exceeds the detector linearity, it is acceptable to reject the highest concentration for those specific PAHs, as long as a minimum of a three-point standard curve is generated for each PAH.

It is recommended that a 4- (or 3-) point initial calibration be established every two weeks, when continuing calibration criteria are not met, or when service is performed on the GC/MS instrument system.

11.4.2 The S/N for the GC signals present in every SICP must be $\geq$ 10:1 for the labeled internal standards and unlabeled calibration compounds.

11.5 Continuing calibration check is performed daily at the beginning of a 24-hour period. The injection of the first continuing calibration begins the 24-hour window, within which all pore water samples must be injected. Duplicate daily standards are analyzed.

11.5.1 To prepare the continuing calibration check solution, into 1.5 mL of reagent water, add 4 µL of the CS3 calibration check spiking solution and 10 µL of the d-PAH internal standards.

11.5.2 Analyze duplicate vials of the continuing calibration check standard solution. Use the same data acquisition parameters as those used during the initial calibration. Check for GC resolution and peak shape. If peak shape or retention times are unacceptable, perform column and injector maintenance. If this fails to correct the problem, the column must be replaced and the calibration repeated.

11.5.3 Criteria for Acceptable Daily Calibration Check. The criteria listed below for acceptable calibration must be met at the beginning of each 24-hour period that samples are analyzed. The mean relative response factor for the duplicate daily calibration standards should agree with those from the 4-point (or 3-point) standard curve within 20% for the two- and three-ring PAHs, and within 25% for the four-ring PAHs. No sample data will be reported if these calibration criteria are not met. If the continuing calibration check criteria are not met, identify the root cause, perform corrective action and repeat the continuing calibration. If the second consecutive continuing calibration check does not meet acceptance criteria, additional corrective action must be performed.

Additionally, after establishment of the 4-point calibration curve, the raw peak areas of each d-PAH for each subsequent daily calibration check, method blank, and sample analyses must be greater than or equal to 50% of the mean raw peak area for each d-PAH internal standard established for the 4-point calibration curve.

11.5.4 The S/N for the GC signals present in every SICP must be $\geq$ 10:1 for the labeled internal standards and unlabeled calibration compounds.

11.6 Method blanks are prepared and analyzed daily in duplicate following the continuing calibration and between analyses of replicate sets of the same pore water sample. See Sec. 11.6.2.2.

11.6.1 For each method blank, add 10 µL of the d-PAH internal standards solution into 1.5 mL of reagent water.

11.6.2 Two types of sources of background PAHs must be considered. For the higher molecular weight PAHs, typical GC/MS criteria for signal to noise are appropriate, since their lower limits of quantitation are normally controlled by GC/MS sensitivity. However, for lower molecular weight PAHs, atmospheric contaminants can cause significant background peaks.

11.6.2.1 Background PAHs from Ambient Air – Concentrations of each PAH in the water blanks should be calculated in the same manner as a sample. Should the blank prior to the subsequent water sample have any detectable background concentration greater than 1/3 of the example lower limits of quantitation given in Table 3, the analyses should not continue until the fiber is sufficiently cleaned as demonstrated by a clean reagent water blank.

11.6.2.2 Carryover from Highly Contaminated Samples – Carryover blanks are analyzed between each new pore water sample (not including replicates). Significant carryover can occur if the previous sample was highly contaminated. Should the blank prior to the subsequent water sample have any detectable background concentrations more than 1/3 of the example lower limits of quantitation given in Table 3, the analyses should not continue until the fiber is sufficiently cleaned as demonstrated by a clean reagent water blank. Alternatively, if the concentrations determined in the blanks are less than 20% of those found in the associated sample(s), the data may be accepted.

11.7 At the laboratory, store samples and extracts in the dark at 0 to 6 °C.

NOTE: Once the sample preparation process has begun, pore waters must be generated and flocculated as quickly as possible, but must be done within 28 days of sediment sample collection. Pore water, groundwater, and tap water samples must then be immediately spiked with 10 µL of d-PAH solution following flocculation.

Solid phase micro-extraction must be completed within 24 hours of flocculation for pore water, groundwater, and tap water samples.

11.8 Generation of pore water from sediment samples.

Stir the slurry and transfer approximately 40 mL (containing a solids and liquids in proportion to the slurry provided) to a clean 40 mL vial. Cap the vial with a PTFE-lined

cap. Place the vials in a centrifuge. Spin for 30 minutes at 1000 g. Using a new, graduated serological pipette, transfer 10 mL of the supernatant to a new 20 mL vial.

11.9 Flocculation of pore water samples.

11.9.1 Once the process has begun, immediately add the working alum solution (see Sec. 7.0) to each vial of water (and QC samples). The volume of the alum solution should be 1/40th of the sample volume. After the addition, swirl the vial for several rotations to incorporate the solution.

11.9.2 Add 3-5 drops of NaOH working solution (see Sec. 7.0) to each vial. Swirl to incorporate the NaOH.

11.9.3 Shake the vial for 15 seconds.

11.9.4 Centrifuge for 30 minutes at 1000 g.

11.9.5 Collect the supernatant into a clean 20 mL vial.

11.9.6 Repeat Secs. 11.10.1 through 11.10.5 once.

11.9.7 Immediately transfer 1.5 mL aliquots to new silanized autosampler vials and immediately add the internal standard solution as described below. Vials are weighed before and after adding the water sample to determine the exact sample water mass.

11.10 Extraction and analysis of flocculated pore water, groundwater, and tap water samples.

11.10.1 Split the prepared water samples into the required number of replicate samples, placing 1.5 mL aliquots of each into a new silanized glass autosampler vials. For QC samples, add 1.5 mL of reagent water.

NOTE: The SPME fiber should be cleaned at the beginning of each sampling set (and after highly contaminated samples) for one hour by placing in the cleaning chamber under helium flow at 320 °C. This can conveniently be performed while the pore waters are being prepared.

11.10.2 Immediately add 10 µL of the d-PAH solution to each sample and QC sample.

NOTE: All of the water preparation steps beginning with the centrifugation and ending with the addition of d-PAH internal standards should be conducted continuously and in the minimum amount of time possible.

11.10.3 Load the autosampler following the recommended analytical sequence in Table 4. Verify the sequence against documented sequence following the loading process.

11.11 The recommended analytical sequence described in Table 4 is based on a 24-hour “clock.”

11.11.1 Two continuing calibration check standards are analyzed (100 min.). The sequence begins with analysis of the first continuing calibration standard.

11.11.2 Analyze two method blanks (50 min. each).

11.11.3 Analyze pore water samples (in duplicate at a minimum) (50 min. each).

11.12 Generate ion chromatograms for the masses listed in Table 1 that encompass the expected retention windows of the target analytes. Integrate the selected ion current profiles of the quantitation ions shown in the table.

For a gas chromatographic peak to be identified as a target analyte, it must meet all of the following qualitative identification criteria for individual analytes.

11.12.1 The quantitation ion must be present, with a S/N of at least 3:1 for environmental samples.

11.12.2 The relative retention time (RRT) of the parent PAHs (and the 2- and 1- methylnaphthalene compounds) compared to the RRT for the labeled-standards must be within ± 3 seconds of the relative retention times obtained from the continuing calibration (or initial calibration if this applies).

11.13 Quantitation for Target Analytes

Results need to be reported in the units commensurate with their intended use and all dilutions need to be taken into account when computing final results.

Sample water concentrations are calculated by dividing the peak area of the sample peak by the peak area of its d-PAH internal standard, and then dividing the result by the calibration area ratio per ng, and dividing that result by the sample water weight.

$$\text{Concentration (ng/mL)} = \frac{(\text{area sample peak})/(\text{area d-PAH peak})}{(\text{area ratio per ng cal. std}) / (\text{sample weight})}$$

The mean calibration area ratio per ng values from the daily calibration check runs is used for sample concentration calculations (assuming QA/QC checks with the full calibration curve meet criteria).

NOTE: The two methylnaphthalene isomers are individual alkyl peaks and are treated as parent PAHs in the calculations.

11.13.1 If no peaks are present at a S/N $\geq$ 3 to 1 in the region of the ion chromatogram where the compounds of interest are expected to elute, report the result as “Not Detected” (i.e., ND) at the reporting limit.

11.13.2 Depending on project objectives, the results may be reported to lower limit of quantitation which is typically defined as 1/34 of a toxic unit. See Sec. 3.2 for additional information related to the lower limit of quantitation.

12.0 DATA ANALYSIS AND CALCULATIONS

See Secs. 11.12 and 11.13 for calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance goals for users of the methods. Instead, performance goals should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 Tables 5 through 12 present precision and bias data from three independent laboratories for spiked reagent water, groundwater, and two sediment pore water matrices. The PAH-impacted sediment samples were collected from an aluminum smelter site and a manufactured gas plant (MGP) site. These data are provided for guidance purposes only.

For the inter-laboratory validation study (ILV), the pore waters were spiked with 12 two- to four-ring PAHs at levels approximately 5 times and 50 times above the performance limit concentrations except for the five heavier PAHs, whose spike levels were based on the saturation solubility of the individual compounds. The highest molecular weight PAHs (i.e. benz(a)anthracene and chrysene), were spiked at five times the lower limit of quantitiation in the high level samples, instead of 50-fold, because of solubility limitations. In the low-level samples, benz(a)anthracene and chrysene were spiked at concentrations less than lower limit of quantitiation for these compounds. Therefore, based on the custodial laboratory recommendation, the low-level spike results for benz(a)anthracene and chrysene were not reported.

Seven replicate SPME analyses of the spiked sample pore waters were performed on each sample by each participating laboratory. Statistical outliers were determined and omitted using a one sided t-test at the 1% significance interval. All statistical outliers were traceable to one replicate analysis of the low-level groundwater sample from one participating laboratory. The high outlying results were caused by ambient (i.e., background) PAH contamination at the laboratory.

13.2.1 PAH concentration had no significant effect on the accuracy and reproducibility of the technique. The ILV recoveries for both the low and high-level spiked pore water samples (smelter and MGP sediments) ranged from 68 to 107 percent. The recoveries for both the low and high-level spiked aqueous samples (reagent water and groundwater) ranged from 81 to 108 percent.

13.2.2 It was assumed that the calculated standard deviation using the combined data from all three participating laboratories was equivalent to the

overall standard deviation (S_T). Replicate determinations of sample PAH concentrations typically had relative standard deviations (RSDs) less than 25 percent for the aqueous samples (reagent water and groundwater), with RSDs less than 33% for the impacted sediment samples (smelter and MGP sediments).

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in the laboratory operation. The USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

In keeping with USEPA goals this method uses a minimum of hazardous materials and results in only small amounts of hazardous waste.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington D.C. 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

15.1 The USEPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

15.2 Some of the reagents and solutions used in this method as well as the effluent from the chromatograph contain PAHs and should be handled and disposed of in an approved manner.

16.0 REFERENCES

1. American Society for Testing Methods (ASTM), E178-02, "Standard Practice for Dealing with Outlying Observations," Volume 14.02.

2. S. B. Hawthorne, C. B. Grabanski, D. J. Miller and J. P. Kreitinger, “Solid Phase Microextraction Measurement of Parent and Alkyl Polycyclic Aromatic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values,” *Environ. Sci. Technol*, 39, 2795-2803, 2005.

3. D. I. Thal, “Preparation of Flocculated Pore Waters and Analysis of Parent and Alkyl Polycyclic Aromatic Hydrocarbons by Isotope Dilution Solid Phase Microextraction/Gas Chomatography/Mass Spectrometry (SPME/GC/MS) (Hawthorne Method),” Standard Operating Procedure KNOX-ID-0019, ©Severn Trent Laboratories. 2006.

4. S. B. Hawthorne, C. B. Grabanski, and D. J. Miller, “Measured Partitioning Coefficients for Parent and Alkyl Polycyclic Aromatic Hydrocarbons in 114 Historically Contaminated Sediments: Part I, Koc Values,” *Environmental Toxicology and Chemistry,* 25, 2901-2911, 2006.

5. U.S. EPA (OSWER), “Development and Validation of SW-846 Methods, Phase 2: Formal Validation,” April, 1992. http://www.epa.gov/epaoswer/hazwaste/test/pdfs/methdev.pdf

6. U. S. Environmental Protection Agency, “Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: PAH Mixtures,” EPA/600/R-02/013, Office of Research and Development, Washington D.C., November 2003.

7. American Society for Testing Methods (ASTM), D7363-07, “Standard Test Method For Determination Of Parent Alkyl Polycyclic Aromatics in Sediment Pore Water Using Solid Phase Microextraction and Gas Chromatography/Mass Spectrometry in Selected Ion Monitoring Mode,” Volume 11.02.

8. D. Mackay and W.U. Shiu, Aqueous solubility of polynuclear aromatic-hydrocarbons, J. Chem. Eng. Data 22 (1977), pp. 399–402.

9. Syracuse Research Corporation, Environmental Database <http://www.syrres.com/esc/chemfate.htm>

17.0 TABLES AND FIGURES

The following pages contain the tables and figures referenced by this method.

TABLE 1

SIM TARGET IONS AND RETENTION TIME WINDOWS

Analyte	Carbon 12 (amu)	Hydrogen 1.007825 (amu)	Mass of Compound (amu)	SIM Ion Group	Target m/z	Retention Time[a] (min) Start	Stop
Naphthalene	10	8	128.063	1	128.1	7	17
1-Methylnaphthalene	11	10	142.078	1	142.1	7	17
2-Methylnaphthalene	11	10	142.078	1	142.1	7	17
Acenaphthylene	12	8	152.063	1	152.1	7	17
Acenaphthene	12	10	154.078	1	154.1	7	17
Fluorene	13	10	166.078	1	166.1	7	17
Anthracene	14	10	178.078	2	178.1	17	21
Phenanthrene	14	10	178.078	2	178.1	17	21
Fluoranthene	16	10	202.078	2,3	202.1	17	30
Pyrene	16	10	202.078	2,3	202.1	17	30
Benz(a)anthracene	18	12	228.094	3	228.1	21	30
Chrysene	18	12	228.094	3	228.1	21	30
d-PAH Internal Standards		Deuterium 2.014102 (amu)					
Naphthalene-d8	10	8	136.113	1	136.1	7	17
1-Methylnaphthalene-d10	11	10	152.141	1	152.1	7	17
Acenaphthene-d10	12	10	164.141	1	164.1	7	17
Fluorene-d10	13	10	176.141	1	176.1	7	17
Phenanthrene-d10	14	10	188.141	2	188.1	17	21
Fluoranthene-d10	16	10	212.141	2,3	212.1	17	30
Pyrene-d10	16	10	212.141	2,3	212.1	17	30
Chrysene-d12	18	12	240.169	3	240.2	21	30

[a]: Retention times must be verified by the user.

TABLE 2

INITIAL CALIBRATION STANDARD SERIES

Analyte	DCM Stock Conc. mg/mL	LCL CS1 ng/1.5 mL	CS2 ng/1.5 mL	CS3 ng/1.5 mL	UCL CS4 ng/1.5 mL
Naphthalene	42	8.3	83	415	1660
1-Methylnaphthalene	24	4. 8	48	239	956
2-Methylnaphthalene	20	4.1	41	204	817
Acenaphthylene	9.0	1.8	18	90	361
Acenaphthene	11	2.2	22	110	440
Fluorene	7.6	1.5	15	76	302
Anthracene	0.60	0.12	1.2	6.0	24
Phenanthrene	5.5	1.1	11	55	220
Fluoranthene	2.1	0.42	4.2	21	84
Pyrene	1.8	0.36	3.6	18	72
Benz(a)anthracene	0.08	0.02	0.16	0.80	3.2
Chrysene	0.03	0.006	0.06	0.30	1.2
Deuterated Analogs of Mix A Compounds	Stock Solution	CS1	CS2	CS3	CS4
Naphthalene-d8	5	50	50	50	50
1-Methylnaphthalene-d10	6	60	60	60	60
Acenaphthene-d10	1.2	12	12	12	12
Fluorene-d10	1.2	12	12	12	12
Phenanthrene-d10	0.96	9.6	9.6	9.6	9.6
Fluoranthene-d10	0.93	9.3	9.3	9.3	9.3
Pyrene-d10	0.84	8.4	8.4	8.4	8.4
Chrysene-d12	0.033	0.33	0.33	0.33	0.33

TABLE 3

EXAMPLE TOXIC UNIT FACTORS AND LOWER LIMITS OF QUANTITATION (LLOQ)

Analyte	Added d-PAH Internal Standard	d-PAH Internal Std. for Calculation	SPME-GC/MS RRF vs. Parent	Conc. For One Toxic Unit, C_{tu}, (ng/mL)	LLOQ (ng/mL)
Naphthalene	A	A	1.00	193	5.7
2-Methylnaphthalene		B	1.00	82	2.4
1-Methylnaphthalene	B	B	1.00	82	2.4
Acenaphthylene		C	1.00	307	9.0
Acenaphthene	C	C	1.00	56	1.6
Fluorene	D	D	1.00	39	1.2
Phenanthrene	E	E	1.00	19	0.56
Anthracene		E	1.00	21	0.61
Fluoranthene	F	F	1.00	7.1	0.21
Pyrene	G	G	1.00	10	0.30
Benz(a)anthracene		H	1.00	2.2	0.07
Chrysene	H	H	1.00	2.0	0.06

Data taken from Ref. 6.

TABLE 4

EXAMPLE OF A 24-HOUR ANALYTICAL SEQUENCE[a]

Run Type	Minutes	Cumulative Minutes to Start	Cumulative Minutes to End	Cumulative Hours to Start[a]	Cumulative Hours to End
Verification Std.	50	0	50	0.0	0.8
Verification Std.	50	50	100	0.8	1.7
Blank	50	100	150	1.7	2.5
Blank	50	150	200	2.5	3.3
Sample	50	200	250	3.3	4.2
Sample	50	250	300	4.2	5.0
Blank	50	300	350	5.0	5.8
Blank	50	350	400	5.8	6.7
Sample	50	400	450	6.7	7.5
Sample	50	450	500	7.5	8.3
Blank	50	500	550	8.3	9.2
Blank	50	550	600	9.2	10.0
Sample	50	600	650	10.0	10.8
Sample	50	650	700	10.8	11.7
Blank	50	700	750	11.7	12.5
Blank	50	750	800	12.5	13.3
Sample	50	800	850	13.3	14.2
Sample	50	850	900	14.2	15.0
Blank	50	900	950	15.0	15.8
Blank	50	950	1000	15.8	16.7
Sample	50	1000	1050	16.7	17.5
Sample	50	1050	1100	17.5	18.3
Blank	50	1100	1150	18.3	19.2

[a] The last pore water sample must be injected within 24 hours of the flocculation step. (i.e., The value for cumulative hours to start must be $\leq$ 24.)

TABLE 5

REAGENT WATER LOW-LEVEL PRECISION AND BIAS DATA

Analyte	True Value (ng/mL)	Number of Retained Values	Overall Mean Recovery (ng/mL)	Overall Mean Recovery (%)	Overall Standard Deviation (S_T)	Relative Standard Deviation (ng/mL)
Naphthalene	28	21	27	99	2.9	11
2-Methylnaphthalene	14	21	12	91	1.7	14
1-Methylnaphthalene	16	21	15	97	1.8	12
Acenaphthylene	6.0	21	5.5	92	0.74	13
Acenaphthene	7.3	21	6.9	94	0.77	11
Fluorene	5.0	21	4.8	96	0.49	10
Phenanthrene	3.7	21	3.5	96	0.34	10
Anthracene	0.40	21	0.32	81	0.10	30
Fluoranthene	1.4	21	1.4	97	0.18	13
Pyrene	1.2	21	1.2	97	0.14	12
Benz(a)anthracene	0.05	21	NR	NR	NR	NR
Chrysene	0.02	21	NR	NR	NR	NR

NR: Not reported.

TABLE 6

REAGENT WATER HIGH-LEVEL PRECISION AND BIAS DATA

Analyte	True Value (ng/mL)	Number of Retained Values	Overall Mean Recovery (ng/mL)	Overall Mean Recovery (%)	Overall Standard Deviation (S_T)	Relative Standard Deviation (ng/mL)
Naphthalene	277	21	263	95	30	11
2-Methylnaphthalene	136	21	118	87	22	19
1-Methylnaphthalene	159	21	150	94	20	13
Acenaphthylene	60	21	56	94	9.6	17
Acenaphthene	73	21	70	96	8.4	12
Fluorene	50	21	48	96	6.0	12
Phenanthrene	37	21	35	96	4.6	13
Anthracene	4.0	21	3.6	89	0.70	20
Fluoranthene	14	21	13	96	1.8	14
Pyrene	12	21	11	95	1.5	13
Benz(a)anthracene	0.53	21	0.48	91	0.04	7.4
Chrysene	0.20	21	0.20	99	0.02	7.6

TABLE 7

GROUNDWATER LOW-LEVEL PRECISION AND BIAS DATA

Analyte	True Value (ng/mL)	Number of Retained Values	Overall Mean Recovery (ng/mL)	Overall Mean Recovery (%)	Overall Standard Deviation (S_T)	Relative Standard Deviation (ng/mL)
Naphthalene	28	20	26	93	1.6	6.1
2-Methylnaphthalene	14	20	13	93	1.1	8.8
1-Methylnaphthalene	16	20	15	92	0.98	6.7
Acenaphthylene	6.0	21	5.4	90	0.51	9.4
Acenaphthene	7.3	20	6.6	91	0.40	6.1
Fluorene	5.0	20	4.6	91	0.26	5.7
Phenanthrene	3.7	21	3.6	99	0.36	10
Anthracene	0.40	21	0.42	105	0.03	7.9
Fluoranthene	1.4	20	1.3	93	0.07	5.1
Pyrene	1.2	21	1.1	92	0.07	6.4
Benz(a)anthracene	0.05	21	NR	NR	NR	NR
Chrysene	0.02	21	NR	NR	NR	NR

NR: Not reported.

TABLE 8

GROUNDWATER HIGH-LEVEL PRECISION AND BIAS DATA

Analyte	True Value (ng/mL)	Number of Retained Values	Overall Mean Recovery (ng/mL)	Overall Mean Recovery (%)	Overall Standard Deviation (S_T)	Relative Standard Deviation (ng/mL)
Naphthalene	277	21	254	92	23	9.0
2-Methylnaphthalene	136	21	118	86	16	13
1-Methylnaphthalene	159	21	145	91	15	11
Acenaphthylene	60	21	54	90	4.9	9.1
Acenaphthene	73	21	69	94	5.3	7.7
Fluorene	50	21	47	94	2.7	5.7
Phenanthrene	37	21	37	101	6.6	18
Anthracene	4.0	21	3.9	97	0.56	14
Fluoranthene	14	21	13	92	0.86	6.7
Pyrene	12	21	11	90	0.61	5.7
Benz(a)anthracene	0.53	21	0.48	91	0.05	11
Chrysene	0.20	21	0.18	88	0.02	8.7

TABLE 9

ALUMINUM SMELTER SEDIMENT LOW-LEVEL PRECISION AND BIAS DATA

Analyte	True Value (ng/mL)	Number of Retained Values	Overall Mean Recovery (ng/mL)	Overall Mean Recovery (%)	Overall Standard Deviation (S_T)	Relative Standard Deviation (ng/mL)
Naphthalene	28	21	27	99	2.9	11
2-Methylnaphthalene	14	21	13	96	1.3	9.9
1-Methylnaphthalene	16	21	15	97	1.7	11
Acenaphthylene	6.0	21	6.2	102	0.54	8.8
Acenaphthene	7.3	21	7.1	96	0.59	8.4
Fluorene	5.0	21	4.9	97	0.42	8.6
Phenanthrene	3.7	21	3.6	99	0.37	10
Anthracene	0.54	21	0.58	107	0.10	18
Fluoranthene	2.5	21	2.3	93	0.46	20
Pyrene	1.7	21	1.5	87	0.33	22
Benz(a)anthracene	0.15	21	NR	NR	NR	NR
Chrysene	0.20	21	NR	NR	NR	NR

NR: Not reported.

TABLE 10

ALUMINUM SMELTER SEDIMENT HIGH-LEVEL PRECISION AND BIAS DATA

Analyte	True Value (ng/mL)	Number of Retained Values	Overall Mean Recovery (ng/mL)	Overall Mean Recovery (%)	Overall Standard Deviation (S_T)	Relative Standard Deviation (ng/mL)
Naphthalene	277	21	260	94	42	16
2-Methylnaphthalene	136	21	111	81	29	26
1-Methylnaphthalene	159	21	147	92	26	18
Acenaphthylene	60	21	60	99	14	24
Acenaphthene	73	21	70	95	11	16
Fluorene	50	21	48	96	7.2	15
Phenanthrene	37	21	34	93	5.6	16
Anthracene	4.0	21	4.2	104	0.63	15
Fluoranthene	14	21	14	99	2.4	17
Pyrene	12	21	11	94	1.8	16
Benz(a)anthracene	0.63	21	0.49	78	0.04	8.5
Chrysene	0.37	21	0.30	81	0.05	18

TABLE 11

MGP SEDIMENT LOW-LEVEL PRECISION AND BIAS DATA

Analyte	True Value (ng/mL)	Number of Retained Values	Overall Mean Recovery (ng/mL)	Overall Mean Recovery (%)	Overall Standard Deviation (S_T)	Relative Standard Deviation (ng/mL)
Naphthalene	28	21	26	95	2.1	8.1
2-Methylnaphthalene	14	21	13	92	1.2	9.3
1-Methylnaphthalene	16	21	15	92	1.3	8.6
Acenaphthylene	6.0	21	5.6	92	0.48	8.6
Acenaphthene	7.3	21	6.7	91	0.51	7.6
Fluorene	5.0	21	4.6	91	0.34	7.5
Phenanthrene	3.7	21	3.3	91	0.21	6.4
Anthracene	0.40	21	0.34	85	0.08	24
Fluoranthene	1.4	21	1.3	92	0.11	8.4
Pyrene	1.2	21	1.1	92	0.09	7.8
Benz(a)anthracene	0.05	21	NR	NR	NR	NR
Chrysene	0.02	21	NR	NR	NR	NR

NR: Not reported.

TABLE 12

MGP SEDIMENT HIGH-LEVEL PRECISION AND BIAS DATA

Analyte	True Value (ng/mL)	Number of Retained Values	Overall Mean Recovery (ng/mL)	Overall Mean Recovery (%)	Overall Standard Deviation (S_T)	Relative Standard Deviation (ng/mL)
Naphthalene	277	21	257	93	36	14
2-Methylnaphthalene	136	21	118	87	24	21
1-Methylnaphthalene	159	21	148	93	22	15
Acenaphthylene	60	21	58	96	9.0	16
Acenaphthene	73	21	70	95	9.8	14
Fluorene	50	21	48	95	6.9	14
Phenanthrene	37	21	35	94	5.4	16
Anthracene	4.0	21	3.5	87	1.1	30
Fluoranthene	14	21	13	94	2.0	16
Pyrene	12	21	11	94	1.6	14
Benz(a)anthracene	0.53	21	0.43	81	0.05	12
Chrysene	0.20	21	0.19	93	0.02	8.8

Appendix C: ASTM Provisional Method D-7363-07

Designation: D 7363 – 07

Standard Test Method for Determination of Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water Using Solid-Phase Microextraction and Gas Chromatography/Mass Spectrometry in Selected Ion Monitoring Mode[1, 2]

This standard is issued under the fixed designation D 7363; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 The U.S. Environmental Protection Agency (USEPA) narcosis model for benthic organisms in sediments contaminated with polycyclic aromatic hydrocarbons (PAHs) is based on the concentrations of dissolved PAHs in the interstitial water or "pore water" in sediment. This test method covers the separation of pore water from PAH-impacted sediment samples, the removal of colloids, and the subsequent measurement of dissolved concentrations of the required 10 parent PAHs and 14 groups of alkylated daughter PAHs in the pore water samples. The "24 PAHs" are determined using solid-phase microextraction (SPME) followed by Gas Chromatography/Mass Spectrometry (GC/MS) analysis in selected ion monitoring (SIM) mode. Isotopically labeled analogs of the target compounds are introduced prior to the extraction, and are used as quantification references.

1.2 Lower molecular weight PAHs are more water soluble than higher molecular weight PAHs. Therefore, USEPA-regulated PAH concentrations in pore water samples vary widely due to differing saturation water solubilities that range from 0.2 µg/L for indeno[1,2,3-cd]pyrene to 31 000 µg/L for naphthalene. This method can accommodate the measurement of milligram per litre concentrations for low molecular weight PAHs and nanogram per litre concentrations for high molecular weight PAHs.

1.3 The USEPA narcosis model predicts toxicity to benthic organisms if the sum of the toxic units (ΣTU_c) calculated for all "34 PAHs" measured in a pore water sample is greater than or equal to 1. For this reason, the performance limit required for the individual PAH measurements were defined as the concentration of an individual PAH that would yield 1/34 of a toxic unit (TU). However, the focus of this method is the 10 parent PAHs and 14 groups of alkylated PAHs (Table 1) that contribute 95 % of the toxic units based on the analysis of 120 background and impacted sediment pore water samples.[3] The primary reasons for eliminating the rest of the 5-6 ring parent PAHs are: *(1)* these PAHs contribute insignificantly to the pore water TU, and *(2)* these PAHs exhibit extremely low saturation solubilities that will make the detection of these compounds difficult in pore water. This method can achieve the required detection limits, which range from approximately 0.01 µg/L, for high molecular weight PAHs, to approximately 3 µg/L for high molecular weight PAHs.

1.4 The test method may also be applied to the determination of additional PAH compounds (for example, 5- and 6-ring PAHs as described in Hawthorne et al).[4] However, it is the responsibility of the user of this standard to establish the validity of the test method for the determination of PAHs other than those referenced in 1.1 and Table 1.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, refer to Section 9.

2. Referenced Documents

2.1 *ASTM Standards:* [5]

D 1192 Guide for Equipment for Sampling Water and

[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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[2] Standard methods under the jurisdiction of ASTM Committee D19 may be published for a limited time preliminary to the completion of full collaborative study validation. Such standards are deemed to have met all other D19 qualifying requirements but have not completed the required validation studies to fully characterize the performance of the test method across multiple laboratories and matrices. Preliminary publication is done to make current technology accessible to users of Standards, and to solicit additional input from the user community.

[3] Hawthorne, S. B., Grabanski, C. B., and Miller, D. J., "Measured Partitioning Coefficients for Parent and Akyl Polycyclic Aromatic Hydrocarbons in 114 Historically Contaminated Sediments: Part I, Koc Values," *Environmental Toxicology and Chemistry*, 25, 2006, pp. 2901-2911.

[4] Hawthorne, S. B., Grabanski, C.B., Miller, D .J., and Kreitinger, J. P., "Solid Phase Microextraction Measurement of Parent and Alkyl Polycyclic Aromatic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values," *Environmental Science Technology*, 39, 2005, pp. 2795-2803.

[5] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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TABLE 1 Relative Response Factors[A]

Analyte	SPME-GC/MS RRF[B] versus Parent	Basis for Performance Limit[C]
Naphthalene	1.00	B
2-Methylnaphthalene[D]	1.00	B
1-Methylnaphthalene	1.00	B
C2-Naphthalenes	1.44	B
C3-Naphthalenes	0.88	B
C4-Naphthalenes	0.71	C
Acenaphthylene	1.00	B
Acenaphthene	1.00	B
Fluorene	1.00	B
C1-Fluorenes	0.73	B
C2-Fluorenes	0.59	B
C3-Fluorenes	0.35	S
Phenanthrene	1.00	B
Anthracene	1.00	B
C1-Phenanthrenes/Anthracenes	0.57	B
C2-Phenanthrenes/Anthracenes	0.32	B
C3-Phenanthrenes/Anthracenes	0.29	B
C4-Phenanthrenes/Anthracenes	0.12	S
Fluoranthene	1.00	B
Pyrene	1.00	B
C1-Fluoranthenes/Pyrenes	0.51	C
Benz[a]anthracene	1.00	B
Chrysene	1.00	B
C1-Chrysenes/Benz[a]anthracenes	0.62	C

[A] From Hawthorne, S. B., Grabanski, C.B., Miller, D .J., and Kreitinger, J. P., "Solid Phase Microextraction Measurement of Parent and Alkyl Polycyclic Aromatic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values," *Environmental Science Technology*, 39, 2005, pp. 2795-2803.

[B] All relative response factors are based on the SPME-GC/MS peak area per ng of the alkyl PAH in a water standard compared to that of its parent PAH as determined by SPME followed by GC/MS.When several isomers were available, (for example, C2-naphthalenes), the mean relative response factor is reported. The relative response factors of alkyl PAHs for which no standards were available were estimated based on the closest analogous alkyl PAH as described in reference 2.1.

[C] Performance limits were determined as 3 times the background concentrations from the SPME fiber based on the analysis of water blanks ("B"), the lowest calibration standard which consistently yielded a signal to noise ratio of at least 3:1 ("C"), or (for when no calibration standard was available) for the lowest concentrations consistently found in pore water samples with a signal to noise ratio of at least 3:1 ("S"). Detection limits for alkyl PAHs are based on a single isomer.

[D] Alkyl PAHs used to determine the SPME-GC/MS relative response factors including alkyl naphthalenes (1-methyl-, 2-methyl-, 1,2-dimethyl-, 1,3-dimethyl-1,8-dimethyl-, 2,7-dimethyl-, 1-ethyl, 2-ethyl, 1,4,5-trimethyl-, 2,3,5-trimethyl-, and 2-isopropyl-), 1-methylfluorene, 2-methyl- and 9-methylanthracene, 1-methyl-, 2-methyl-, and 3-methylphenanthrene, 9,10-dimethylanthracene, 2-ethylanthracene, 2-tertbutylanthracene, 1-methyl-7-isopropylphenanthrene, 1-methylpyrene, 7-methylbenz[a]anthracene, and 7,12-dimethylbenz[a]anthracene.

Steam in Closed Conduits[6]
D 1193 Specification for Reagent Water
D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D 3370 Practices for Sampling Water from Closed Conduits
D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
E 178 Practice for Dealing With Outlying Observations

3. Terminology

3.1 *Definitions:*

3.1.1 *calibration standard*—a solution prepared from a secondary standard, stock solution, or both, and used to calibrate the response of the instrument with respect to analyte concentration.

[6] Withdrawn.

3.1.2 *calibration verification standard (VER)*—the mid-point calibration standard (CS3) that is analyzed daily to verify the initial calibration.

3.1.3 *CS1, CS2, CS3, CS4*—shorthand notation for calibration standards.

3.1.4 *data acquisition parameters*—parameters affecting the scanning operation and conversion of the analytical signal to digitized data files. These include the configuration of the ADC circuitry, the ion dwell time, the MID cycle time, and acquisition modes set up for the method. Examples of acquisition modes for the HP5973 include SIM mode, and Low Mass Resolution Mode.

3.1.5 *performance limit*—performance limit for individual PAH is defined as the concentration of an individual PAH that would yield 1/34 of a toxic unit. For performance limit of individual PAH, refer to Table 2 (see 4.6).

3.1.6 *deuterated PAH (d-PAH)*—polycyclic aromatic hydrocarbons in which deuterium atoms are substituted for all hydrogens (that is, perdeuterated). In this method, d-PAHs are used as internal standards.

3.1.7 *GC*—gas chromatograph or gas chromatography.

3.1.8 *HRGC*—high resolution GC.

3.1.9 *LRMS*—low resolution MS.

3.1.10 *internal standards*—isotopically labeled analogs (d-PAHs) of the target analytes that are added to every sample, blank, quality control spike sample, and calibration solution. They are added to the water samples immediately after completing the flocculation step and transferring the water aliquot to the autosampler vial, and immediately after adding the calibration PAH solution to water calibration standards, but before SPME extraction. The internal standards are used to calculate the concentration of the target analytes or estimated detection limits.

3.1.11 *laboratory blank*—see *method blank*.

3.1.12 *method blank*—an aliquot of reagent water that is extracted and analyzed along with the samples to monitor for laboratory contamination. Blanks should consistently meet concentrations at or less than one-third of the performance limits for individual PAHs stated in Table 2. Alternatively, if the PAH concentrations calculated from the water blank immediately preceding the test samples are <20 % of the test sample concentrations, the blank is acceptable.

3.1.13 *low calibration level (LCL)*—the level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

3.1.14 *high or upper calibration level (UCL)*—the concentration or mass of analyte in the sample that corresponds to the highest calibration level in the initial calibration. It is equivalent to the concentration of the highest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

3.1.15 *MS*—mass spectrometer or mass spectrometry.

3.1.16 *PAH*—polycyclic aromatic hydrocarbon, or alternately, polynuclear aromatic hydrocarbon.

TABLE 2 Toxic Unit Factors and Performance Limits[A]

Analyte	Added d-PAH Internal Standard	d-PAH Internal Std. for Calculation	SPME-GC/MS RRF versus Parent	Conc. for One Toxic Unit, C_{tu}, (ng/mL)	Performance Limit (ng/mL)
Naphthalene	A	A	1.00	193.47	5.69
2-Methylnaphthalene		B	1.00	81.69	2.40
1-Methylnaphthalene	B	B	1.00	81.69	2.40
C2-Naphthalenes		A	1.44	30.24	0.89
C3-Naphthalenes		A	0.88	11.10	0.33
C4-Naphthalenes		A	0.71	4.05	0.12
Acenaphthylene		C	1.00	306.85	9.03
Acenaphthene	C	C	1.00	55.85	1.64
Fluorene	D	D	1.00	39.30	1.16
C1-Fluorenes		D	0.73	13.99	0.41
C2-Fluorenes		D	0.59	5.30	0.16
C3-Fluorenes		D	0.35	1.92	0.06
Phenanthrene	E	E	1.00	19.13	0.56
Anthracene		E	1.00	20.72	0.61
C1-Phenanthrenes/Anthracenes		E	0.57	7.44	0.22
C2-Phenanthrenes/Anthracenes		E	0.32	3.20	0.09
C3-Phenanthrenes/Anthracenes		E	0.29	1.26	0.04
C4-Phenanthrenes/Anthracenes		E	0.12	0.56	0.02
Fluoranthene	F	F	1.00	7.11	0.21
Pyrene	G	G	1.00	10.11	0.30
C1-Fluoranthenes/Pyrenes		G	0.51	4.89	0.14
Benz[a]anthracene		H	1.00	2.23	0.066
Chrysene	H	H	1.00	2.04	0.060
C1-Chrysenes/Benz[a]anthracenes		H	0.62	0.86	0.025

[A] From Hawthorne, S. B., Grabanski, C.B., Miller, D .J., and Kreitinger, J. P., "Solid Phase Microextraction Measurement of Parent and Alkyl Polycyclic Aromatic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values," *Environmental Science Technology*, 39, 2005, pp. 2795-2803.

3.1.17 *percent difference (%D)*—the difference between the analyzed concentration and expected concentration, expressed as a percentage of the expected concentration.

3.1.18 *relative response factor (RRF)*—the empirically determined ratio between the area ratio (analyte to internal standard) and the unit mass of analyte in the calibration standard (area ratio/ng) for available alkyl PAHs in a given homolog and their parent PAH.

3.1.19 *selected ion monitoring (SIM)*—a mode of operation for the mass spectrometer in which specific ions are monitored. This mode of operation differs from the full scan mode, in which the MS acquires all ions within a range. Because the spectrometer is monitoring fewer ions in the SIM mode, more acquisition (dwell) time is possible for each ion. This results in greater instrument sensitivity for the selected ions. Spectral scanning and library searching, used for tentatively identified compounds, are not supported in this mode.

3.1.20 *signal-to-noise ratio*—the ratio of the mass spectrometer response of a GC peak to the background noise signal.

4. Summary of Test Method

4.1 Either the use of an autosampler, or a manual approach can be used to perform the SPME extraction and the subsequent injection of collected analytes into the GC/MS. An autosampler (Leap Technologies Compi-Pal or equivalent) is much preferred over the manual method because: *(1)* the autosampler yields lower and more reproducible blanks, *(2)* the manual method requires the use of a stir bar that can cause sample cross-contamination, *(3)* the manual method is highly labor-intensive and requires multiple timed manipulations per analysis leading to operator fatigue and resultant errors, and *(4)* the autosampler reduces the technician time required to prepare samples for a 24-h run sequence to approximately 3 h, while the manual method requires 24-h operator attendance. Therefore, the method procedures are written assuming the use of an autosampler, with modifications to the autosampler procedures listed for the manual method.

AUTOSAMPLER METHOD

4.2 *Pore Water Separation and Preparation*—The pore water is separated from wet sediment samples by centrifugation and supernatant collection. Colloids are removed from the separated pore water samples by flocculation with aluminum potassium sulfate (alum) and sodium hydroxide as described in Hawthorne et al.[4] A second flocculation and centrifugation, followed by supernatant collection completes the colloid removal. The prepared pore water samples are then split into the required number of replicate aliquots (1.5 mL each) and placed into silanized glass autosampler vials. The 8 perdeuterated PAH internal standards (d-PAHs) are then added immediately. All of the water preparation steps beginning with the centrifugation and ending with the addition of d-PAH internal standards should be conducted continuously and in the minimum amount of time possible.

4.2.1 The SPME fiber should be cleaned at the beginning of each sampling set (and after very contaminated samples) for 1 h by placing in the cleaning chamber under helium flow at 320°C. This can conveniently be performed while the pore waters are being prepared.

4.3 *Solid-Phase Microextraction*—The SPME extraction of the pore water samples is performed using a commercially available (available from Sigma-Aldrich, formerly Supleco, or equivalent) 7 µm film thickness polydimethylsiloxane (PDMS)-coated fused silica fiber for 30 min while the water sample is mixed by the precession of the autosampler mixing chamber at a rate of 250 revolutions per minute. The target

PAHs and d-PAH internal standards adsorb to the nonpolar PDMS phase at equivalent rates. The use of the d-PAHs (that is, isotopic dilution) to quantitate the target PAHs compensates for variations in equilibrium partitioning and kinetics.

4.4 *GC/MS SIM Analysis*—Following the sorption period, the SPME fiber is immediately desorbed to a GC/MS injection port in the splitless mode at 320°C for 5 min. The GC/MS system specified uses a 60 m narrow-bore (250 µm ID) HP5-MS or equivalent capillary column to achieve high resolution for PAHs. Following the 5 min desorption period, the SPME fiber is inserted into the cleaning port and additionally cleaned for 15 min under helium flow at 320°C. At the end of the cleaning period, sorption of the next water sample is begun.

MANUAL METHOD

4.5 *Alternate Procedures for Manual Method*—Samples are prepared as for the autosampler method, except that a small Teflon-coated stir bar is placed in the silanized autosampler vial prior to adding the water and d-PAH internal standard solution. A new stir bar should be used for each sample, calibration standard, and blank to avoid cross-contamination caused by carryover on the stir bar. To perform the SPME step, the vial is set on a stir plate and the stirring rate adjusted so that no large vortex is formed. The SPME fiber should be inserted into the water so that the entire 1-cm active length is exposed to the water sample, but not so low that the fiber comes into contact with the stir bar or that the metal needle sheath contacts the water. All time sequences should be the same as specified for the autosampler method. A spare GC split/splitless injection port at 320°C and under helium flow can be used for the 15-min cleaning step between samples as well as for the initial 1-h cleaning step at the beginning of each experimental day.

4.6 The mass spectrometer is operated in the SIM mode for the molecular ions of the target PAHs and d-PAHs to achieve low limits of detection. Analyte concentrations are quantified by three methods:

4.6.1 PAHs for which an exact deuterated analog is included in the internal standard mix are quantified by isotope dilution.

4.6.2 Parent PAHs (that is, unsubstituted PAHs) for which an exact deuterated analog is not included in the internal standard mix are quantified by reference to a deuterated analog of a PAH with the same number of rings as the analyte.

4.6.3 Alkyl PAHs are quantified using the experimentally and empirically-determined relative response factors from Hawthorne et al.[4] and as shown in Table 1. The laboratory may use updated response factors, if additional alkyl PAH standards become commercially available. However, the laboratory must correct for purities of less than 98 %.

4.7 *Conversion of Quantified Concentration to Toxic Units*—The USEPA narcosis model predicts toxicity to benthic organisms if the sum of the toxic units calculated for all "34 PAHs" measured in a pore water sample is greater than or equal to 1. For this reason, the performance limits required for the individual PAH measurements were defined as the concentration of an individual PAH that would yield 1/34 of a toxic unit. See Table 2. This distribution reflects the relative concentrations of PAHs expected to be found in pore water because the lower molecular weight PAHs are more soluble and have lower organic carbon partition coefficients (Koc), and reflects the lower partitioning of lower molecular weight PAHs to the receptor organism since they have smaller octanol/water coefficients (Kow). The performance limits are essentially benchmarks to ensure that the adequate sensitivity is achieved to predict toxicity.

5. Significance and Use

5.1 This method directly determines the concentrations of dissolved PAH concentrations in environmental sediment pore water samples. The method is important from an environmental regulatory perspective because it can achieve the analytical sensitivities to meet the goals of the USEPA narcosis model for protecting benthic organisms in PAH contaminated sediments. Regulatory methods using solvent extraction have not achieved the wide calibration ranges from nanograms to milligrams per litre and the required levels of detection in the nanogram-per-litre range. In addition, conventional solvent extraction methods require large aliquot volumes (litre or larger), use of large volumes of organic solvents, and filtration to generate the pore water. This approach entails the storage and processing of large volumes of sediment samples and loss of low molecular weight PAHs in the filtration and solvent evaporation steps.

5.2 This method can be used to determine nanogram to milligram per litre PAH concentrations in pore water. Small volumes of pore water are required for SPME extraction, only 1.5 mL per determination and virtually no solvent extraction waste is generated.

6. Interferences

6.1 Non-target hydrocarbons can cause peaks on selected ion current profiles (SICPs) intended for other PAHs. Pattern recognition must be employed for identifying interfering peaks, and peak series that should not be considered for the homolog or target PAH under consideration. Analysts should be intimately familiar with both parent and alkyl PAH analyses in complex environmental samples. Representative samples having higher PAH concentrations should periodically be analyzed by full scan GC/MS so that pattern recognition of alkyl PAHs (and interfering species) can be verified by their full mass spectra. This procedure is particularly important for newer operators.

6.2 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferences under the conditions of analysis by performing laboratory method blanks. Analysts should avoid using PVC gloves, powdered gloves, or gloves with measurable levels of phthalates.

NOTE 1—The use of high purity reagents and solvents helps minimize interference problems.

7. Apparatus

7.1 *Centrifuge*, capable of sustaining 1000 g with cups for securing 40 mL and 20 mL vials.

7.2 *SPME Fiber Holder*, compatible with 7-µm SPME fiber and compatible with either the autosampler or the manual method.

TABLE 3 Primary Material Hazards

Material	Hazards	Exposure Limit[A]	Signs and Symptoms of Exposure
Alum (Aluminum Potassium Sulfate)	Irritant	2 mg/M^3 TWA	May cause skin irritation, especially under repeated or prolonged contact, or when moisture is present. May irritate or burn the eyes. Dust or mist inhalation at levels above the TLV may cause irritation to the respiratory tract. May irritate the gastrointestional tract.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Dichloromethane (DCM)	Carcinogen, Irritant	25 ppm-TWA, 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 mg/M^3 TWA	Causes skin irritation, chemical burns, permanent injury or scarring, and blindness. Vinegar is a mild acid that will neutralize lye if it were to make contact with the skin. Harmful if inhaled or ingested. Causes Sore throat, cough labored breathing, shortness of breath, and abdominal pain. Symptoms may be delayed.

[A] Exposure limit refers to the OSHA regulatory exposure limit.

7.3 *SPME Fibers*, 7-µm diameter, coated with polydimethylsiloxane (PDMS).

7.4 *PTFE Coated Stir Bars (Stir Fleas)*, of a size effective for stirring 1.5 mL water without vortexing (for manual method only).

7.5 *Magnetic Stir Plate (for manual method only).*

7.6 *SPME Holder Stand (for manual method only) or GC/MS Autosampler*, capable of SPME extraction and injection.

7.7 *Cleaning Port*, capable of purging SPME fibers in a helium-swept atmosphere at 320°C.

7.8 *GC/MS Analysis*:

7.8.1 *Gas Chromatograph* shall have split/splitless injection port for capillary column, temperature program with isothermal hold.

7.8.2 *GC Column*, 60 mm × 0.25 mm ID × 25 µm film thickness HP5-MS or equivalent.

7.8.3 *Inlet Liner*, 2 mm ID silanized glass.

7.8.4 *GC Inlet*, 320°C, splitless mode.

7.8.5 *Oven Program*—Isothermal 5 min hold at 40°C. Ramp at 50°C/min to 110°C, followed by a temperature ramp of 12°C/min to 320°C (hold for 10 min).

7.8.6 *Mass Spectrometer*—Electron impact ionization with the ionization energy optimized for best instrument sensitivity (typically 70 eV), stability and signal to noise ratio. Shall be capable of repetitively selectively monitoring at least 12 m/z during a period of approximately 1 s and shall meet all manufacturers' specifications.

7.8.7 *GC/MS Interface*—The mass spectrometer (MS) shall be interfaced to the GC such that the end of the capillary column terminates within 1 cm of the ion source but does not intercept the electron or ion beam.

7.8.8 *Data System*, capable of collecting, recording, and storing MS data.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[7]

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water that meets the purity specifications of Type I or Type II water, presented in Specification D 1193.

8.3 *40 mL Vials*, with Teflon-lined caps.

8.4 *20 mL Vials*, with Teflon-lined caps.

8.5 *Silanized 2.0 mL Autosampler Vials*.

8.6 *Internal Standard Stock Solution*—A dichloromethane solution of d-PAH internal standards used for preparing spiking solutions by dilution into acetone (see 12.2).

8.7 *Internal Standard Spiking Solution*—A dilution of the internal standard stock solution in acetone used to spike d-PAH internal standards into all sample, calibration, and blank water vials.

8.8 *Calibration Stock Solution*—A dichloromethane solution of PAHs used for preparing calibration standards (see 12.2).

8.9 *Calibration Spiking Solutions*—A series of solutions prepared by diluting the calibration stock solution with acetone (see 12.2).

8.10 *Calibration Standards*—Prepared by adding internal standard and calibration spiking solutions in reagent water (see 12.2).

8.11 *Acetone*.

8.12 *Dichloromethane* (DCM).

8.13 *Sodium Hydroxide* (NaOH).

8.14 *Aluminum Potassium Sulfate Dodecahydrate* ($AlK(SO_4)_2 \cdot 12H_2O$).

8.15 *Alum Solution*—Add 20 g ($AlK(SO_4)_2 \cdot 12H_2O$) to 80 mL reagent water.

9. Hazards

9.1 The effluents of sample splitters for the gas chromatograph and roughing pumps on the mass spectrometer must be

[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

vented to the laboratory hood exhaust system or must pass through an activated charcoal filter.

9.2 *Primary Materials Used*—The table contains a summary of the primary hazards listed in the MSDS. A complete list of materials used in the method can be found in the reagents and materials section. Practitioners must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

10. Sampling and Sample Preservation

10.1 Collect the sediment sample in accordance with Practices D 3370 and Specification D 1192, as applicable.

10.2 Prior to shipment, the samples should be mixed well. Sieve the slurry of sediment and site water through a 2-mm screen to remove debris. If the sieved slurry is to be stored or shipped before use, store in 250 mL to 1 L jars with PTFE-lined lids. Great care must be taken to clean the lid of the jar before capping with the lid to avoid leakage of the water during shipment.

10.3 Ship in an ice chest with adequate ice to maintain 0 to 6°C. Store at the laboratory in the dark at 0 to 6°C.

11. Preparation of Apparatus

11.1 Set up the GC system using the following parameters.

11.1.1 GC Column Agilent HP-5MS column (0.25 µm film thickness, 0.25 mm ID) or equivalent.

11.1.2 Inlet liner 2-mm ID silanized glass.

11.1.3 GC Inlet 320°C, splitless mode.

11.1.4 *Oven Program*—Isothermal 5 min hold at 40°C. Ramp at 50°C/min to 110°C, followed by a temperature ramp of 12°C/min to 320°C. (Hold for 10 min.)

MS Quad Temperature	150°C, maximum 200°C
MS Source Temperature	230°C, maximum 250°C

11.1.5 Set up SIM Groups to monitor the quantitation and internal standard ions shown in Table 4. Each ion dwell time should be set at 25 ms. Twelve ions are monitored in each group.

NOTE 2—Some ions (for example, m/z 184.1 for C4 naphthalenes) are included in two ion groups to ensure that the target peaks are adequately monitored. Table 4 should be used with the chromatograms in Appendix X1 to aid the analyst in setting proper retention time windows and recognition of target and contaminant peaks, especially for the alkyl clusters.

12. Calibration

12.1 Determine the absolute and relative retention times of the first and last characteristic peak in each homolog with the aid of the examples in Appendix X1.

12.1.1 Set up a SIM program with the necessary ions to acquire all the alkyl-PAH homologs using the ion groups shown in Table 4 and 25 ms dwell time per ion.

12.1.2 Update the expected retention times in the method section of the quantitation software using the d-PAH internal standards of previous runs as relative retention time markers and the representative chromatograms in Appendix X1. Assure that the SIM windows for the homologs are set to at least 8 s before the first, and 30 s after the last characteristic peaks to assure coverage of the elution range.

12.2 *Analyze Initial Calibration*:

TABLE 4 SIM Ion Groups and Retention Time Windows

NOTE—Retention times must be verified by the user.

Analyte	SIM Ion Group	Target m/z	Retention Time (min)	
			Start	Stop
Naphthalene	1	128.1	7	17
2-Methylnaphthalene	1	142.1	7	17
1-Methylnaphthalene	1	142.1	7	17
C2-Naphthalenes	1	156.1	7	17
C3-Naphthalenes	1	170.1	7	17
C4-Naphthalenes	1,2	184.1	7	21
Acenaphthylene	1	152.1	7	17
Acenaphthene	1	154.1	7	17
Fluorene	1	166.1	7	17
C1-Fluorenes	2	180.1	17	21
C2-Fluorenes	2	194.1	17	21
C3-Fluorenes	2,3	208.1	17	25
Phenanthrene	2	178.1	17	21
Anthracene	2	178.1	17	21
C1-Phenanthrenes/Anthracenes	2	192.1	17	21
C2-Phenanthrenes/Anthracenes	2,3	206.1	17	30
C3-Phenanthrenes/Anthracenes	2,3	220.1	17	30
C4-Phenanthrenes/Anthracenes	3	234.1	21	30
Fluoranthene	2,3	202.1	17	30
Pyrene	2,3	202.1	17	30
C1-Fluoranthenes/pyrenes	3	216.1	21	30
Benz[a]anthracene	3	228.1	21	30
Chrysene	3	228.1	21	30
C1-Chrysenes	3	242.1	21	30
d-PAH Internal Standards				
Naphthalene-d8	1	136.1	7	17
1-Methylnaphthalene-d10	1	152.1	7	17
Acenaphthene-d10	1	164.1	7	17
Fluorene-d10	1	176.1	7	17
Phenanthrene-d10	2	188.1	17	21
Fluoranthene-d10	2,3	212.1	17	30
Pyrene-d10	2,3	212.1	17	30
Chrysene-d12	3	240.2	21	30

12.2.1 Prepare stock solutions of PAHs and internal standard stock solutions of d-PAHs at approximately the concentrations shown in Table 5. These concentrations were based on the PAH distributions previously determined in 120 sediment pore water samples. Stocks are prepared in DCM. Spiking solutions are prepared by dilution of intermediate stocks in acetone. For calibration solutions, spiking solutions are added to reagent water.

12.2.1.1 Prepare calibration standard spiking solutions. These are prepared by adding acetone to the stock to give the calibration solution concentrations (CS1–CS4), as described below:

(1) For CS1, take 5 µL stock to 100 mL in acetone.

(2) For CS2 take 50 µL to 100 mL in acetone.

(3) For CS3, take 25 µL to 10 mL in acetone.

(4) For CS4, take 100 µL to 10 mL in acetone.

12.2.1.2 Spike 4 µL of each calibration solution into 1.5 mL of reagent water to give a calibration series with the low calibration limits (LCLs) and upper calibration limits (UCLs) shown in Table 5. Spike 10 µL of internal standard spiking solution at the concentrations shown in Table 5 into each vial.

12.2.1.3 Extract and analyze the calibration series.

(1) Extract and analyze two water blank solutions.

(2) Extract and analyze the water calibration solutions, as described in 13.4 and 13.5. Begin with the CS1-spiked sample,

TABLE 5 Initial Calibration Standard Series

Analyte	DCM Stock Conc. mg/mL	LCL			UCL
		CS1	CS2	CS3	CS4
		ng/1.5 mL	ng/1.5 mL	ng/1.5 mL	ng/1.5 mL
Naphthalene	41.5	8.3	83	415	1660
1-Methylnaphthalene	23.9	4.78	47.8	239	956
2-Methylnaphthalene	20.42	4.084	40.84	204.2	816.8
Acenaphthylene	9.02	1.804	18.04	90.2	360.8
Acenaphthene	11	2.2	22	110	440
Fluorene	7.55	1.51	15.1	75.5	302
Anthracene	0.6	0.12	1.2	6	24
Phenanthrene	5.5	1.1	11	55	220
Fluoranthene	2.11	0.422	4.22	21.1	84.4
Pyrene	1.8	0.36	3.6	18	72
Benz[a]anthracene	0.08	0.016	0.16	0.8	3.2
Chrysene	0.03	0.006	0.06	0.3	1.2
Deuterated Analogs of Mix A Compounds	Stock Solution	CS1	CS2	CS3	CS4
Naphthalene-d8	5	50.0	50.0	50.0	50.0
1-Methylnaphthalene-d10	6	60.0	60.0	60.0	60.0
Acenaphthene-d10	1.23	12.3	12.3	12.3	12.3
Fluorene-d10	1.2	12.0	12.0	12.0	12.0
Phenanthrene-d10	0.96	9.6	9.6	9.6	9.6
Fluoranthene-d10	0.93	9.3	9.3	9.3	9.3
Pyrene-d10	0.84	8.4	8.4	8.4	8.4
Chrysene-d12	0.033	0.33	0.33	0.33	0.33

followed by sequentially more concentrated calibration standards. Follow by two water blanks.

12.2.1.4 Calculate the performance parameters for the calibration.

(1) Generate ion chromatograms for the masses listed in Table 4 that encompass the expected retention windows of the target analytes. Integrate the selected ion current profiles of the quantitation ions shown in the table. Integration of alkyl clusters should be as the total area of the cluster integrated from the baseline before the first peak in the cluster to the baseline after the last peak in the cluster peaks. Cluster peaks should never be integrated using the valley-to-valley method. The peak areas of non-target peaks (see Appendix X1) must be removed from the alkyl cluster peak area before any calculation.

(2) Calculate the area ratio (analyte peak area divided by internal standard peak area) per unit mass of analyte, using the area of the appropriate internal standard listed in Table 1. Quantitative calculations are based on a comparison of the area ratio per ng from the calibration and sample waters. The area ratio per ng is calculated for calibration runs by dividing the calibration peak area by the peak area of its most closely associate d-PAH internal standard (the deuterated parent PAH, in most cases), and dividing this result by the ng of the calibration PAH present in the vial (that is, its mass in the vial, not its concentration). Calibration standards are given in Table 5.

$$area\ ratio\ per\ ng\ (ar\ rat/ng) = \frac{[(peak\ area\ cal.\ std)/(peak\ area\ d{-}PAH)]}{(mass\ of\ std\ in\ cal\ vial)} \quad (1)$$

(3) Calculate the mean ar rat/ng. The mean relative response factor for these duplicate daily calibration standards should agree with those from the 4-point (or 3-point) standard curve within 20 % for the two and three-ring PAHs, and within 25 % for the four-ring PAHs. No sample data will be reported if these calibration criteria are not met. Calculate the mean area ratio/ng and the standard deviation of the relative response factors for each calibration standard solution using the following equations:

$$\overline{ar\ rat/ng} = \frac{1}{n}\sum_{i=1}^{n}(ar\ rat/ng)_i \quad (2)$$

where:

$(ar\ rat/ng)_i$ = ar rat/ng calculated for calibration solution "i" using the equation in 12.2.1.4*(2)*, and

n = number of calibration points in the curve.

(4) Calculate the percent relative standard deviation:

$$\%RSD = \frac{SD}{\overline{ar\ rat/ng}} \times 100 \quad (3)$$

where:

$\overline{ar\ rat/ng}$ = mean ar rat/ng calculated above, and

SD = sample standard deviation of the replicate area rat/ng values used to calculate the mean ar rat/ng.

12.3 *Criteria for Acceptable Initial Calibration*—Prior to analyzing any samples, the standard curves are prepared using the identical analysis procedures as used for sample waters. To be acceptable, the linearity of each PAH standard curve should be $r^2 > 0.99$, and the relative response factor per ng for each concentration should show a relative standard deviation of <25 % for two- to three-ring PAHs, and <30 % for four-ring PAHs. See Section 16. If acceptable initial calibration is not achieved, identify the root cause, perform corrective action, and repeat the initial calibration. If the root cause can be traced to an abnormal disruption of an individual acquisition (for example, injector malfunction) repeat the individual analysis and recalculate the percent relative standard deviation. If the

calibration is acceptable, document the problem and proceed; otherwise repeat the initial calibration.

12.3.1 Because of the large range of calibration concentrations required, the wide range of water solubilities of the individual PAHs, and the desire to require only one stock calibration solution, some PAHs may only have a three point linear calibration curve that meets the above criteria. This is most likely to occur for the higher molecular weight PAHs, because the dilution of lowest calibration standard is likely to be below detection limits for many labs (and is also below the required detection limits needed for the method, so it does not negatively impact the analyses). In such cases, the lowest calibration standard is ignored, and the "J" level adjusted appropriately. Less frequently, the highest concentrations of the lowest molecular weight PAHs may exceed the linear dynamic range of the GC/MS response. In such cases the laboratory should investigate lowering the MS multiplier voltage to autotune voltage or slightly below and rerun the calibration curve. If the highest calibration standard still exceeds the detector linearity, it is acceptable to reject the highest concentration for those specific PAHs (and adjust the "E" value accordingly), as long as a minimum of a three-point standard curve is generated for each PAH.

12.3.1.1 It is recommended that a 4-point (or 3-point) initial calibration be established every two weeks, when continuing calibration criteria are not met, or when service is performed on the GC/MS instrument system.

12.3.2 The signal to noise ratio (S/N) for the GC signals present in every selected ion current profile (SICP) must be ≥10:1 for the labeled internal standards and unlabeled calibration compounds.

12.4 *Calibration Verification*—Continuing calibration is performed daily at the beginning of a 24-h period. The injection of the first continuing calibration begins the 24-h window, within which all pore water samples must be injected. Duplicate daily standards are analyzed.

12.4.1 Into 1.5 mL of reagent water, add 4 µL of the CS3 spiking solution and 10 µL of the d-PAH internal standards.

12.4.2 Analyze duplicate vials of the Calibration Standard Solution CS3. Use the same data acquisition parameters as those used during the initial calibration. Check for GC resolution and peak shape. If peak shape or retention times are unacceptable, perform column and injector maintenance. If this fails to correct the problem, the column must be replaced and the calibration repeated.

12.4.3 *Criteria for Acceptable Daily Calibration Check*—The criteria listed below for acceptable calibration must be met at the beginning of each 24-h period that samples are analyzed. The mean relative response factor for these duplicate daily calibration standards should agree with those from the 4-point (or 3-point) standard curve within 20 % for the two- and three-ring PAHs, and within 25 % for the four-ring PAHs. No sample data will be reported if these calibration criteria are not met. If the continuing calibration criteria are not met, identify the root cause, perform corrective action and repeat the continuing calibration. If the second consecutive continuing calibration does not meet acceptance criteria, additional corrective action must be performed.

12.4.4 The signal to noise ratio (S/N) for the GC signals present in every selected ion current profile (SICP) must be ≥10:1 for the labeled internal standards and unlabeled calibration compounds.

12.5 *Method Blanks*—Method blanks are prepared and analyzed daily in duplicate following the continuing calibration and between analysis of replicate sets of the same pore water sample. See 12.5.2.2.

12.5.1 For each method blank, add 10 µL of the d-PAH internal standards solution into 1.5 mL of reagent water.

12.5.2 Two types of sources of background PAHs must be considered. For the higher molecular weight PAHs, typical GC/MS criteria for signal to noise are appropriate, since their detection limits are normally controlled by GC/MS sensitivity. However, for lower molecular weight PAHs, atmospheric contaminants can cause significant background peaks, especially for low MW alkyl PAHs. This problem is most likely to be significant in urban areas impacted by atmospheric PAHs (for example, from diesel exhaust), and with laboratories using manual techniques, rather than the SPME autosampler.

12.5.2.1 *Background PAHs from Ambient Air*—Concentrations of each PAH in the water blanks should be calculated in the same manner as a sample. Should the blank prior to the subsequent pore water sample have detectable background concentrations more than ⅓ of the target detection limit given in Table 3, the analyses should not continue until the fiber is sufficiently cleaned as demonstrated by a clean water blank. The mean of the calculated concentrations of the PAHs in the blanks analyzed immediately before and immediately after sample pore waters should be subtracted from the sample pore water concentrations.

12.5.2.2 *Carryover from Highly Contaminated Samples*—Carryover blanks are analyzed between each new pore water sample (not including replicates). Significant carryover can occur if the previous sample was highly contaminated. Should the blank prior to the subsequent pore water sample have detectable background concentrations more than ⅓ of the target detection limit, the analyses should not continue until the fiber is sufficiently cleaned as demonstrated by a clean water blank. Alternatively, if the concentrations determined in the blanks are less than 20 % of those found in the related sample, the data can be accepted.

13. Procedure

13.1 At the laboratory, store samples and extracts in the dark at 0 to 6°C.

13.2 *Holding Times*:

13.2.1 Pore waters must be generated within 28 days of sediment sample collection.

13.2.2 Pore waters must be generated and flocculated as quickly as possible, and then immediately spiked with 10 µL of d-PAH solution.

13.2.3 Solid phase micro-extraction must be completed within 24 h of flocculation.

13.3 *Generation of Pore Water*:

13.3.1 Stir the slurry and transfer approximately 40 mL (containing a solids and liquids in proportion to the slurry provided) to a clean 40 mL vial. Cap the vial with a PTFE-lined cap. Place the vials in a centrifuge. Spin for 30 min at 1000 g.

TABLE 6 Example of a 24-h Analytical Sequence[A]

Example Analytical Sequence					
Run Type	Minutes	Cumulative Minutes to Start	Cumulative Minutes to End	Cumulative Hours to Start[A]	Cumulative Hours to End
Standard	50	0	50	0.0	0.8
Standard	50	50	100	0.8	1.7
Blank	50	100	150	1.7	2.5
Blank	50	150	200	2.5	3.3
Sample	50	200	250	3.3	4.2
Sample	50	250	300	4.2	5.0
Blank	50	300	350	5.0	5.8
Blank	50	350	400	5.8	6.7
Sample	50	400	450	6.7	7.5
Sample	50	450	500	7.5	8.3
Blank	50	500	550	8.3	9.2
Blank	50	550	600	9.2	10.0
Sample	50	600	650	10.0	10.8
Sample	50	650	700	10.8	11.7
Blank	50	700	750	11.7	12.5
Blank	50	750	800	12.5	13.3
Sample	50	800	850	13.3	14.2
Sample	50	850	900	14.2	15.0
Blank	50	900	950	15.0	15.8
Blank	50	950	1000	15.8	16.7
Sample	50	1000	1050	16.7	17.5
Sample	50	1050	1100	17.5	18.3
Blank	50	1100	1150	18.3	19.2

[A] The last pore water sample must be injected within 24 h of the flocculation step (that is, the value for cumulative hours to start must be ≤24).

Using a new, graduated serological pipette, transfer 10 mL of the supernatant to a new 20 mL vial.

13.3.2 *Flocculation of Pore Water*—Flocculation must be performed no more than 24 h prior to extraction.

13.3.2.1 If a flocculation blank is to be analyzed, create the blank by placing 10 mL of reagent water in clean a 40 mL vial. Process this blank along with pore water samples.

13.3.2.2 Add the working alum solution (see Section 9) to each vial of pore water (and QC samples). The volume of the alum solution should be 1/40th of the sample volume. After the addition, swirl the vial for several rotations to incorporate the solution.

13.3.2.3 Add 3 to 5 drops of NaOH working solution (see Section 9) to each vial. Swirl to incorporate the NaOH.

13.3.2.4 Shake the vial for 15 s.

13.3.2.5 Centrifuge for 30 min at 1000 g.

13.3.2.6 Collect the supernatant into a clean 20 mL vial.

13.3.2.7 Repeat 13.3.2.2 through 13.3.2.6 once.

13.3.2.8 Immediately transfer 1.5 mL aliquots to new silanized autosampler vials and immediately add the internal standard solution as described below. Vials are weighed before and after adding the water sample to determine the exact sample water mass.

13.4 *Extraction and Analysis of Flocculated Pore Water*:

13.4.1 Split the prepared pore water samples into the required number of replicate samples, placing 1.5 mL aliquots of each into a new silanized glass autosampler vial. For QC samples, add 1.5 mL of reagent water.

NOTE 3—The SPME fiber should be cleaned at the beginning of each sampling set (and after very contaminated samples) for 1 h by placing in the cleaning chamber under helium flow at 320°C. This can conveniently be performed while the pore waters are being prepared.

13.4.2 Immediately add 10 µL of the d-PAH solution to each sample and QC sample.

NOTE 4—All of the water preparation steps beginning with the centrifugation and ending with the addition of d-PAH internal standards should be conducted continuously and in the minimum amount of time possible.

13.4.3 Load the autosampler following the recommended analytical sequence in Table 6. Verify the sequence against documented sequence following the loading process.

13.5 The recommended analytical sequence described in Table 6 is based on a 24-h "clock."

13.5.1 Two calibration verification standards are analyzed (122 min). The sequence begins with analysis of the first continuing calibration standard.

13.5.2 Analyze two method blanks (61 min each).

13.5.3 Analyze pore water samples (in duplicate at a minimum) (61 min each).

14. Data Analysis and Calculations

14.1 Generate ion chromatograms for the masses listed in Table 4 that encompass the expected retention windows of the target analytes (see Appendix X1). Integrate the selected ion current profiles of the quantitation ions shown in the table.

14.1.1 *Qualitative Identification Criteria for Individual Analytes*—For a gas chromatographic peak to be identified as a target analyte, it must meet all of the following criteria:

14.1.1.1 The quantitation ion must be present, with a signal-to-noise ratio of at least 3:1 for environmental samples.

14.1.1.2 The relative retention time (RRT) of the parent PAHs (and the 2 and 1-methylnaphthalene compounds) compared to the RRT for the labeled-standards must be within ±3 s of the relative retention times obtained from the continuing calibration (or initial calibration if this applies). Alkyl clusters must be identified based on their relative retention times to the parent PAHs and related d-PAHs, and also by observation of their characteristic fingerprints by an experienced analyst.

14.1.2 *Qualitative Identification Criteria for Total Homolog Groups* (for example, total C2 or C3 alkylnaphthalenes)—Integration of the alkyl PAHs requires hands-on labor from a highly experienced analyst. Retention time windows, like those used for the parent PAHs are inadequate for identifying alkyl clusters (that can be minutes wide). Proper identification of alkyl clusters is critical, as is the proper identification of non-target species that occur at the same nominal mass. Mental pattern recognition must be used to avoid including non-target species that may occur at the same mass and retention time window as the target alkyl PAHs. All alkyl clusters should be integrated baseline to baseline to sum the total area of the cluster (adjusting the baseline for detector drift), but not valley to valley. Manual control of the integration is required for alkyl clusters.

14.1.2.1 Representative selected ion chromatograms from coal tar contaminated sediment pore water for all target species are shown in Appendix X1. The top chromatogram on each page is the d-PAH internal standard used for the parent and alkyl PAHs associated with that parent. For example, the first page shows d8-naphthalene (m/z 136) followed by naphthalene (m/z 128), the two methylnaphthalene isomers (m/z 142), the C2 naphthalene cluster (m/z 156), the C3-naphthalene cluster (m/z 170), and the C4 naphthalene cluster (m/z 184). The chromatogram also shows a typical interference that occurs in sediments for the C4-naphthalene cluster, that is, the dibenzothiophene isomers that occur in the same selected ion chromatogram as the C4-naphthalene cluster. These interfering dibenzothiopenes are crossed out, and the correct cluster for integration (based on full scan analyses of several different contaminated sediment pore waters) are indicated by brackets. Similar designations are used to indicate common interfering peaks and the correct target species in the subsequent chromatograms.

14.1.3 The retention time (RT) of the analyte must be no more than 5 s before the expected RT of the first isomer in the homolog, based on the continuing windowing solution analysis.

14.1.4 The retention time (RT) of the analyte must be no more than 5 s after the expected RT of the last isomer in the homolog, based on the continuing windowing solution analysis.

14.2 *Quantitation for Target Analytes*:

14.2.1 Sample water concentrations are calculated by dividing the peak area of the sample peak by the peak area of its d-PAH internal standard, and then dividing the result by the calibration area ratio per ng, and dividing that result by the sample water weight.

$$Concentration\ (ng/mL) = \frac{(area\ sample\ peak)/(area\ d-PAH\ peak)}{(area\ ratio\ per\ ng\ cal.\ std) \times (sample\ weight)} \quad (4)$$

14.2.2 The mean calibration area ratio per ng values from the daily calibration runs is used for sample concentration calculations (assuming QA/QC checks with the full calibration curve meet criteria).

14.2.3 The concentrations of alkyl PAH clusters are based on the calibration response of their parent PAH as adjusted for the relative response factor (rrf) for that cluster of species (including SPME and GC/MS responses) taken from Table 1. Thus, the concentrations of alkyl clusters are calculated by:

$$Concentration\ (ng/mL) = \frac{(area\ sample\ cluster)/(area\ d-PAH\ peak)}{(area\ ratio\ per\ ng\ parent\ cal\ std) \times (sample\ weight)} \quad (5)$$

NOTE 5—The two methylnaphthalene isomers are individual alkyl peaks (not clusters as in all other alkyl cases) and are treated as parent PAHs in the calculations.

14.2.4 If no peaks are present at a signal to noise value ≥3 to 1 in the region of the ion chromatogram where the compounds of interest are expected to elute, report the result as "Not Detected" (that is, ND) at the reporting limit.

14.2.5 Depending on project objectives, the results may be reported to TDLs or estimated detection limits (EDLs).

14.2.5.1 If project-specific guidance requires analysis-specific EDLs, calculate the detection limit for that compound according to the following equation:

$$Estimated\ Detection\ Limit = \frac{N \times 2.5}{His \times (ar\ rat/ng)} \quad (6)$$

where:

N = height of peak to peak noise of quantitation ion signal in the region of the ion chromatogram where the compound of interest is expected to elute,

His = peak height of quantitation ion for appropriate internal standard, and

$ar\ rat/ng$ = mean ar rat/ng of compound obtained during daily calibration.

14.2.5.2 If project-specific guidance requires total toxic units (TTU) to be reported, calculate the detection limit for that compound according to the following equations:

$$TU_c = Ctu \times result(ng/mL)^{-1} \quad (7)$$

$$Total\ Toxic\ Units\ (TTU) = \sum_{1}^{34} TU_c \quad (8)$$

where:

TU_c = toxic unit concentration for each individual compound or homolog (ng/mL),

Ctu = concentration for one toxic unit (ng/mL), see Table 2,

$result$ = individual pore water result for a compound or homolog (ng/mL), and

TTU = total toxic units for all 34 compounds and homologs.

14.2.6 Flag all compound results in the sample which were estimated below the lowest calibration level with a "J" qualifier.

14.2.7 Flag all compound results in the sample which were estimated above the upper calibration level with an "E" qualifier.

15. Precision and Bias

15.1 *Single Analyst Precision Statement*:

15.1.1 The recommendations of the ASTM task group members were followed in performing the single-laboratory study. Three environmental sediment samples were selected from archived sediments to represent low, medium, and high

TABLE 7 Precision Statement for SPME Pore Water PAHs

Target Analyte	Statistic/Parameter	Study Pore Water Samples		
		HP-24	HP-3	HP-4
		Low	Medium	High
Naphthalene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	ND[A]	130.9	975.3
	Single Operator Std. Deviation (So)		4.2	42.6
	Relative Standard Deviation (%)		3.2	4.4
2-Methylnaphthalene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	ND	20.2	245.4
	Single Operator Std. Deviation (So)		0.64	9.89
	Relative Standard Deviation (%)		3.2	4.0
1-Methylnaphthalene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	ND	81.7	209.6
	Single Operator Std. Deviation (So)		2.4	7.1
	Relative Standard Deviation (%)		3.0	3.4
C2-Naphthalenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.33	125.4	324.2
	Single Operator Std. Deviation (So)	0.0259	8.61	23.7
	Relative Standard Deviation (%)	7.8	6.9	7.3
C3-Naphthalenes	Number of Retained Values	7	7	6
	Mean Recovery (ng/mL)	0.41	124.9	212.5
	Single Operator Std. Deviation (So)	0.029	12.7	5.99
	Relative Standard Deviation (%)	7.1	10.2	2.8
C4-Naphthalenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.14	44.6	53.0
	Single Operator Std. Deviation (So)	0.025	6.05	5.3
	Relative Standard Deviation (%)	17.7	13.6	10.0
Acenaphthylene	Number of Retained Values	7	7	6
	Mean Recovery (ng/mL)	ND	0.16	7.52
	Single Operator Std. Deviation (So)		0.020	0.09
	Relative Standard Deviation (%)		12.5	1.3
Acenaphthene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.21	44.1	84.8
	Single Operator Std. Deviation (So)	0.0125	1.28	2.79
	Relative Standard Deviation (%)	6.1	2.9	3.3
Fluorene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.11	23.2	31.6
	Single Operator Std. Deviation (So)	0.0071	0.75	1.48
	Relative Standard Deviation (%)	6.7	3.2	4.7
C1-Fluorenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.11	22.4	25.8
	Single Operator Std. Deviation (So)	0.011	0.86	1.50
	Relative Standard Deviation (%)	10	3.8	5.8
C2-Fluorenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	ND	12.7	16.1
	Single Operator Std. Deviation (So)		0.88	1.85
	Relative Standard Deviation (%)		6.9	11.5
C3-Fluorenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	ND	ND	ND
	Single Operator Std. Deviation (So)			
	Relative Standard Deviation (%)			
Phenanthrene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.1	31.3	39.2
	Single Operator Std. Deviation (So)	0.0069	1.84	3.16
	Relative Standard Deviation (%)	6.8	5.9	8.1
Anthracene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.03	6.2	8.2
	Single Operator Std. Deviation (So)	0.0007	0.37	0.72
	Relative Standard Deviation (%)	2.6	5.9	8.9
C1-phenanthrenes/anthracenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.13	31.9	45.2
	Single Operator Std. Deviation (So)	0.0088	1.97	5.76
	Relative Standard Deviation (%)	6.9	6.2	12.7
C2-Phenanthrenes/Anthracenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.01	10.3	16.1
	Single Operator Std. Deviation (So)	0.0014	0.98	3.43
	Relative Standard Deviation (%)	11	9.5	21.3
C3-Phenanthrenes/Anthracenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	ND	4.4	4.4
	Single Operator Std. Deviation (So)		0.62	1.55
	Relative Standard Deviation (%)		14.1	35.5
C4-Phenanthrenes/Anthracenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	ND	1.2	ND
	Single Operator Std. Deviation (So)		0.24	

TABLE 7 *Continued*

Target Analyte	Statistic/Parameter	Study Pore Water Samples		
		HP-24	HP-3	HP-4
		Low	Medium	High
	Relative Standard Deviation (%)		20.6	
Fluoranthene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.04	5.6	5.8
	Single Operator Std. Deviation (So)	0.0028	0.61	0.87
	Relative Standard Deviation (%)	6.7	10.9	15.1
Pyrene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.06	6.2	7.7
	Single Operator Std. Deviation (So)	0.0038	0.75	1.28
	Relative Standard Deviation (%)	6.2	12.1	16.8
C1-Fluoranthenes/Pyrenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.04	5.0	6.1
	Single Operator Std. Deviation (So)	0.0033	0.78	1.79
	Relative Standard Deviation (%)	7.3	15.8	29.2
Benz[a]anthracene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	ND	0.76	0.75
	Single Operator Std. Deviation (So)		0.16	0.33
	Relative Standard Deviation (%)		20.8	44.5
Chrysene	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	0.01	0.77	0.79
	Single Operator Std. Deviation (So)	0.0009	0.16	0.35
	Relative Standard Deviation (%)	10.7	20.5	44.7
C1-Chrysenes	Number of Retained Values	7	7	7
	Mean Recovery (ng/mL)	ND	0.54	0.50
	Single Operator Std. Deviation (So)		0.11	0.33
	Relative Standard Deviation (%)		21.2	64.9
Total Toxic Units	Number of Retained Values	7	7	7
	Mean Recovery (units)	0.15	50.4	81.4
	Single Operator Std. Deviation (So)	0.01	3.52	5.23
	Relative Standard Deviation (%)	4.8	7.0	6.4

[A] ND: Analyte not detected in the associated sample.

concentrations of pore water PAHs. Efforts were made to ensure that sediments were chosen that had a full distribution of target PAH ring sizes, a range of PAH concentrations found in environmental sediment samples, and a representative range in total organic carbon concentration and texture.

15.1.2 The quantitations were based on three- or four-point calibration curves as verified by daily analysis of duplicate calibration verification standards at the medium-high concentration level. Prior to sample analysis, the initial calibration curves must have a coefficient of determination greater than 0.990, and the relative response factors must have a relative standard deviation of less than 25 % for two to three-ring PAHs, and less than 30 % for four-ring PAHs. The calibration verification mean relative response factor must agree with those of the initial calibration curve within 20 % for two to three-ring PAHs, and less than 25 % for four-ring PAHs. No sample data were reported if these criteria were not met. All method blanks met the requirement that the concentrations be at or less than 20 % of the Performance Limits for individual PAHs.

15.1.3 As directed in section 10.3 of Practice D 2777, the data were evaluated for outliers. The data were evaluated using the one-sided t-test at the upper 5 % significance level as described in Practice E 178, Section 6. Two outlying observations were found for high-level sample HP-4. One C3-naphthalenes result and one acenaphthylene result for sample HP-4 were outliers. The mean and single operator standard deviation were recalculated for sample HP-4 C3-naphthalenes and acenaphthylene without the outlying observations (that is, n = 6).

15.1.4 The precision statements for each analyte are shown on Table 7. For this single-laboratory study, it was assumed that the calculated standard deviation is equivalent to the single operator standard deviation (S_o). Replicate determinations of sample PAH concentrations typically had relative standard deviations (RSDs) less than 10 %, with somewhat higher RSDs for higher molecular weight compounds. The only unusually high RSDs occurred for the highest molecular weight PAHs from high-level sample HP 4. The reason for this is that the saturation limits may have been reached for the high molecular weight PAHs (that is, C1-phenanthrenes/anthracenes through C1-chrysenes).

15.1.5 Finally, the variation of individual PAH determinations had no significant effect on the repeatability of the total toxic unit determinations. See Table 7. This was demonstrated even though the statistical outliers found in sample HP-4 were not omitted in the calculation of total toxic units. The RSDs for the total toxic unit results ranged from 5 to 7 %.

15.2 *Single Analyst Bias Statement*:

15.2.1 A single laboratory study was performed using the perdeuterated PAHs d12-benz(a)anthracene and d10-2-methylnaphthalene spiked at low, medium, and high levels into environmental sediment samples. The quality control statements for each analyte level sample, obtained from the perdeuterated spike study, are shown in Tables 8-10. The quality control statements can also be considered precision and bias statements because the true spiking levels of the perdeuterated PAHs were known. The graphs and regression equations show the relationship between single-operator standard deviation and concentration, and mean measured value and

TABLE 8 HP-24 Low Concentration Quality Control

Analyte	True Spiked Value (ng/mL)	Number of Retained Values	Mean Recovery (ng/mL)	Mean Recovery (%)	Single Standard Deviation (So)	Relative Standard Deviation (%)
2-Methylnaphthalene-d10	4.68	7	4.33	92.6	0.3161	7.3
Benz[a]anthracene-d12	0.0429	7	0.0352	81.9	0.0031	8.8

TABLE 9 HP-3 Medium Concentration Quality Control

Analyte	True Spiked Value (ng/mL)	Number of Retained Values	Mean Recovery (ng/mL)	Mean Recovery (%)	Single Standard Deviation (So)	Relative Standard Deviation (%)
2-Methylnaphthalene-d10	26.7	7	26.7	100.1	0.859	3.2
Benz[a]anthracene-d12	0.25	7	0.199	81.0	0.015	7.5

TABLE 10 HP-4 High Concentration Quality Control

Analyte	True Spiked Value (ng/mL)	Number of Retained Values	Mean Recovery (ng/mL)	Mean Recovery (%)	Single Standard Deviation (So)	Relative Standard Deviation (%)
2-Methylnaphthalene-d10	283.9	7	230.7	81.3	11.0	4.8
Benz[a]anthracene-d12	2.61	7	2.13	81.7	0.13	5.9

concentration for both perdeuterated PAHs (see Figs. 1-4). The figures show the linearity of precision and accuracy with increasing concentration. The d12-benz(a)anthracene recoveries were consistently around 80 %. This may possibly indicate the consistent suppression of the mass spectral signal by a near-eluting compound. The recoveries for d10-2-methylnaphthalene ranged from 81 to 112 %. The repeatability for the known spike recoveries was consistent; the known spike RSDs ranged from 3 to 9 %. PAH concentration had no significant effect on the repeatability of the technique.

16. Quality Control Criteria

16.1 *Initial Calibration*:

16.1.1 The following acceptance criteria will be used for initial calibration: *(1)* The signal to noise (S/N) ratio for the GC signals present in every selected ion current profile (SICP) must be ≥10:1 for the labeled internal standards and calibration compounds; *(2)* The percent relative standard deviation (RSD) for the mean area ratio/ng for labeled internal standards and the calibration compounds must be less than 30 % for high molecular weight PAHs and less than 25 % for low molecular weight PAHs, and the $r^2 > 0.99$. The calibration curve must not be forced through the origin; *(3)* The number of calibration standards may be reduced from four to three based on the criteria in 12.3 of this test method.

16.1.2 The following corrective action will be adopted for initial calibration: *(1)* Initial calibration must be re-established if the RSD(s) exceed the limit(s); *(2)* The calibration will not be re-established in response to a nonconforming RSD if the sample results are less than the PQL.

16.2 *Daily Duplicate Calibration Verifications*:

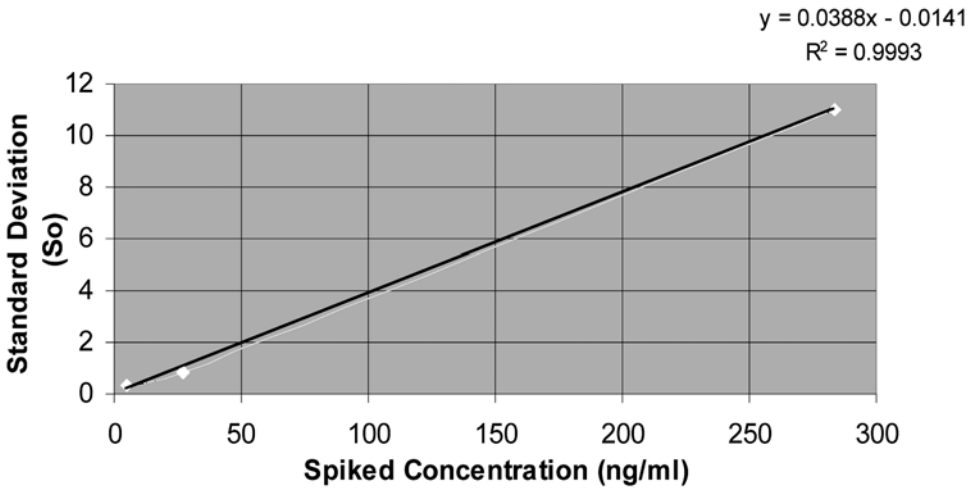


FIG. 1 2-Methylnaphthalene-d10 Single Standard Deviation versus Spiked Concentration

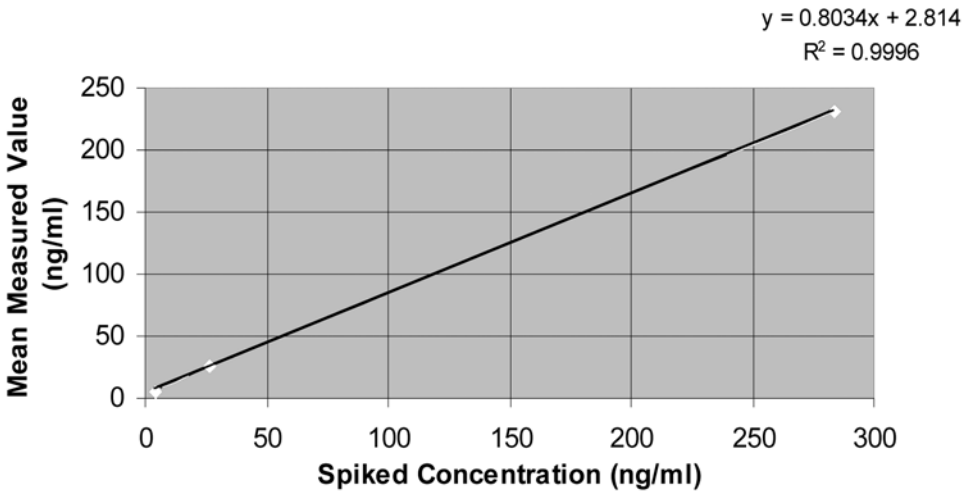


FIG. 2 Methylnaphthalene-d10 Mean Measured Value versus Spiked Concentration

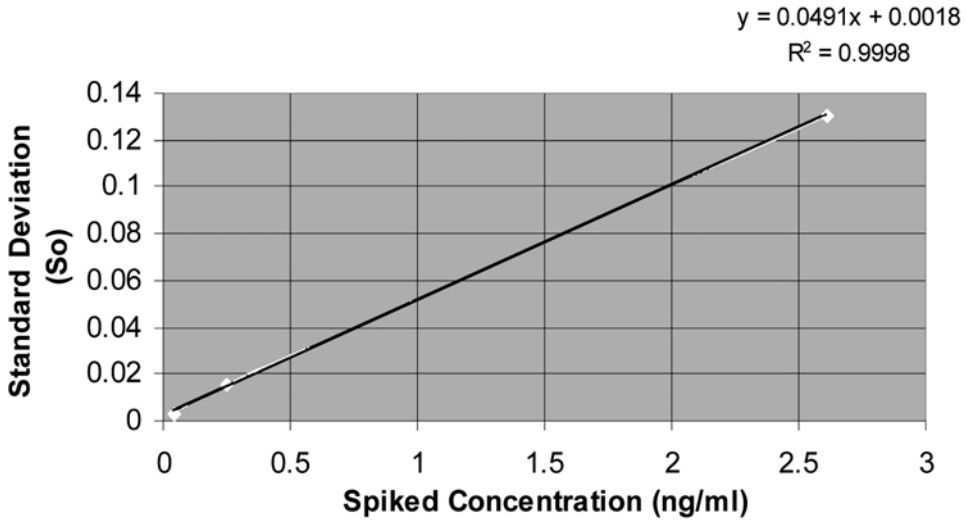


FIG. 3 Benz[a]anthracene-d12 Single Standard Deviation versus Spiked Concentration

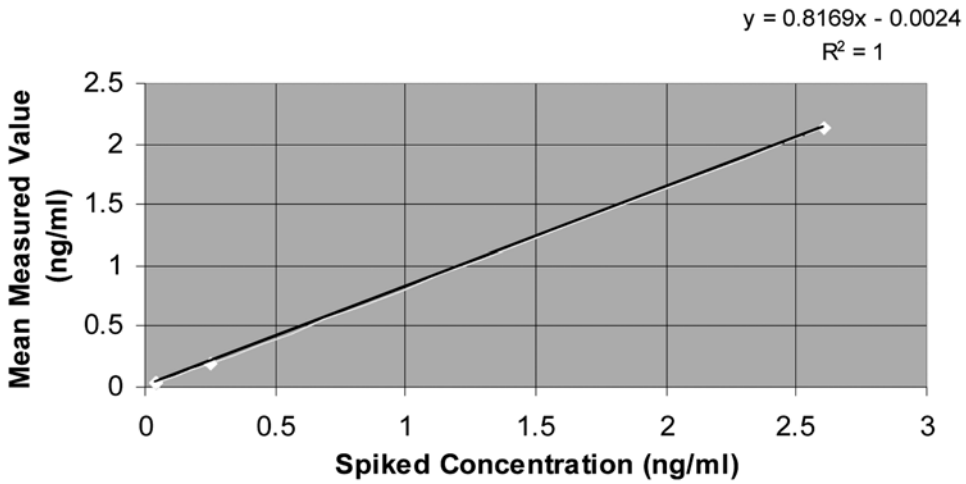


FIG. 4 Benz[a]anthracene-d12 Mean Measured Value versus Spiked Concentration

16.2.1 The following acceptance criteria will be used for daily duplicate calibration verifications: *(1)* The S/N ratio for the GC signals present in every SICP must be ≥10:1 for the labeled internal standards and the calibration compounds; *(2)*

The percent differences for the measured area ratio/ng of all analytes must be within ±25 % for high molecular weight PAHs and less than ±20 % for low molecular weight PAHs of the mean values established during the initial calibration.

16.2.2 The following corrective action will be adopted for daily duplicate calibration verifications if the first acceptance criterion is not satisfied: a new initial calibration curve must be established before sample extracts can be analyzed.

16.3 *Flocculation Blanks*:

16.3.1 The following acceptance criterion will be used for flocculation blanks: Prepared as needed to assess contamination from flocculation reagents and handling. Target analytes must not be detected above ⅓ of the target detection limits or >20 % of the associated sample result(s).

16.3.2 The following corrective action will be adopted for flocculation blanks: Locate the source of the contamination; correct the problem. Re-extract and reanalyze associated samples that are less than ten times the level of the contaminants present in the method blank.

16.4 *Extraction and Analytical Blanks*:

16.4.1 The following acceptance criterion will be used for extraction and analytical blanks: Analyzed between every sample to monitor the baseline. Target analytes must not be detected above ⅓ of the target detection limits or >20 % of the associated sample result(s).

16.4.2 The following corrective action will be adopted for extraction and analytical blanks: Locate the source of the contamination; correct the problem. Re-extract and reanalyze associated samples that are less than ten times the level of the contaminants present in the method blank.

16.5 *Signal to Noise Ration*:

16.5.1 The following acceptance criterion will be used for signal to noise ratio: The signal to noise (S/N) ratio for the GC signals present in every selected ion current profile (SICP) must be ≥3:1 for target compounds in environmental samples and ≥10:1 for the labeled internal standards.

16.5.2 The following corrective action will be adopted for signal to noise ratio: Reanalyze the sample unless obvious matrix interference is present.

APPENDIX

(Nonmandatory Information)

X1. ION PLOTS

X1.1 Selected ion chromatograms from a typical coal tar impacted pore water of d-PAH internal standards (top chromatogram of each page), and the related target parent and alkyl PAHs. Target species are indicated with brackets, and interfering species are marked with an “X.”

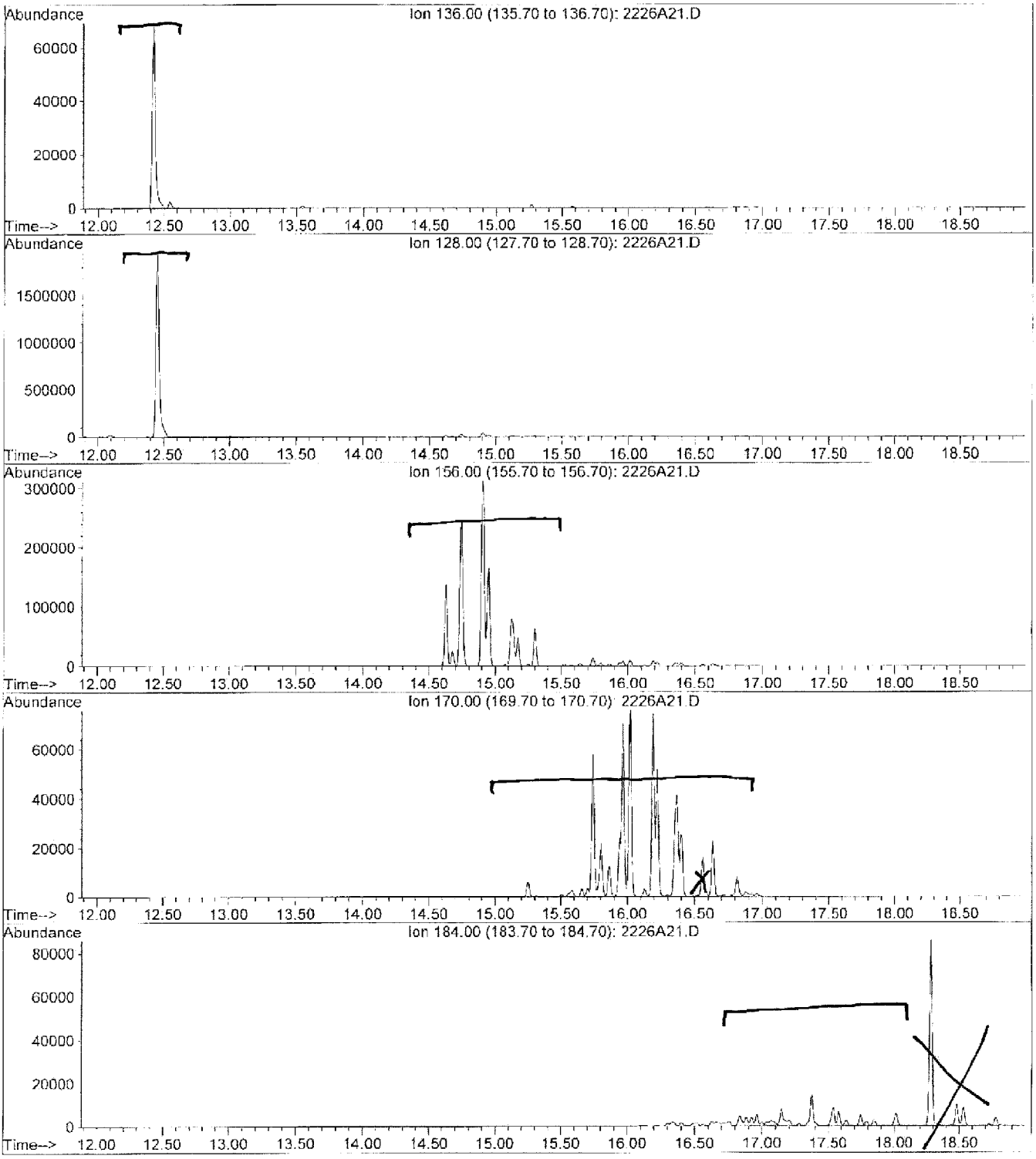


FIG. X1.1 Naphthalenes

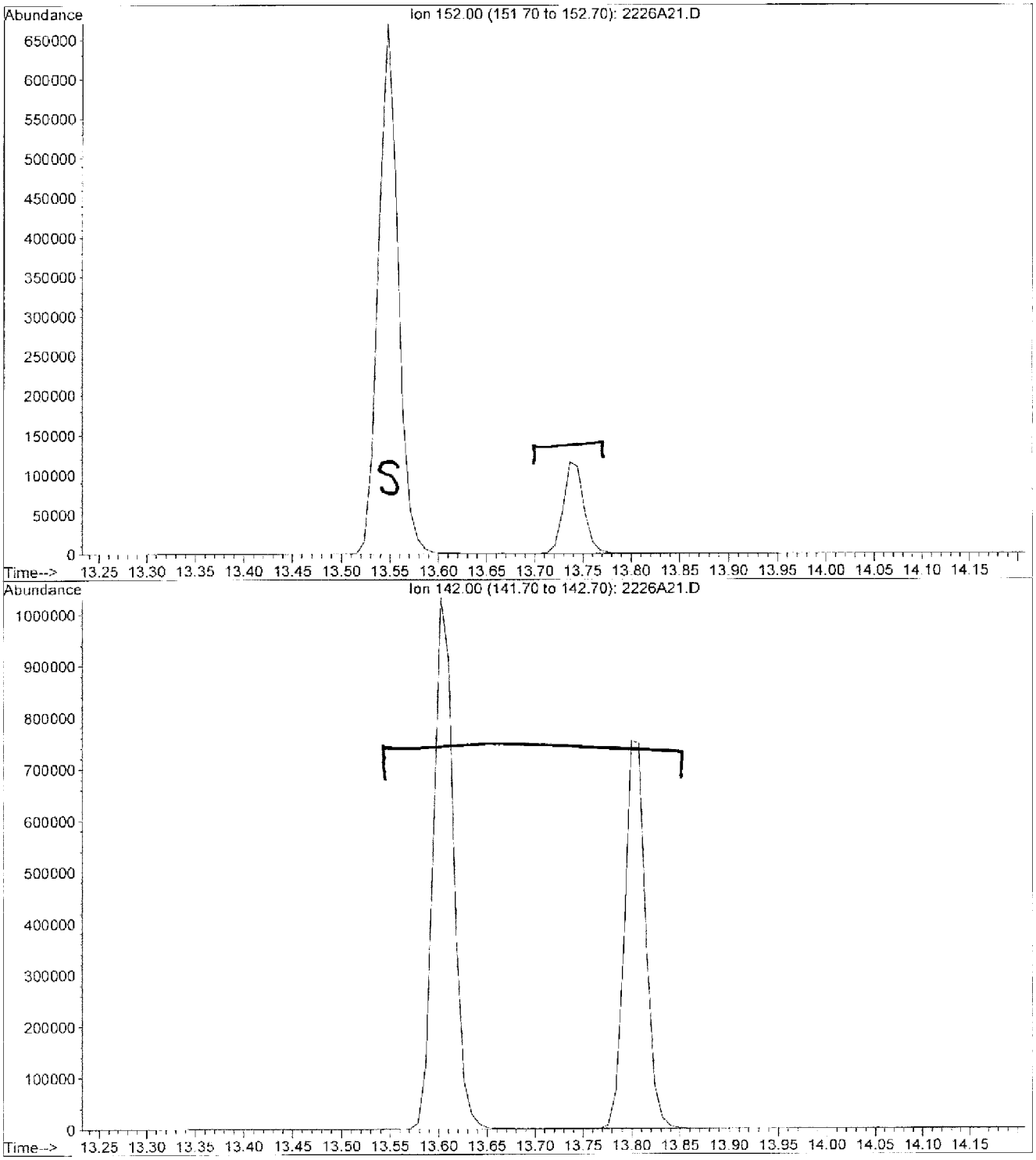


FIG. X1.2 Methylnaphthalenes
("s" is a spiked d_{10}-methylnaphthalene surrogate)

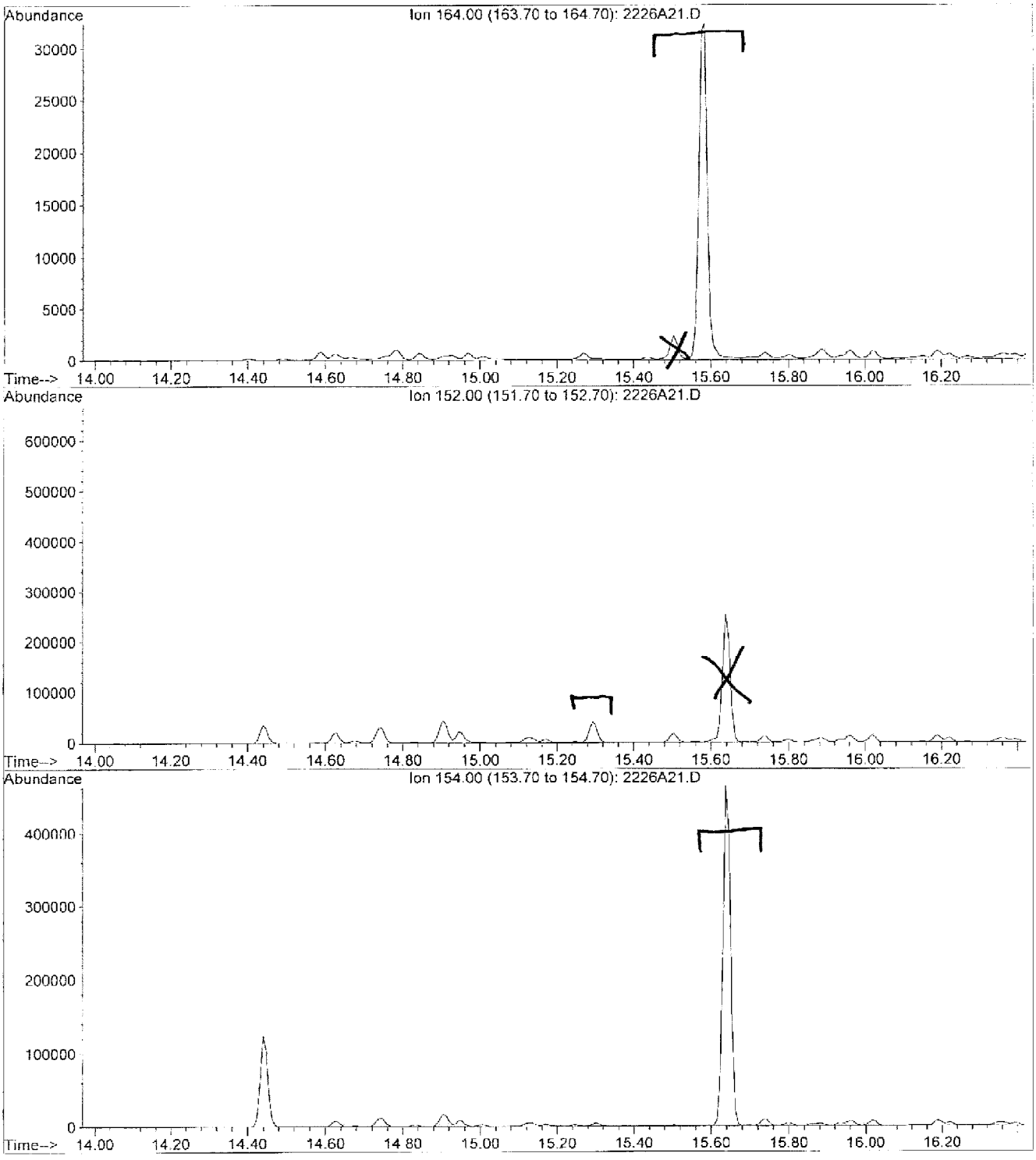


FIG. X1.3 Acenaphthylene/Acenaphthene

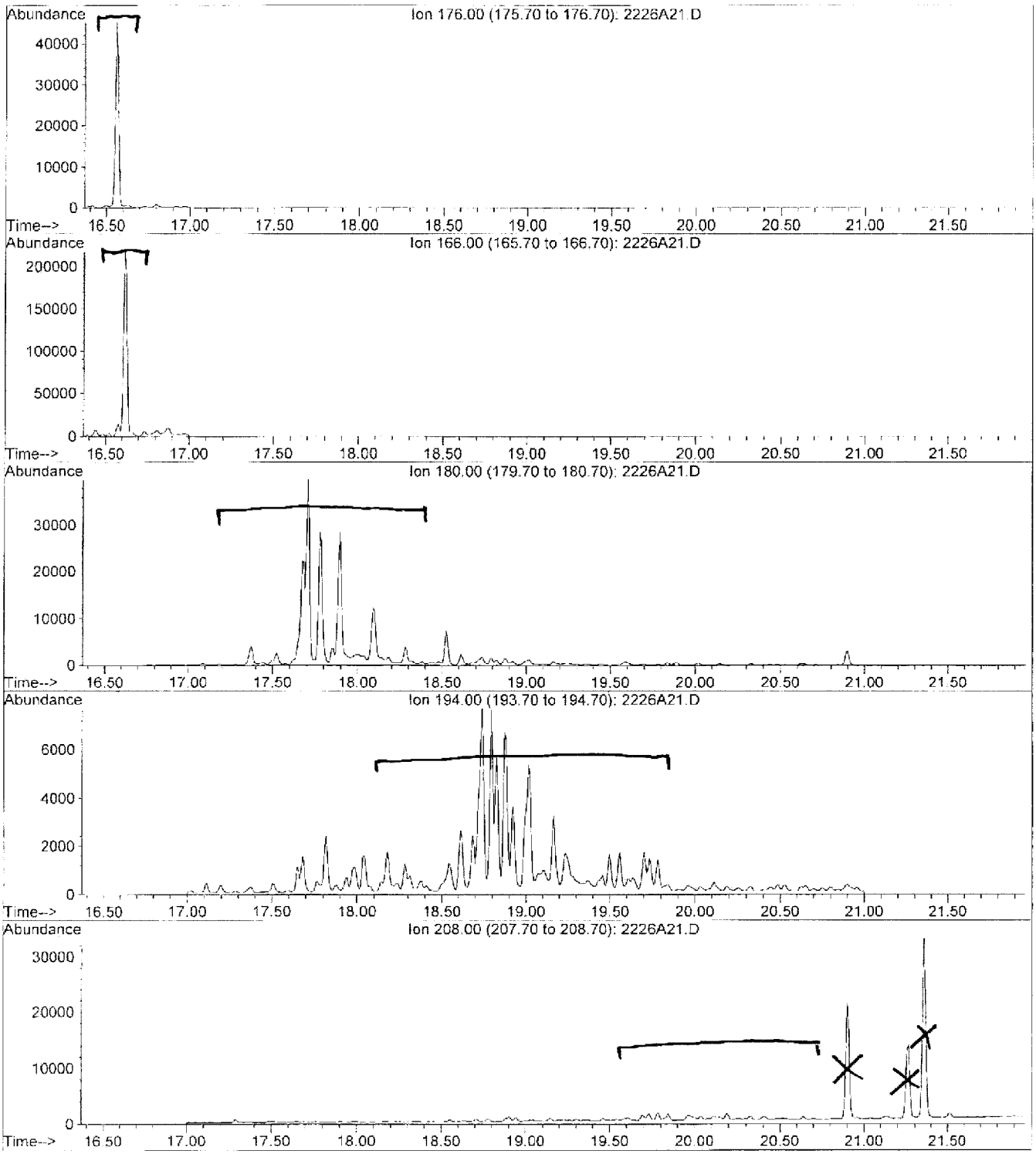


FIG. X1.4 Fluorenes

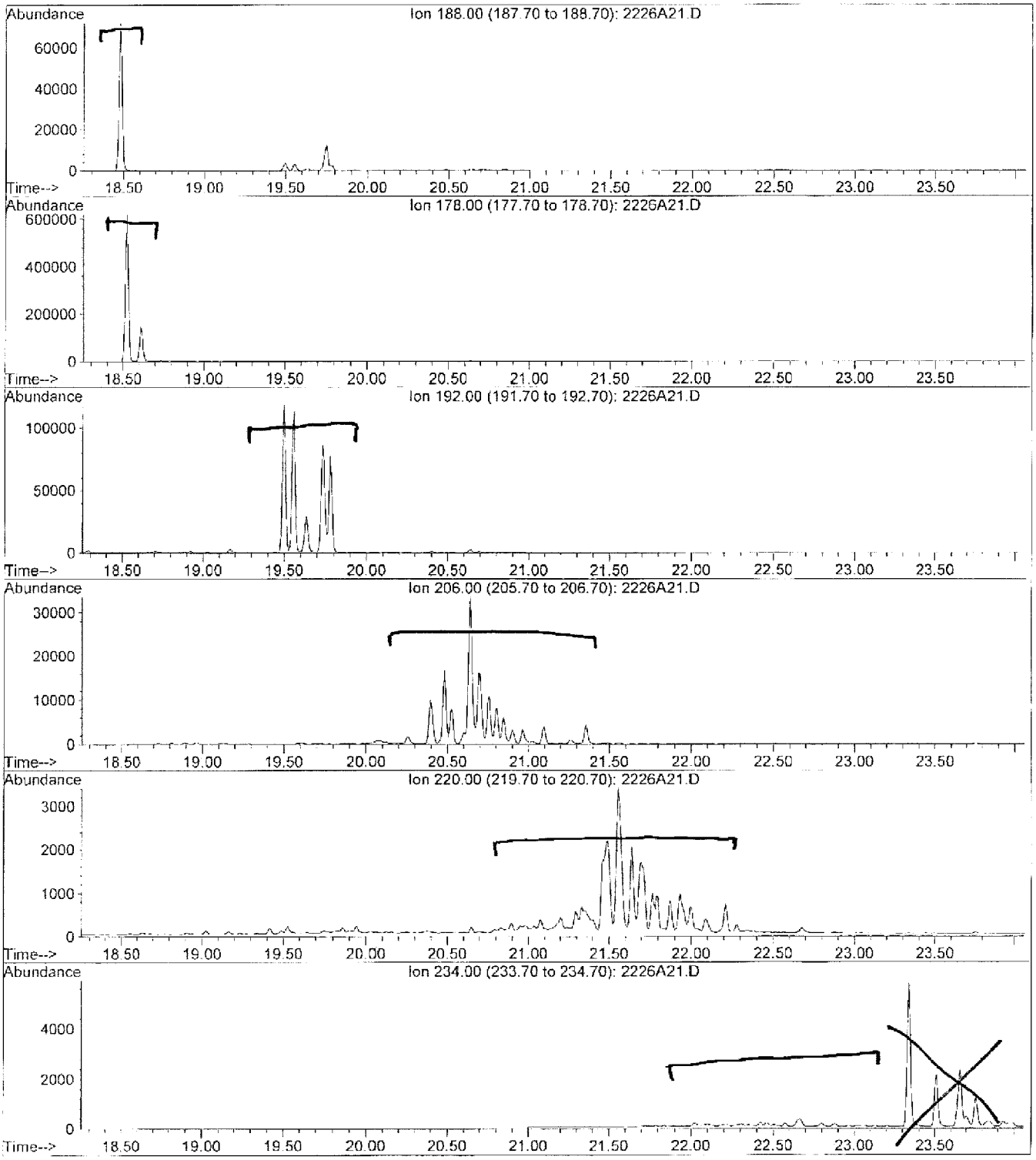


FIG. X1.5 Phenanthrenes/Anthracenes

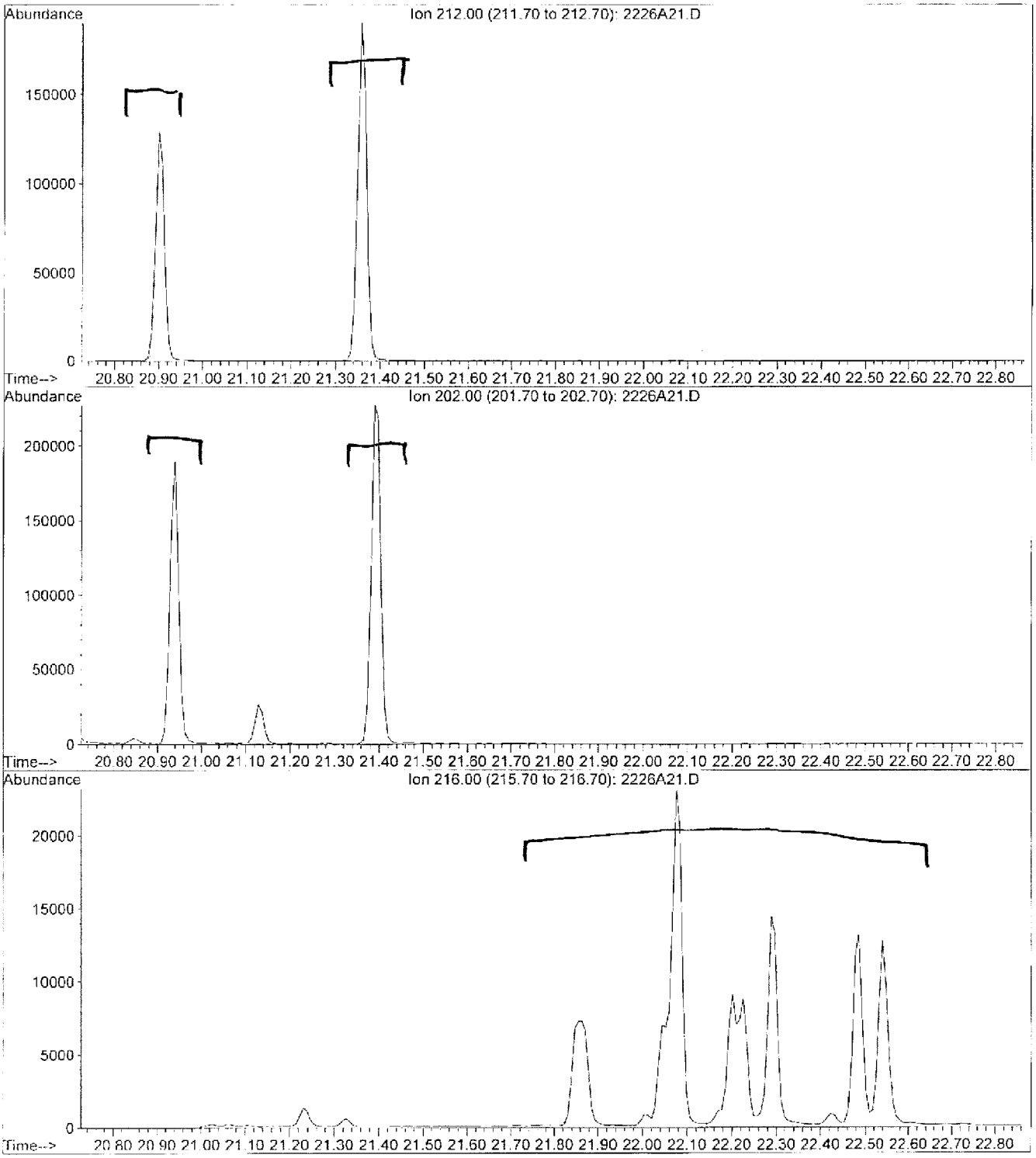


FIG. X1.6 Fluoranthenes/Pyrenes

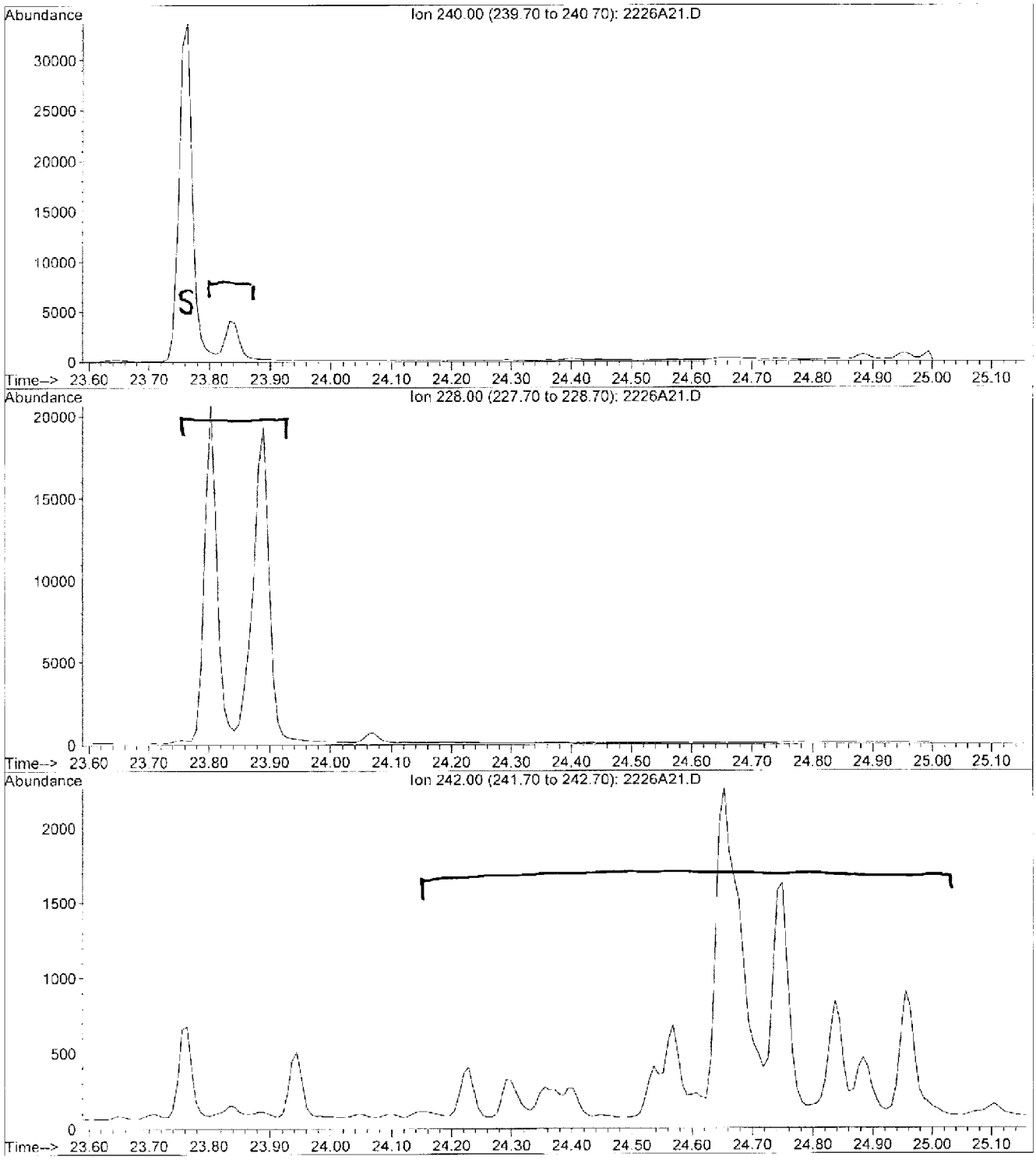


FIG. X1.7 Benz[a]anthracenes/Chrysenes
("s" is a spiked d_{12}-benz[a]anthracene surrogate)

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Appendix D: Site-Specific Health and Safety Plan

Prepared for:
United States Department of Defense, Environmental Security Technology Certification Program
Washington Navy Yard, Anacostia River, Washington, D.C.

HEALTH AND SAFETY PLAN

Sediment Sampling and Site Investigation
Washington Navy Yard
Anacostia River
Washington, D.C

ENSR Corporation
September 2007
Document No.: 09000-422

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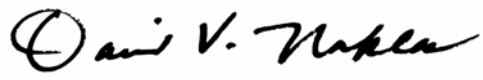
Prepared for:
United States Department of Defense, Environmental Security Technology Certification Program
Washington Navy Yard, Anacostia River, Washington D.C.

HEALTH AND SAFETY PLAN

Sediment Sampling and Site Investigation
Washington Navy Yard
Anacostia River
Washington, D.C



Prepared By Peter Sullivan, RHSM — September 10, 2007 Date



Reviewed By David V. Nakles, PE, PhD — September 19, 2007 Date

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September 2007
Document No.: 09000-422

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Contents

Emergency Information and Hazard Assessment 1

EMERGENCY REFERENCES 1

1.0 INTRODUCTION 1

1.1 ENSR Safety Policy 1
1.1.1 Maximum Duration of the Work Day for Field Activities 1
1.1.2 Short Service Employee 1
1.2 Health And Safety Plan (HASP) 1
1.2.1 HASP Purpose 1
1.2.2 HASP Applicability 1
1.3 Organization/Responsibility 2
1.3.1 ENSR Project Manager 2
1.3.2 ENSR Regional Health and Safety Manager 2
1.3.3 ENSR Site Safety Officer 3
1.3.4 ENSR Field Personal 4
1.3.5 Contractors 4
1.4 Management of Change/Modification of the HASP 4
1.4.1 Management of Change 4
1.4.2 HASP Modification 5

2.0 Site Description and History 6

2.1 Site Description 6
2.2 Site History 8
2.3 Contaminants of Concern 8

3.0 Scope of Work and Assumptions 9

3.1 Project Goals 9
3.2 Field Tasks 9
3.3 Assumptions 9

4.0 Chemical Hazard Assessment and Control 10

4.1 Chemical Contaminants of Concern 10
4.1.1 Polynuclear Aromatic Hydrocarbons (PNA's, PAH's, Polycyclic-. Semi-volatiles) 10
4.1.2 Copper 10
4.1.3 Lead, Inorganic 10
4.2 Summary of Hazardous Properties of Potential Contaminants 11
4.3 Hazard Substances Brought On Site by ENSR 12

4.4 Chemical Exposure and Control 12
4.4.1 Chemical Exposure Potential 12
4.4.2 Chemical Hazard Control 12
4.4.3 Hazardous Waste Management 12

5.0 Physical Hazards and Controls 13

5.1 Back Safety 13
5.2 Boating Hazards 13
5.2.1 Safe Boating Procedures 13
5.3 Cuts and Lacerations 17
5.4 Driving Safety 18
5.5 Hand Safety 18
5.5.1 Glove Selection 18
5.5.2 Hand Tools 19
5.6 Insects and Spiders 21
5.7 Noise Exposure 21
5.8 Poisonous Plants 22
5.9 Slips, trips and fall hazards 23
5.10 Traffic Safety 23
5.11 Water Hazards 24
5.12 Weather 24
5.12.1 Inclement Weather 24
5.12.2 Heat Stress 25
5.12.3 Cold Stress 26
5.12.4 Work/Rest Cycles for Cold Weather 27
5.13 Confined Spaces 28

6.0 Air Monitoring 29

6.1 Monitors 29
6.1.1 Photoionization Detector 29
6.2 Personal Air Sampling 29
6.3 Calibration and Recordkeeping 29

7.0 Personal Protective Equipment 30

7.1 Personal Protective Equipment 30
7.2 Respiratory Protection 31
7.3 Other Safety Equipment 31

8.0 Site Control 32

8.1 Designation of Zones....32
8.1.1 Exclusion Zone....32
8.1.2 Contamination Reduction Zone....32
8.1.3 Support Zone....32
8.1.4 Site Access Control....32
8.1.5 Parking and Staging Areas....32
8.1.6 Pedestrian Walkways....32
8.2 General Site Safety Practices....32

9.0 Decontamination....34
9.1 Personal Decontamination....34
9.2 PPE Decontamination....34
9.3 Equipment Decontamination....34

10.0 Medical Monitoring and Training Requirements....35
10.1 Medical Monitoring....35
10.2 Health and Safety Training....35
10.2.1 HAZWOPER....35
10.2.2 Pre-Entry Briefing....35

11.0 Emergency Response....36
11.1 Employee Training....36
11.2 Alarm System/Emergency Signals....36
11.3 Escape Routes and Procedures....36
11.4 Employee Accounting Method....37
11.5 Injuries and Illnesses....37
11.5.1 First Aid....37
11.5.2 Professional Treatment....37
11.6 Designation of responsible parties....37
11.7 Emergency Response Drills....37
11.8 Incident Reporting and Investigation....37

List of Attachments

Attachment A - Health and Safety Plan Review Form
Attachment B - Job Safety Analysis
Attachment C - Pre-Entry Briefing Attendance Form
Attachment D - Supervisor's Incident Investigation Report
Attachment E - Material Safety Data Sheets

Emergency Information and Hazard Assessment

Sediment Sampling and Site Investigation
Washington Navy Yard – Anacostia River
Washington, D.C.

EMERGENCY REFERENCES

Ambulance:	911
Fire:	911
Police:	911
Medical Services:	
DC General Hospital	202-698-5000

Directions from the Washington Navy Yard to DC General Hospital are below.

1:	Start out going EAST on M ST SE toward 11TH ST SE.	<0.1 miles
2:	Turn LEFT onto 11TH ST SE.	0.1 miles
3:	Turn RIGHT onto K ST SE.	<0.1 miles
4:	Turn SLIGHT LEFT onto POTOMAC AVE SE.	0.2 miles
5:	Turn SLIGHT LEFT.	<0.1 miles
6:	Turn SLIGHT RIGHT onto POTOMAC AVE SE.	0.3 miles
7:	Turn LEFT onto 18TH ST SE.	0.1 miles
8:	Turn RIGHT onto MASSACHUSETTS AVE SE.	<0.1 miles

9: End at **1900 Massachusetts Ave Se**
Washington, DC 20003-2542, US

When using any driving directions or map, it's a good idea to do a reality check and make sure the road still exists, watch out for construction, and follow all traffic safety precautions. This is only to be used as an aid in planning.

Underground Utilities

http://www.digsafely.com/contacts.htm

Washington, District of Columbia

Miss Utility

(800) 257-7777

Emergency Chemical Information

Chem-trec (Emergency Chemical Spill Response Information) (800) 424-9300

Poison Control Center

http://www.aapcc.org/poison4.htm

National Capital Poison Center

(800) 222-1222

Emergency Muster Point

In case of a site/facility emergency, please meet at the on-shore sample processing location to be determined at the beginning of sampling. The escape route from the site and an emergency muster point will be determined and provided to all workers during the project mobilization.

Client Contacts:

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Alameda, CA 94502
Telephone: 510-748-6900 Fax: 510-748-6915

Hazard Assessment

Chemical Hazards

Copper	Lead	Nickel

Physical Hazards

Cold	Pinch Points	Tripping
Heat	Poisonous Plants	Weather
Insects	Sharp Objects	Working In Water
Lifting	Splashing Liquids	Working Over Water
Noise	Traffic	

Personal Protective Equipment

PPE Item	General	Sample Collection	Sample Processing
Hard Hat	1 & 2	✓	✓
Traffic Vests	2	2	2
Steel Toed Safety Shoes	1	✓	✓
Safety Glasses with Sideshields	1	✓	✓
Goggles or Faceshield		3	3
Hearing Protection		4	4
Tyvek Coveralls		5	5
Nitrile Gloves	5	5	5
Ivy Block® or Ivy Screen® barrier cream	5 & 6	5 & 6	5 & 6
Type III Personal Floatation Device	7	7	7
Chest Waders		8	

✓ Required PPE

1 All employees must comply with United States Department of Defense safety requirements.

2 Traffic vests and hardhats are required when working within twenty feet of any public road or any private road with active traffic.

3 Goggles and/of face shields will be worn when splash hazards are present while conducting work.

4 Hearing protection should be worn around equipment if normal conversation cannot be understood.

5 Tyvek coveralls and Nitrile gloves are only required of those that are likely to come in direct contact with potentially contaminated sediments and water. Tyvek coveralls and Nitrile gloves will be worn to protect workers from poison ivy and poison oak when contact cannot be avoided.

6 Ivy Block® or Ivy Screen® barrier cream should be worn on exposed skin where there is a potential for exposure to poison ivy or oak.

7 Personal Floatation Devices are required when work is conducted in water three feet or greater in depth.

8 If sample collection involves entering shallow waters, employees will wear chest waders to avoid contact with potentially contaminated sediments and water.

Air Monitoring Instruments

Task	Instrument	Action Limit and Action
All tasks involving potential exposure to contaminated sediments and water	Photoionization Detector	25 ppm as isobutylene Don respiratory protection as discussed in Section 7

Respiratory Protection

Task	Action Limit	Respiratory Protection	Level
All tasks involving potential exposure to contaminated soils and/or groundwater	25 ppm as Isobutylene for 1 minute	Half or full face mask respirator with combination organic vapor/HEPA cartridges	C
	50 ppm as Isobutylene	Full face respirator with organic vapor/HEPA cartridges	C
	100 ppm as isobutylene	Supplied air respirator	B



I, certify that this hazard assessment and evaluation was performed on September 10, 2007.

1.0 INTRODUCTION

1.1 ENSR Safety Policy

ENSR Corporation is committed to providing our employees with a safe and healthy work environment. It is not only our obligation to each other, but also a sound business practice to do so. Work related injuries and illnesses cause needless pain and suffering, cost money, and adversely affect our reputation with our clients. It is our firm belief that all work related injuries and illnesses are preventable, and it is therefore our goal to have a workplace that is free from occupational injuries and illnesses. Every attempt shall be made to eliminate the possibility of injuries and illnesses. No aspect of the company's activities, including expediency and cost, shall take precedence over the health and safety of our employees.

1.1.1 Maximum Duration of the Work Day for Field Activities

An employee may not work a shift that exceeds 16 hours in duration. For the purpose of this policy, the work shift includes time spent at lunch and on break. If an employee works more than one shift during the course of a calendar day, the total number of hours worked in that day cannot exceed 16 hours. Exception: If work is to be done continuously in ambient air temperatures of less than 20^{o} F, the Site Safety Officer and Field Manager will use a guideline of limiting work shifts to 10 hours in duration, including 8 hours working outdoors and 2 hours of time spent at lunch, breaks, and travel. Refer to Section 5.12.3, Cold Stress, fur further work day guidelines.

1.1.2 Short Service Employee

A Short Service Employee (SSE) is an employee with fewer than six months experience working supervised on field projects or an employee who has not completed required training or received required certifications.

Short Service Employees will not be assigned to this project unless they are supervised on site by a qualified person.

Short Service Employees will be identified in the field by a three inch long strip of red vinyl tape placed vertically on both sides of the SSE's hard hat. Note that clients may have specific procedures for identifying SSEs. The Project Manager must determine the identification method required by the client or project.

Field crews with less than five workers shall have no more than one SSE.

Field crews with five or more workers shall not have more than 20% SSE personnel.

1.2 Health And Safety Plan (HASP)

1.2.1 HASP Purpose

The purpose of this HASP is to identify hazards associated with this project and specify engineering and administrative controls and personal protective equipment necessary to mitigate the risks associated with these hazards. This HASP addresses the hazards recognized prior to writing or updating the documents. As new hazards are encountered, a Job Hazard Assessment (JHA) or Job Safety Analysis (JSA) must be conducted and the results input into the HASP.

This HASP also assigns responsibilities for the implementation of safety programs on this project and defines monitoring and emergency response planning specific to the project.

1.2.2 HASP Applicability

This site-specific Health and Safety Plan (HASP) has been developed by ENSR Corporation (ENSR). It establishes the health and safety procedures required to minimize potential risk to ENSR and contractor

personnel involved with the sediment sampling and site investigation at the Washington Navy Yard, located on the Anacostia River in Washington, D.C.

The provisions of this plan apply to ENSR and ENSR subcontractor personnel who will potentially be exposed to safety and/or health hazards related to activities described in Section 3.0 of this document.

Client and third party employees performing work that potentially exposes them to the chemical hazards at the site must work under their own HASP or read, sign, and work under the requirements of this HASP.

This HASP has been written to comply with the requirements of the Occupational Safety and Health Administration (OSHA) Personal Protective Equipment Standard (29 CFR 1910.132) for all activities and the OSHA Hazardous Waste Operations and Emergency Response Standard (29 CFR 1910.120) for tasks where there are potential exposures to subsurface contaminants. All activities covered by this HASP must be conducted in complete compliance with this HASP and with all applicable federal, state, and local health and safety regulations. Personnel covered by this HASP who cannot or will not comply will be excluded from site activities.

This plan will be distributed to each employee involved with the proposed activities at the site, including subcontractor employees. Each employee must sign a copy of the attached health and safety plan sign-off sheet (see Attachment A).

This HASP only pertains to the tasks that are listed in Section 3.0. A task specific HASP or addendum to this HASP will be developed at a later date for any other subsequent investigative/remedial activities at the site.

1.3 Organization/Responsibility

The implementation of health and safety at this project location will be the shared responsibility of the ENSR Project Manager (PM), the ENSR Regional Health and Safety Manager (RHSM), the ENSR Project Site Safety Officer (SSO) and other ENSR personnel and ENSR's contractors implementing the proposed scope of work.

1.3.1 ENSR Project Manager

The ENSR PM (David Nakles) is the individual who has the primary responsibility for ensuring the overall health and safety of this project. As such, the PM is responsible for ensuring that the requirements of this HASP are implemented. Some of the PM's specific responsibilities include:

- Assuring that all personnel to whom this HASP applies, including ENSR subcontractors, have received a copy of it;
- Providing the RHSM with updated information regarding conditions at the site and the scope of site work;
- Providing adequate authority and resources to the on-site SSO to allow for the successful implementation of all necessary safety procedures;
- Supporting the decisions made by the SSO and RHSM;
- Maintaining regular communications with the SSO and, if necessary, the RHSM;
- Coordinating the activities of all ENSR subcontractors and ensuring that they are aware of the pertinent health and safety requirements for this project, and
- Conducting random project audits.

1.3.2 ENSR Regional Health and Safety Manager

The ENSR RHSM (Peter Sullivan) is the individual responsible for the preparation, interpretation and modification of this HASP. Modifications to this HASP which might result in less stringent precautions cannot be undertaken by the PM or the SSO without the approval of the RHSM. Specific duties of the RHSM include:

- Writing, approving and amending the HASP for this project;
- Advising the PM and SSO on matters relating to health and safety on this site;
- Recommending appropriate personal protective equipment (PPE) and respiratory equipment to protect personnel from potential site hazards;
- Facilitating Incident investigations; and,
- Maintaining regular contact with the PM and SSO to evaluate site conditions and new information which might require modifications to the HASP; and
- Conducting random project audits.

1.3.3 ENSR Site Safety Officer

All ENSR field technicians are responsible for implementing the safety requirements specified in this HASP. However, one field technician will serve as the SSO. The SSO will be appointed by the PM. The SSO will be on-site during all activities covered by this HASP. The SSO is responsible for enforcing the requirements of this HASP once work begins. The SSO has the authority to immediately correct all situations where noncompliance with this HASP is noted and to immediately stop work in cases where an immediate danger is perceived. Some of the SSO's specific responsibilities include:

- Assuring that all personnel to whom this HASP applies, including all subcontractors, have reviewed this HASP, and submitted a completed copy of the HASP review and acceptance form (Attachment A);
- Assuring that all personnel to whom this HASP applies have attended a pre-entry briefing and any subsequent safety meetings that are conducted during the implementation of the program;
- Maintaining a high level of health and safety consciousness among employees implementing the proposed investigative activities;
- **Securing Work Permits. The SSO must determine what, if any, work permits must be secured from the facility prior to the commencement of activities. If required, the SSO must determine how long the work permit is good for and verify that all the provisions of the work permit can be met by ENSR and its subcontractors.**
- Procuring the air monitoring instrumentation required and performing air monitoring for investigative activities;
- Procuring and distributing the PPE and safety equipment needed for this project for ENSR employees;
- Verifying that all PPE and health and safety equipment used by ENSR is in good working order;
- Verifying that ENSR contractors are prepared with the PPE, respiratory protection and safety equipment required for this program;
- Preparing an initial Job Safety Analysis (JSA) during the initial mobilization and revising the Job Safety Analysis if conditions or tasks change and communicating with all workers the results of the Job Safety Analysis. See attachment B for a JSA form. The JSA will be reviewed daily by all workers and updated as needed.
- Notifying the PM of all noncompliance situations and stopping work in the event that an immediate danger situation is perceived;
- Monitoring and controlling the safety performance of all personnel within the established restricted areas to ensure that required safety and health procedures are being followed;
- Conducting accident/incident investigations and preparing accident/incident investigation reports;
- Conducting the pre-entry briefing prior to beginning work and subsequent safety meetings as necessary; and

- Initiating emergency response procedures in accordance with Section 11.0 of this HASP.

1.3.4 ENSR Field Personal

All ENSR field personnel covered by this HASP are responsible for following the health and safety procedures specified in this HASP and for performing their work in a safe and responsible manner. Some of the specific responsibilities of the field personnel are as follows:

- Assess each task prior to beginning work on that task for hazards and necessary precautions.
- Assess the work area for changing conditions and new hazards and address the hazards;
- Stop work and initiate corrective actions if work site hazards create unacceptable risk;
- Reading this HASP in its entirety prior to the start of on-site work;
- Submitting a completed HASP Review and acceptance form (Attachment A)to the ENSR SSO prior to the start of work;
- Attending the required pre-entry briefing prior to beginning on-site work and any subsequent safety meetings that are conducted during the implementation of the program;
- Bringing forth any questions or concerns regarding the content of the HASP to the PM or the SSO prior to the start of work;
- Reporting all Incidents, injuries and illnesses, regardless of their severity, to the ENSR SSO; and,
- Complying with the requirements of this HASP and the requests of the SSO.

1.3.5 Contractors

Additionally, contractors hired by ENSR are responsible for:

- Reading the HASP in its entirety prior to the start of on-site work;
- Attending the required pre-entry briefing prior to beginning on-site work and any subsequent safety meetings that are conducted during the implementation of the program;
- Ensuring that their equipment is in good working order via daily inspections;
- Operating their equipment in a safe manner;
- Appointing an on-site safety coordinator to interface with the ENSR SSO;
- Providing ENSR with copies of material safety data sheets (MSDS) for all hazardous materials brought on-site; and,
- Providing all the required PPE, respiratory equipment and safety supplies to their employees.

1.4 Management of Change/Modification of the HASP

1.4.1 Management of Change

This document discusses the physical hazards associated with the proposed activities. However, unanticipated site-specific conditions or situations might occur during the implementation of this project. Also, ENSR and/or the contractors may elect to perform certain tasks in a manner that is different from what was originally intended due to a change in field conditions. As such, this HASP must be considered a working document that is subject to change to meet the needs of this dynamic project.

ENSR and/or ENSR's contractors will complete a Job Safety Analysis (JSA) when new tasks or different investigative techniques not addressed in the HASP are proposed. The use of new techniques will be reviewed and if new hazards are associated with the proposed changes, they will be documented on the JSA

form. An effective control measure must also be identified for each new hazard. JSA forms will be reviewed by the SSO prior to being implemented. Once approved, the completed forms will be reviewed with all field staff during the daily safety meeting. A blank JSA form is presented as Attachment B.

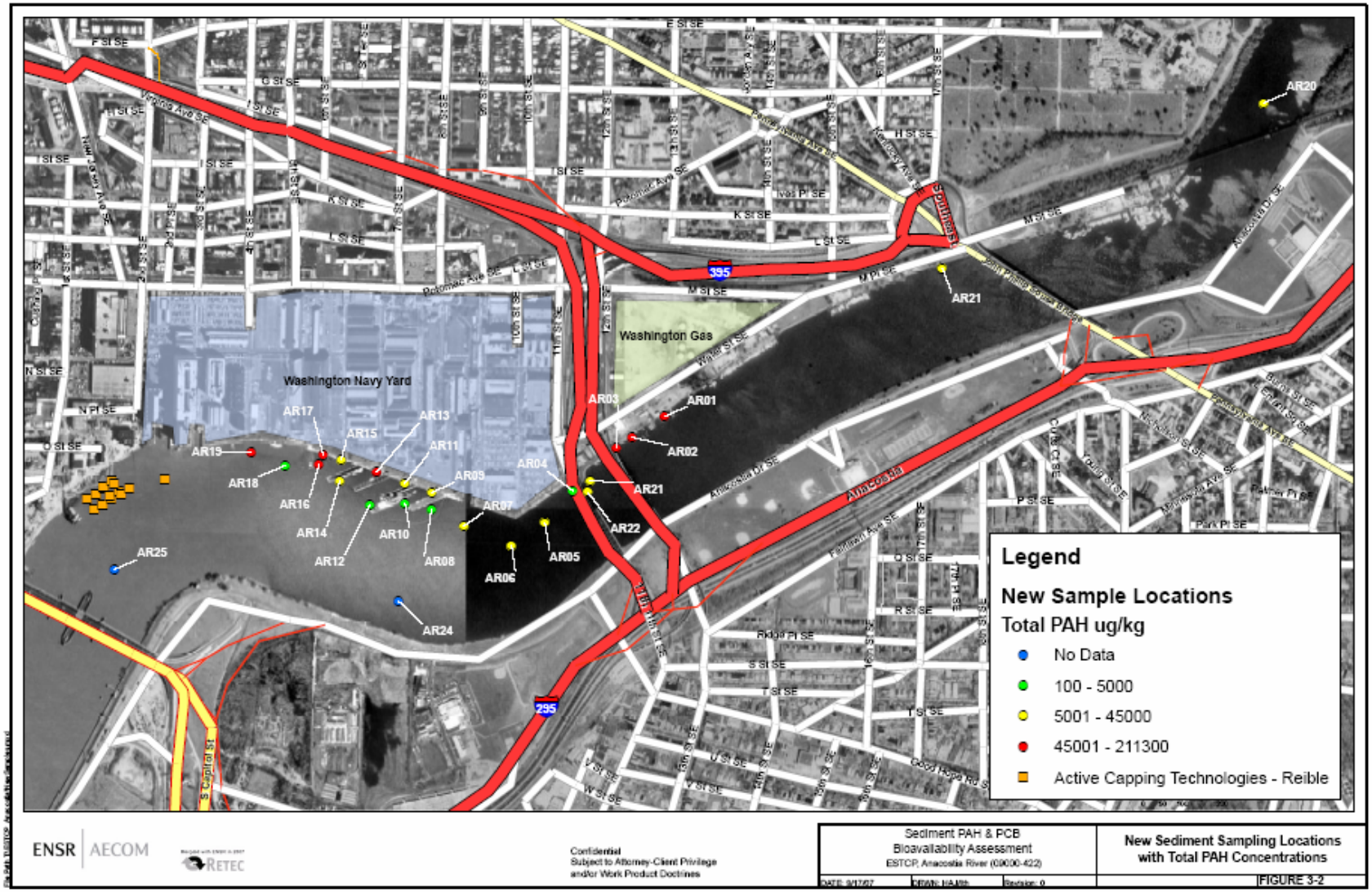
1.4.2 HASP Modification

Should significant information become available regarding potential on-site hazards, it will be necessary to modify this HASP. All proposed modifications to this HASP must be reviewed and approved by the ENSR RHSM before such modifications are implemented. Any significant modifications must be incorporated into the written document as addenda and the HASP must be reissued. The ENSR PM will ensure that all personnel covered by this HASP receive copies of all issued addenda. Sign-off forms will accompany each addendum and must be signed by all personnel covered by the addendum. Sign-off forms will be submitted to the ENSR PM. The HASP addenda should be distributed during the daily safety meeting so that they can be reviewed and discussed. Attendance forms will be collected during the meeting.

2.0 Site Description and History

2.1 Site Description

The Washington Navy Yard is located on the Anacostia River in Washington, D.C. The following aerial photograph depicts the site and anticipated locations of sediment sampling activities.


Washington Navy Yard
Washington Gas
AR01
AR02
AR03
AR04
AR05
AR06
AR07
AR08
AR09
AR10
AR11
AR12
AR13
AR14
AR15
AR16
AR17
AR18
AR19
AR20
AR21
AR22
AR24
AR25
Legend
New Sample Locations
Total PAH ug/kg
No Data
100 - 5000
5001 - 45000
45001 - 211300
Active Capping Technologies - Reible
ENSR | AECOM
RETEC
Confidential
Subject to Attorney-Client Privilege
and/or Work Product Doctrines
Sediment PAH & PCB
Bioavailability Assessment
ESTCP, Anacostia River (09000-422)
New Sediment Sampling Locations
with Total PAH Concentrations
FIGURE 3-2

2.2 Site History

Environmental impacts at the facility are being investigated due to historical operation of a manufactured gas plant adjacent to the project site. The Washington Navy Yard is the U.S. Navy's oldest shore establishment, in operation since the first decade of the 19th century. It evolved from a shipbuilding center to an ordnance plant and then to the ceremonial and administrative center for the Navy. The yard is home to the Chief of Naval Operations and is headquarters for the Naval Historical Center, the Marine Corps Historical Center, and numerous naval commands.

On July 28, 1998 the WNY yard was listed on the National Priorities List (NPL) by the U.S. EPA, pursuant to the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980, and the Superfund Amendments and Reauthorization Act (SARA) of 1986. The Chesapeake Bay Foundation and Anacostia Watershed Society have been working with the Navy and EPA to develop an environmental project that would benefit the community. Local community concern is focused on pollution in the Anacostia River. EPA Region III is also working to identify other Federal and non-Federal facilities that may be contributing to pollution in the Anacostia River as part of our involvement with the Anacostia Watershed Toxics Alliance. The Navy took sediment samples in the Anacostia River in September/October 2006.

2.3 Contaminants of Concern

The contaminants of concern at this site are PAHs with concentrations varying from 0.1 to 211 ppm.

3.0 Scope of Work and Assumptions

3.1 Project Goals

The purpose of the investigation is to determine the environmental liabilities and ecological risks associated with PAH impacts of sediments at the facility.

3.2 Field Tasks

The specific field tasks being implemented at the site include:

- Collection of surface and sub-surface water samples with field screening/analysis activities. Samples will be collected while wading in shallow water as well as from a boat in the Anacostia River;
- Collecting sediment samples with field screening/analysis activities. Samples will be collected while wading in shallow water as well as from a boat in the Anacostia River; and,
- Field processing sediment samples to homogenize samples prior to packaging and shipment for laboratory analysis.

3.3 Assumptions

- All work can be performed using Level D Personal Protective Equipment;
- Personal floatation devices will be worn by all personnel working in or adjacent to water that is 3 feet or greater in depth; and,
- No confined spaces will be entered on this project.

4.0 Chemical Hazard Assessment and Control

4.1 Chemical Contaminants of Concern

4.1.1 Polynuclear Aromatic Hydrocarbons (PNA's, PAH's, Polycyclic-. Semi-volatiles)

Polynuclear Aromatic Hydrocarbons are various combinations of three or more closed (benzene) rings, together with attached molecular structures. They occur naturally in coal, petroleum, tars, pitches, and woods, and may be formed in fires involving heavy hydrocarbon materials.

Examples of PAH's, or PNA's, are anthracene, benzo(a)pyrene, chrysenes, fluoranthcene, naphthacene, and pyrenes, among many others. Many of the PAHs are carcinogenic. As a class they should be treated as carcinogens and exposures kept to a minimum. There are no OSHA permissible exposure limits for most of the specific compounds, however the "Coal Tar Pitch Volatiles" PEL should be used (0.2 mg/m3). PAHs are generally solids and not very volatile, making ingestion or inhalation of dust or smoke the likely routes of exposure.

4.1.2 Copper

Copper is a reddish-brown metal that is widely used in the electronics industry, plumbing, heating, roofing, chemical, pharmaceutical machinery and in building construction.

Exposure to copper dust and fume can irritate the eyes, nose and throat causing coughing, wheezing, nosebleeds, ulcers, and a hole in the "bone" dividing the inner nose. Copper fume may cause "metal fume fever". This is a flu-like illness with symptoms of metallic taste, fever, and chills, aches, chest tightness and cough. A dermal exposure to copper may result in a skin allergy. Repeated high exposures to copper can adversely affect the liver.

The Occupational Safety and Health Administration (OSHA) has set an occupational exposure limit of 1 milligram of copper dust and mist per cubic meter of air (1 mg/m^3) and 0.2 $mg/m^{3\ for}$ copper fume for an 8-hour workday, 40-hour workweek.

4.1.3 Lead, Inorganic

The OSHA PEL for inorganic lead is 50 $\mu g/m^3$, and is recommended to be 40 $\mu g/m^3$ if workers are of reproductive age. In general, the inhalation of metal dusts is irritating to the upper respiratory tract and nasal mucous membranes. Most metal dusts cause dermatitis and/or eye irritation. The early symptoms of lead poisoning, as a result of overexposure (either through ingestion or inhalation) include fatigue, sleep disturbance, headache, aching bones and muscles, digestive irregularities, abdominal pains, and decreased appetite. Chronic overexposures to lead affect the central nervous system and male and female reproductive systems. Lead has also been identified as a fetotoxin.

Nickel compounds are also used for nickel plating, to color ceramics, to make some batteries, and as substances known as catalysts that increase the rate of chemical reactions. Nickel and its compounds have no characteristic odor or taste.

Nickel can enter the body by:

- By breathing air or smoking tobacco containing nickel
- By eating food containing nickel, which is the major source of exposure for most people
- By drinking water which contains small amounts of nickel
- By handling coins and touching other metals containing nickel

The most common adverse health effect of nickel in humans is an allergic reaction. People can become sensitive to nickel when jewelry or other things containing it are in direct contact with the skin. Once a person is sensitized to nickel, further contact with it will produce a reaction. The most common reaction is a skin rash at the site of contact.

Less frequently, some people who are sensitive to nickel have asthma attacks following exposure to nickel. People who are sensitive to nickel have reactions when it is in contact with the skin, and some sensitized persons react when they eat nickel in food, drink it in water, or breathe dust containing it.

Lung effects, including chronic bronchitis and reduced lung function, have been observed in workers who breathed large amounts of nickel. Current levels of nickel in workplace air are much lower than in the past, and today few workers show symptoms of nickel exposure.

People who are not sensitive to it must eat very large amounts of nickel to show adverse health effects. Workers who accidentally drank water containing very high levels of nickel (100,000 times more than in normal drinking water) had stomachaches and effects to their blood and kidneys.

Animal studies show that breathing high levels of nickel compounds may result in inflammation of the respiratory tract. Eating or drinking large amounts of nickel has been reported to cause lung disease in dogs and rats and to affect the stomach, blood, liver, kidneys, immune system, and reproduction and development in rats and mice.

The Occupational Safety and Health Administration (OSHA) has set an occupational exposure limit of 1 milligram of nickel per cubic meter of air (1 mg/m^3) for an 8-hour workday, 40-hour workweek.

4.2 Summary of Hazardous Properties of Potential Contaminants

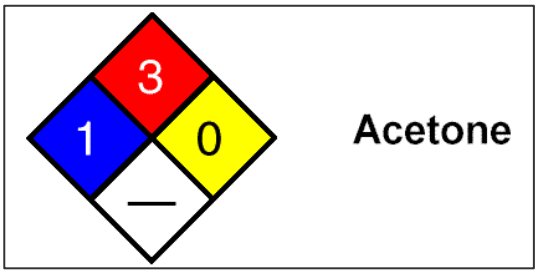
Chemical Name	PEL[1]	TLV[2]	VP[3]	VD[4]	SG[5]	SOL[6]	FP[7]	LEL[8]	UEL[9]
Copper Fume Dusts/mist	0.1 1.0 mg/m^3	0.2 1 mg/m^3	NA	NA	NA	NA	NA	NA	NA
Lead	50 µg/m^3	50 µg/m^3	NA	NA	11.3	NA	NA	NA	NA
Nickel Metal Soluble compound Insol. compound	 1 mg/m^3	1.5mg/m^3 0.1mg/m^3 0.2mg/m^3	NA	NA	NA	NA	NA	NA	NA

[1] Permissible Exposure Limit in ppm
[2] Threshold Limit Value in ppm
[3] Vapor Pressure in mm Hg
[4] Vapor Density (air = 1)
[5] Specific Gravity (water = 1)
[6] Solubility in Water in %
[7] Flash Point in °F
[8] Lower Explosive Limit in % by volume
[9] Upper Explosive Limit in % by volume
NA = Not Applicable
? = Not known
C = Ceiling limit not to be exceeded

4.3 Hazard Substances Brought On Site by ENSR

A material safety data sheet (MSDS) must be available for each hazardous substance that ENSR or its contractors bring on the property. This includes solutions/chemicals that will be used to decontaminate sampling equipment and gases needed to calibrate air monitoring equipment.

In addition, all containers of hazardous materials must be labeled in accordance with OSHA's Hazard Communication Standard. Either the original manufacturer's label or an NFPA 704M label specific for the material (as shown at the right) is considered to be an acceptable label.



4.4 Chemical Exposure and Control

4.4.1 Chemical Exposure Potential

Employees can be exposed by inhalation to the chemicals of concern during the sediment sampling and processing activities. Another route of potential exposure to the contaminants of concern is via direct dermal contact with sediments and water during sampling.

Although highly unlikely, exposure to all of the contaminants of concern can occur via ingestion (hand-to-mouth transfer). The decontamination procedures described in Section 9.0 address personal hygiene issues that will limit the potential for contaminant ingestion.

4.4.2 Chemical Hazard Control

The chemical hazards associated with the investigative and sampling activities can be controlled in several ways, including:

ENSR will perform air monitoring (Section 6) in the worker's breathing zone to determine exposure to the chemicals of concern during the installation of soil borings and the sampling program. If exposures exceed the action levels, respiratory protection as discussed in Section 7, will be donned.

To avoid direct dermal contact with contaminated media, protective clothing, as described in Section 7 will be required when collecting samples and decontaminating sampling equipment.

4.4.3 Hazardous Waste Management

All wastes generated at the facility will be containerized in accordance with client requirements. Once containerized, the project team will assist in the characterization and profiling of wastes generated to support the client in management and disposal of the wastes.

5.0 Physical Hazards and Controls

5.1 Back Safety

Using the proper techniques to lift and move heavy pieces of equipment is important to reduce the potential for back injury. The following precautions should be implemented when lifting or moving heavy objects:

- Bend at the knees, not the waist. Let your legs do the lifting;
- Do not twist while lifting;
- Bring the load as close to you as possible before lifting;
- Be sure the path you are taking while carrying a heavy object is free of obstructions and slip, trip and fall hazards;
- Use mechanical devices to move objects that are too heavy to be moved manually; and,
- If mechanical devices are not available, ask another person to assist you.

5.2 Boating Hazards

Work involving boats has many risks to employees. Potential hazards are: collisions with other craft or obstacles, embarking or disembarking accidents (slips, trips, falls, and trauma accidents), line or rope entanglement, noise, and weather-related incidents. Fueling, pulling loads, poor sea conditions, difficulty in communicating, inadequate boat or motor for sea conditions, and improper safety gear all create additional hazards to personnel. Drowning and hypothermia are very real hazards if boating is not done properly and safely.

5.2.1 Safe Boating Procedures

The project manager should assure that the boat is appropriate for this project, and that the captain is competent for the job.

The following safe work practices are to be followed by those personnel working on the boat:

5.2.1.1 General Precautions

- Prior to leaving shore, a plan of the day's activities, including time and place of departure, anticipated return time, and list of the persons on board the vessel should be left with a responsible person on shore in case of emergency.

  The crew will remain in radio contact with a designated observer or responsible personnel on shore during these survey activities. This on-shore contact will initiate emergency actions from the shore when required.

- Type I, II, or III life vests must be worn at all times when working on or near water.

- The maximum capacity (weight and passenger number) of the boat shall not be exceeded at any time (this number is listed on the boat tag).

- All equipment must be secured to the boat or securely stowed during transit.

- All passengers are to be informed of the locations of all safety equipment on the boat, including first-aid kit, fire extinguisher, life-vests and throw-rings, radio, etc.

- Appropriate footwear should be worn when it is necessary to access the shoreline by wading, and non-skid footwear must be worn on board.

- Employees should dress appropriately for the weather (sunscreen must be worn when sunburn is a threat.

- If the pilot cannot proceed safely without navigation assistance, a spotter or lookout will be stationed safely to provide such assistance.

- The boat must always proceed at a safe, legal, speed, under control, and ready to stop within a safe distance. The safe speed of the vessel shall be determined by visibility, traffic density, turning and stopping ability of the boat, any posted speed limits, and the state of the wind, water, and current.

- Visible lights necessary for the operation of a vessel, less than 12 meters in length, between sundown and sunrise include:

    - masthead light, visible for 2 miles

    - sidelights, visible for 1 mile

    - sternlight, visible for 2 miles

    - towing light, visible for 2 miles

    - all round white, red, green, or yellow light, visible for 2 miles

- Boats less than 12 meters in length must carry some means of making an efficient audible signal.

5.2.1.2 Boat operation fundamentals:

- Except when a boat is at anchor, aground, or made fast to the shore, it is considered underway and the "Rules of the Road" apply.

- The Golden Rule for small boat handling is safety first and keep to the right. Always proceed in a steady, determined fashion. Indecisive action or actions with an obscure motive may confuse other vessels.

- Vessels that are being passed have the right-of-way over the passing vessel.

- Boats underway must stay clear of boats fishing while at anchor or while traveling.

- When two vessels are crossing each other's path, the motor vessel which has the other on the port (left) side shall have the right-of-way.

5.2.1.3 Boat/Dock Work

- Care should be taken while working from a vessel and when tying up, towing, or boarding a vessel. Charts for the area should be used to locate navigational obstacles and facilities in the area, and personnel should learn to use these aids for both day and night operations.

- NEVER allow persons to place themselves or any part of any person between vessels or a vessel and the dock when the vessel may contact the dock or other vessel.

5.2.1.4 Water operations will be governed by these minimum requirements:

- All boats will comply with Coast Guard regulations for their size and class, and carry safety gear. This safety gear should include: compass, first-aid kit, fire extinguisher, personal flotation devices (PFDs) for each person on board, radio, and required navigational lights.

- Radio equipment on all boats and vessels shall be in good working order and compatible with command communication networks.

- All boats will be outfitted with the necessary navigation equipment to assure safe transportation.

- Boats without navigational aids (radar) will not travel at night or in a fog.

- Rules of the Road as described in U.S.C.G. water safety (Appendix E) will be the operating criteria for operators of skiffs, Zodiacs and other small work boats. Supervisors are responsible for assuring that all small boat operators understand these rules.

- When personnel are going from a boat to another vessel, boat to shore, or working where there is a danger of falling into the water, they will wear U.S.C.G. Type I, II, or III PFDs, Type V suits, or jackets.

- Handle anchors and anchor ropes carefully. A common accident type is catching one's hand between boat sides and anchor rope.

- Extreme care is needed when beaching the boat due to strong underwater currents and rocks.

- To assure safe boat operations, personnel will be instructed **not to**:

    - Stand up or move around in small boats while they are underway.

    - Overload the boat or distribute loads unevenly.

    - Decelerate suddenly, allowing the stern wake to overtake and swamp the boat by washing over the transom.

- To assure safe boat operations, personnel will be instructed **to**:

    - Sit down, hold onto boat while underway.

    - Wear life vest from boat to boat, boat to shore.

- Handling gasoline for small outboard motors:

    - Always fuel boat in good light and ventilation. Fill all portable tanks on the dock, **not** while in the boat.

    - When boat is tied up at a fueling dock:

        - Don't smoke, light matches or lighters, or operate electrical switches.

        - Stop engines, motors, fans--anything that might cause a spark.

        - Extinguish all flames. Remember pilot lights on gas stoves and refrigerators.

    - Before you start to fuel:
        - Check the mooring of your boat and get everybody out of your boat.
        - Close ports, windows, doors and hatches (keep fumes and vapors off the boat).
        - Check your tanks, filler pipes, tank vents and flame screens.
        - Check to see how much fuel the tank will take.
    - During fueling:
        - Keep the nozzle of the hose in contact with the can opening to prevent static sparks.
        - Guard against spillage. If fuel spills, wipe it up immediately. Don't let vapor get below deck.
    - After fueling:
        - Replace caps (covers) or fill openings.
        - Open up the boat **completely** and ventilate.
        - Air out the boat for **five** minutes.
        - Give low spots (engine bilge, tank spaces) the sniff test. If you smell gasoline vapor--look for spillage and leaks.
        - Wipe up all spills. [Dispose of wipe-up rags properly.]
    - Fueling is an important operation which should be done by the skipper.

- A winch will not be available due to the small boat size. Therefore, spools of cable may be present on deck. Employees should be aware of the trip hazard presented, and especially the potential for entanglement in cable being unreeled over the side of the boat.
- Rescue Actions for Person Overboard
    - Basic principle and method of recovery:
        - Swing the stern away from the person with full rudder. If possible, stop the shaft before the person reaches the propeller.
        - Always assign someone to keep the person in the water in sight and do nothing else.
        - In addition to using the boat as a means of recovery, swimmers with PFD's or exposure suit and tending lines should be ready to go into the water.

(Adapted from "Communication, Aerial Surveillance and Boating Safety in Spill Response - Section IV. Safe Boat Handling" presented at the AIHC&E, Boston, MA by Ruth McIntyre-Birkner, May 31, 1992; Vessel Safety

Manual, North Pacific Fishing Vessel Owner's Association, produced in cooperation with the U.S. Coast Guard, 1986; and 33 CFR - Navigation and Navigable Waters, Subchapter D-International Navigation Rules).

5.2.1.5 Float Plan

The float plan and diving plan will address possible emergencies which may occur, including storms, drowning, onboard fires and injuries, and other potential emergencies.

The decision of whether or not an incident poses a real or potential hazard to human health or the environment is to be made by the Site Manager or SSO.

The decision to return to shore, or a specific save haven, is to be made by the Site Manager or SSO after his or her assessment of the situation, except that the captain has authority in matters of boating and seamanship.

5.2.1.6 Incident Definition Criteria

An incident is defined as:

- An onboard fire
- An injured, unaccounted-for, or unconscious employee
- A storm or high seas
- Any threat to the safety of employees

Prior to commencement of the project work, the site manager or SSO shall establish emergency procedures, safe havens, and procedures for accounting for all personnel.

5.2.1.7 Emergency Procedures

The SSO/Site Manager will coordinate activities in an emergency situation (except that the captain will have authority in matters of boating or seamanship). Provisions for transportation of injured personnel to an emergency treatment center will be in place prior to starting on-site work. The SSO shall ensure that all personnel working at the site know how to communicate with the appropriate local emergency response units, and provide adequate and clear directions between the ENSR work site and the location of those units, prior to commencing any on-site operations. **A list of emergency response contacts and telephone numbers is included in Hazard Assessment located at the beginning of this Plan.** A copy of this information must be posted in a conspicuous location on-site before operations commence.

5.3 Cuts and Lacerations

Geoprobe soil samples are collected in acetate liners that must be cut open in order to collect the sample. Additionally, tubing will need to be cut to facilitate groundwater sampling. Tube-cutters are available and should be used to eliminate this hazard. However, if it is necessary to use knives or blades, follow the safety precautions listed below:

- Keep your free hand out of the way
- Secure the acetate liner so it won't roll or move while you are cutting
- Use only sharp blades; dull blades require more force which results in less knife control
- Pull the knife at an angle to your body; pulling motions are easier to manage
- Don't put your knife in your pocket

- Use a hooked knife (i.e. linoleum knife) or a utility knife with a self-retracting blade
- Wear leather or Kevlar® gloves when using knives or blades.

5.4 Driving Safety

Drivers must be licensed to drive the class of vehicle they are operating and trained in defensive driving. Only ENSR personnel may drive ENSR vehicles or vehicles rented for ENSR business; client, subcontractor, or other work-related personnel may ride. Drivers and passengers must comply with all traffic laws and posted signs, and will not operate a vehicle if under the influence of impairing medication, alcohol, or any other substance.

Planning / Preparation

- Prior to departure, check traffic reports, weather conditions, road construction, and road closures. If necessary, develop an alternate route and new, approved JMP (Journey Management Plan).
- Prior to entering the vehicle, inspect the vehicle.
- Leave early to allow for contingencies.

DOT

If you are to operate a vehicle exceeding 10,000 pounds (or vehicle and trailer with a combined weight over 10,000 pounds), or you are to transport greater than 1,000 pounds of hazardous materials, you MUST comply with DOT regulations. These are NOT addressed in this HASP; contact the H&S Department if this applies.

Distractions

You must NOT operate a vehicle while talking on your cell phone, regardless of "hands free" or not. If you receive a call, pull over to answer it. Do NOT allow other distractions to interfere with your safe operation of the vehicle.

Secure Packing

Do not move your vehicle unless all equipment and supplies are secured. Items and material which may roll, slide, or move about in your vehicle while traveling are a major hazard. Secure the load!

Emergency Procedures:

Always move out of traffic if possible; even if those in front of you have stopped. Stopping on an active highway can precipitate being hit from the rear. If you must stop on an active roadway, leave at least one car length in front of you, and watch the rear mirror, so you can ease up if someone behind can't stop. Keep your flashers on in this situation. If you are the only driver coming to a stop on an active roadway, leave the flashers on and when safe to do so, exit the car and get to a safe location.

If you must stop due to vehicle failure, etc. try to coast out of traffic. Put on your flashers, and tie a white handkerchief, etc. on the driver's side door or mirror. If you remain in the vehicle, lock the doors. Use your cell phone to summon help.

5.5 Hand Safety

5.5.1 Glove Selection

To protect onsite workers from hand injuries, the following gloves will be used for when performing a specific duty:

Brightly colored gloves will be used to help emphasize and easily locate the hands. The color of gloves will be changed monthly to draw attention to the hands.

Pinch points are found between a moving object and a stationary object, or between two continuously moving objects. Yellow hand stickers will be placed on equipment to remind workers of pinch points.

5.5.2 Hand Tools

Rules for the safe use of hand tools:

- Select the right size tool for the job. Don't use "cheaters" and avoid pulling old tools from the waste stream. There's a reason why they were thrown away!
- All hand tools must be in safe condition.
- Handles must be sound, straight and tight-fitting.
- Always inspect tools before use and replace or repair worn or damaged tools.
- Always keep the cutting edges sharp and never test a cutting edge with your finger.
- When working on an elevated surface (ladder, truck, scaffold), ensure your tools are secure. Falling tools can cause serious injury.
- Always carry your tools correctly and never put sharp or pointed tools in your pocket.
- When carrying hand tools, always point the cutting edge to the ground.
- Always keep your tools in a dry place to prevent rust.
- Cutting tools must be kept sharp and properly shaped.
- Secure work pieces prior to cutting or drilling.
- Keep the unused hand and other people away from the tool.

Use the following types of tools while working:

Screwdrivers:

Most screwdrivers are not designed to be used on electrical equipment. Use an insulated screwdriver for electrical work.

Do not hold an object in the palm of one hand and press a screwdriver into it; place the object on a bench or table. Never hammer with a screwdriver. Never use a screwdriver with a broken handle, bent or burred blade, etc.

Pliers:

Do not use pliers as a substitute for hammers, wrenches, pry bars, etc. Use insulated pliers when doing electrical work. Inspect the pliers frequently to make certain that they are free of breaks or cracks.

Use the right type of pliers for the specific task – adjustable, locking (Vise Grip®), standard, bolt size fit, pipe wrench.

Hammers:

Use the correct hammer for the specific type of striking work (task) to be done. Always wear safety glasses when using a hammer to strike an object. Always use the claw portion of a hammer to remove nails and not as

a pick or awl. Have an unobstructed view and swing when using a hammer. Watch for overhead interference on back and forward swing. Use a good grip and use something other then your hand to hold a nail when starting hammering. Check for defects on the handle and head before using. If the hammer head shows signs of mushrooming, replace it immediately.

Handles may be wood, tubular/solid steel or fiberglass. Replace any hammer with a loose handle before the head flies off and causes injury to you or someone else. Tighten loose handles with the proper wedges; never use nails or staples for wedges. If a steel or fiberglass handle is loose replace it, since it is more difficult to repair than a wooden one. Some fiberglass handles can be tightened with the aid of a repair kit with epoxy materials.

Wrenches:

Select the correct size of wrench for the job. Never use a pipe wrench as a wrench handle extension. Too much leverage can ruin a tool and cause injury.

To avoid sudden slips, stand in a balanced position and always pull on the wrench instead of pushing against the fixed jaw, particularly when a pinch point is created. Wear gloves when using a wrench in a confined space.

Whenever possible use a box end wrench instead of an open end wrench to avoid slipping.

Chisels:

Always wear safety goggles or a face shield when using a chisel. Drive chisels outward and away from your body. Do not use chisels to pry. Keep edges sharp for most effective work and protect when not in use. Driven tools (chisels, punches, etc.) must be dressed to remove any mushrooming. Use the proper hammer when using a chisel.

Knives:

Always perform a through Job Safety Analysis (JSA) to define the proper cutting tool for the task.

Always place the item to be cut on a solid surface, attempt to hold the cut item without your hand and cut in a direction away from the body and hand.

Always keep hands and body clear of the knife stroke. Always keep the cutting tool blades sharp.

Make sure there is plenty of open space around you when using any cutting tool.

Use the following safer tools in replace of FOBK:

- Self-retracting utility knives
- Guarded utility knives
- Shears, snips, and/or scissors
- Concealed blade cutters
- Pipe cutters
- Specialty cutters (e.g. Geoprobe Acetate Liner Cutter)
- Ratcheting tools

5.6 Insects and Spiders

Spiders and wasps may be found in derelict buildings, sheltered areas, and even on open ground. Exercise care when collecting samples and avoid reaching into areas where visibility is limited. If stung by a wasp or bee, or bitten by a spider, notify a co-worker or someone who can help if you should you have an allergic reaction. Stay calm and treat the area with ice or cold water. Seek medical attention if you have any reactions to the sting such as developing a rash, excessive swelling or pain at the site of the bite or sting or any swelling or numbness beyond the site of the bite or sting.

Black Widow Spider



Abdomen usually shows hourglass marking.

The female is 3-4 centimeters in diameter.

Have been found in well casings and flush-mount covers.

Not aggressive, but more likely to bite if guarding eggs.

Light, local swelling and reddening of the bite are early signs of a bite, followed by intense muscular pain, rigidity of the abdomen and legs, difficulty breathing, and nausea.

If bitten, see physician as soon as possible.

Brown Spiders (Recluse)



Central and South U.S., although in some other areas, as well.

¼-to-½-inch-long body, and size of silver dollar.

Hide in baseboards, ceiling cracks, and undisturbed piles of material.

Bite either may go unnoticed or may be followed by a severe localized reaction, including scabbing, necrosis of affected tissue, and very slow healing.

If bitten, see physician as soon as possible.

Studies have determined that repellants containing DEET as a main ingredient are most effective against spiders, mosquitoes, and other insects. DEET can be directly applied to the exposed skin of adults and/or clothing. Permanone® is another repellent however, it can only be directly applied to clothing.

5.7 Noise Exposure

The use of drilling equipment can expose the field team to noise levels that exceed the OSHA PEL of 90 dB for an 8-hour day. Exposure to noise can result in the following:

- Temporary hearing losses where normal hearing returns after a rest period;
- Interference with speech communication and the perception of auditory signals;
- Interference with the performance of complicated tasks; and,
- Permanent hearing loss due to repeated exposure resulting in nerve destruction in the hearing organ.

Since personal noise monitoring will not be conducted during the proposed activities, employees must follow this general rule of thumb: If the noise levels are such that you must shout at someone 5 feet away from you, you need to be wearing hearing protection. Employees can wear either disposable earplugs or earmuffs but all hearing protection must have a minimum noise reduction rating (NRR) of 27 dB.

5.8 Poisonous Plants

All undeveloped property potentially has poison ivy, oak, or sumac growing in areas where vegetation is not controlled. These plants can also be found in cultivated and landscaped areas. Use common sense when

Poison Ivy Grows in West, Midwest, Texas, East. Several forms – vine, trailing shrub, or shrub. Three leaflets (can vary 3-9). Leaves green in summer, red in fall. Yellow or green flowers. White berries.	 
Poison Oak Grown in the East (NJ to Texas), Pacific Coast. 6-foot tall shrubs or long vines. Oak-like leaves, clusters of three. Yellow berries.	 
Poison Sumac Grows in boggy areas, especially in the Southwest and Northern states. Shrub up to 15 feet tall. Seven to 13 smooth-edged leaflets. Glossy pale yellow or cream-colored berries.	 

If you must enter areas containing such plants, wear protective clothing, such as Tyvek® coveralls, Nitrile or latex gloves, and boot covers. The use of a barrier cream such as Ivy Block can prevent the active agent in poisonous plants from affecting skin and Tecnu cleansing wipes can remove the plant oil from exposed skin.

Avoid using mowers and weed trimmers in areas where poison ivy and oak are likely. Additional care should be taken during early winter after the leaves have fallen from the poisonous plants; the poison still exists in the vines and stubble remaining above the ground. Wash any contaminated skin immediately with cold water and mild soap.

5.9 Slips, trips and fall hazards

On any work area, it is expected that the ground might be uneven. The ground surface might be unreliable due to settling. Surface debris might be present and wet or swampy areas can exist.

Employees should walk around, not over or on top of debris or trash piles. When carrying equipment, identify a path that is clear of any obstructions. It might be necessary to remove obstacles to create a smooth, unobstructed access point to the work areas on site.

During the winter months, snow shovels and salt crystals or calcium chloride should be kept on site to keep work areas free of accumulated snow and ice. Furthermore, use sand or other aggregate material to help keep work surfaces from being slippery, especially where salt/calcium chloride cannot be used. In addition, make sure work boots have soles that provide good traction. When walking on ice is necessary crampons or Yaktrax® should be used.

Maintaining a work environment that is free from accumulated debris is the key to preventing slip, trip and fall hazards at construction sites. Essential elements of good housekeeping include

- Orderly placement of materials, tools and equipment;
- Placing trash receptacles at appropriate locations for the disposal of miscellaneous rubbish;
- Prompt removal and secure storage of items that are not needed to perform the immediate task at hand; and,
- Awareness on the part of all employees to walk around, not over or on, equipment that might have been stored in the work area.

5.10 Traffic Safety

Basic Procedures

To make certain that motorists are aware of our presence, all employees who are potentially exposed to traffic hazards should **wear orange or yellow ANSI Class II or III safety vests**. Work area should be delineated with traffic cones, or other suitable warning barriers, to prevent motorists from inadvertently driving through. As for vests, cones or other barrier materials should be reflectorized if work will be performed during dusk or evening hours. Where it is not feasible to implement such procedures, a standby observer should be assigned to warn the work crew of any impending traffic hazards.

Work On/Adjacent to Public Roadways

For projects that involve potential exposure to traffic on or adjacent to public roadways, consult the "**Work Zone Traffic Control" handbook**, under "Traffic Control" on ENSR's H&S Website, at the following web address: http://intranet.ensr.com/healthweb

The handbook was developed by the State of Maine DOT and provides examples of traffic control applications for typical road work situations (e.g., closure of one lane of a two lane road, stationary work on the shoulder of a road, mobile work along the shoulder of a road, etc.). Although it was written to reflect the basic requirements of Part VI of the Federal Highway Administration's (FHWA) Manual of Uniform Traffic Control Devices (MUTCD), this handbook is not a regulatory document. Since specific requirements will vary from state to state, and within a state, by county, city or town;

Flagging/Redirecting Traffic

Specific requirements exist when traffic must be redirected around a work area that is on or adjacent to a public roadway. In certain locations only police officers may redirect traffic. As a minimum, OSHA requires that flaggers be formally trained in accordance with the requirements specified in ANSI D6.1-1971. As a result, ENSR personnel should not redirect traffic on public roadways.

http://www.atssa.com/cs/flagger

- Texas Flagging Requirements:
- Certification required every three years;
- Hardhat required. DOT must wear white. Contractors wear any color;
- Reflectorized safety vests required. and

All training courses handled by ATSSA and Texas Engineering Extension Services.

When traffic must be redirected, and the local police do not perform that role, a traffic control firm should be hired (these are frequently listed in the yellow pages under "safety").

5.11 Water Hazards

While on a barge or boat or any equipment working over water greater than three feet deep, Coast Guard approved Type III personal floatation devices must be worn or Type-V suits, or jackets. Vests will also be worn when moving from boat to barge or boat to shore.

A throwable flotation device with tether will be in the boat or on shore when ever ENSR or ENSR subcontractors are working in or over water.

ENSR employees will not enter standing or flowing water deeper than three feet unless they are tethered to a stationary object located at lest ten feet from the edge of the water. In all cases employees entering surface water will wear personal flotation devices and a second person will be stationed on the shore to assist during emergencies. Do not enter water unless you know the maximum depth in the area you are working.

Note that the water can contain bacteria and fungal organisms that can cause infections on the skin and internally. Employees should avoid direct contact with the. If splashing water is produced during the operations face shields, chest waders, and/or aprons should be considered.

When crossing shallow creeks it is recommended that workers use a walking stick to stabilize themselves and to assist others in crossing the creek.

5.12 Weather

5.12.1 Inclement Weather

The Site Safety Officer will check the weather forecast for the project area each morning prior to mobilization. Predicted weather conditions will be included in the Job Safety Analysis. Weather changes should initiate a review and update of the JSA as necessary.

Severe weather can occur with little warning. The employee must be aware of the potentials for lightning, flash flooding and high wind events.

Be Prepared, Know What is Coming your Way

- Listen to the radio for severe weather alerts.

- Check the Storm Prediction Center's web page for alerts and warnings.

  http://www.spc.noaa.gov/products/wwa/

- Pay attention to the weather in your area, up wind of your location, and in the watershed up stream from your location.
- When in the field, be aware of the route you must take to get to shelter.
- When working in low areas be aware of the potential for flash flooding and the route to higher ground.

5.12.2 Heat Stress

Types of Heat Stress

Heat related problems include **heat rash, fainting, heat cramps, heat exhaustion and heat stroke**. **Heat rash** can occur when sweat isn't allowed to evaporate; leaving the skin wet most of the time and making it subject to irritation. **Fainting** may occur when blood pools to lower parts of the body and as a result, does not return to the heart to be pumped to the brain. Heat related fainting often occurs during activities that require standing erect and immobile in the heat for long periods of time. **Heat cramps** are painful spasms of the muscles due to excessive salt loss associated with profuse sweating.

Heat exhaustion results from the loss of large amounts of fluid and excessive loss of salt from profuse sweating. The skin will be clammy and moist and the affected individual may exhibit giddiness, nausea and headache.

Heat stroke occurs when the body's temperature regulatory system has failed. The skin is hot, dry, red and spotted. The affected person may be mentally confused and delirious. Convulsions could occur. EARLY RECOGNITION AND TREATMENT OF HEAT STROKE ARE THE ONLY MEANS OF PREVENTING BRAIN DAMAGE OR DEATH. A person exhibiting signs of heat stroke should be removed from the work area to a shaded area. The person should be soaked with water to promote evaporation. Fan the person's body to increase cooling.

Increased body temperature and physical discomfort also promote irritability and a decreased attention to the performance of hazardous tasks.

Early Symptoms of Heat-Related Health Problems:

decline in task performance	excessive fatigue
incoordination	reduced vigilance
decline in alertness	muscle cramps
unsteady walk	dizziness

Susceptibility to Heat Stress Increases due to:

lack of physical fitness	obesity
lack of acclimatization	drug or alcohol use
increased age	sunburn
dehydration	infection

People unaccustomed to heat are particularly susceptible to heat fatigue. First timers in PPE need to gradually adjust to the heat.

The Effect of Personal Protective Equipment

Sweating normally cools the body as moisture is removed from the skin by evaporation. However, the wearing of certain personal protective equipment (PPE), particularly chemical protective coveralls (e.g., Tyvek), reduces the body's ability to evaporate sweat and thereby regulate heat buildup. The body's efforts to maintain an acceptable temperature can therefore become significantly impaired by the wearing of PPE.

Measures to Avoid Heat Stress:

The following guidelines should be adhered to when working in hot environments:

- Establish work-rest cycles (short and frequent are more beneficial than long and seldom).
- Identify a shaded, cool rest area.
- Rotate personnel, alternative job functions.
- Water intake should exceed sweat produced. Most workers exposed to hot conditions drink less fluids than needed because of an insufficient thirst. **DO NOT DEPEND ON THIRST TO SIGNAL WHEN AND HOW MUCH TO DRINK**. Consume enough liquid to force urination every two hours. In humid climates ice water or ice should be consumed to help maintain normal body temperature since evaporation does not provide an efficient mechanism for heat removal.
- Eat light meals before and during work shifts. Avoid highly salted foods.
- Drink sports drinks such as Gatorade® diluted 1:1 with water.
- Save most strenuous tasks for non-peak heat hours such as the early morning or at night.
- Avoid alcohol during prolonged periods of heat. Alcohol will cause additional dehydration.
- Avoid double shifts and/or overtime.

The implementation and enforcement of the above mentioned measures will be the joint responsibility of the Project Manager and health and the Site Safety Officer. Potable water and fruit juices should be made available each day for the field team.

Heat Stress Monitoring Techniques

Site personnel should regularly monitor their heart rate as an indicator of heat strain by the following method:

Radial pulse rates should be checked by using fore-and middle fingers and applying light pressure top the pulse in the wrist for one minute at the beginning of each rest cycle. If the pulse rate exceeds 110 beats/minute, the next work cycle will be shortened by one-third and the rest period will be kept the same. If, after the next rest period, the pulse rate still exceeds 110 beats/minute, the work cycle will be shortened again by one-third.

5.12.3 Cold Stress

Type of Cold Stress

Cold injury is classified as either localized, as in frostbite, frostnip or chilblain; or generalized, as in hypothermia. The main factors contributing to cold injury are exposure to humidity and high winds, contact with wetness and inadequate clothing.

The likelihood of developing frostbite occurs when the face or extremities are exposed to a cold wind in addition to cold temperatures. The freezing point of the skin is about 30^{o} F. When fluids around the cells of the

body tissue freeze, skin turns white. This freezing is due to exposure to extremely low temperatures. As wind velocity increases, heat loss is greater and frostbite will occur more rapidly.

Symptoms of Cold Stress

The first symptom of frostbite is usually an uncomfortable sensation of coldness, followed by numbness. There might be a tingling, stinging or aching feeling in the affected area. The most vulnerable parts of the body are the nose, cheeks, ears, fingers and toes.

Symptoms of hypothermia, a condition of abnormally low body temperature, include uncontrollable shivering and sensations of cold. The heartbeat slows and can become irregular, the pulse weakens and the blood pressure changes. Pain in the extremities and severe shivering can be the first warning of dangerous exposure to cold.

Maximum severe shivering develops when the body temperature has fallen to 95^{o} F. Productive physical and mental work is limited when severe shivering occurs. Shivering is a serious sign of danger. Immediately remove any person who is shivering from the cold.

Methods to Prevent Cold Stress

When the ambient temperature, or a wind chill equivalent, falls to below 40^{o} F (American Conference of Governmental Industrial Hygienists recommendation), site personnel who must remain outdoors should wear insulated coveralls, insulated boot liners, hard hat helmet liners and insulated hand protection. Wool mittens are more efficient insulators than gloves. Keeping the head covered is very important, since 40% of body heat can be lost when the head is exposed. If it is not necessary to wear a hard hat, a wool knit cap provides the best head protection. A facemask may also be worn.

Persons should dress in several layers rather than one single heavy outer garment. The outer piece of clothing should ideally be wind and waterproof. Clothing made of thin cotton fabric or synthetic fabrics such as polypropylene is ideal since it helps to evaporate sweat. Polypropylene is best at wicking away moisture while still retaining its insulating properties. Loosely fitting clothing also aids in sweat evaporation. Denim is not a good protective fabric. It is loosely woven which allows moisture to penetrate. Socks with a high wool content are best. If two pairs of socks are worn, the inner sock should be smaller and made of cotton, polypropylene or similar types of synthetic material that wick away moisture. If clothing becomes wet, it should be taken off immediately and a dry set of clothing put on.

If wind conditions become severe, it might become necessary to shield the work area temporarily. The SSO and the PM will determine if this type of action is necessary. Heated break trailers or a designated area that is heated should be available if work is performed continuously in the cold at temperatures, or equivalent wind chill temperatures, of 20^{o} F.

Dehydration occurs in the cold environment and can increase the susceptibility of the worker to cold injury due to significant change in blood flow to the extremities. Drink plenty of fluids, but limit the intake of caffeine

5.12.4 Work/Rest Cycles for Cold Weather

If wind chill temperatures fall below **minus 25^{o} F**, breaks from the cold will occur at a rate of one every hour. If wind chill temperatures fall below **minus 45^{o} F,** all work will cease and persons will be required to go indoors. Also see Section 1.1.1 regarding shift duration. However, these guidelines can be modified at any time based on actual site conditions and professional judgment rendered by either the Field Manger and/or SSO. For example, the Field Manger and/or SSO will evaluate field crew fitness; the condition of their cold-weather gear, including boots; and will observe employees alertness, including fatigue and rate of cold tolerance/acclimation.

If weather conditions warrant, portable tents might become necessary to shield the work area from wind, rain, snow, etc. The SSO and the Field Manager will determine if this type of action is necessary. However, under no conditions will the tents be heated and as a precautionary measure, a Photoionization Detector (PID) with a 10.6 ev lamp will be used to monitor the breathing zone of personnel inside the tent. See Section 6 for action levels based on PID readings. A JSA should be prepared and discussed with all workers detailing the precautions for working in these cold weather conditions.

5.13 Confined Spaces

No confined space entries are planned or allowed on this project.

When working in industrial settings, it is common to need to enter a confined space to make observations, collect samples, or perform other duties. ENSR employees or sub contractors must not enter any confined space containing a hazard...

A confined space is defined as any space that meets the following criteria:

- Is not designed for human occupancy
  (excludes vehicles, elevator cabins etc,)
  (includes elevator shafts and wells, tanks, vaults, etc.)
- Is large enough to physically enter with the whole body, and
- Has a restricted exit path (you must climb over pipes, through man ways, etc.)

If the confined space contains any hazard, entry may only be made if permitted in writing by the space owner or the Regional Health and Safety Manger, the entry is monitored by an observer, and with the prior written approval of the ENSR Regional Health and Safety Manager.

Typical hazards include but are not limited to:

Flammable materials	Exposed electrical circuits	Oxygen deficient atmosphere
Toxic materials	Falls greater than six feet	
Corrosive materials	Moving machinery	

If there is any doubt about whether a space meets the above criteria, call the ENSR Health and Safety Staff.

6.0 Air Monitoring

6.1 Monitors

6.1.1 Photoionization Detector

As a precautionary measure, a Photoionization Detector (PID) with a 10.6 ev lamp will be used to monitor the breathing zone of personnel during the proposed activities. If the PID indicates sustained (15 minute) breathing zone vapor concentrations in excess of 25 ppm as isobutylene, respiratory protection, as described in Section 7.2 of this document, will be donned.

Task	Instrument	Action Limit and Action
All tasks involving potential exposure to contaminated sediments and water	Photoionization Detector	25 ppm as isobutylene Don respiratory protection as discussed in Section 7

6.2 Personal Air Sampling

The need for personal air sampling is not anticipated by ENSR during the activities covered by this HASP. The ENSR Project Manager, or the ENSR Regional Health and Safety Manager can prescribe personal air sampling based on observations or concerns recognized during the project.

6.3 Calibration and Recordkeeping

Equipment used by ENSR will be calibrated in accordance with ENSR's standard operating procedures. A log of the calibrations and readings will be kept in the field notebook. Daily calibration information will also be recorded in the field notebook.

7.0 Personal Protective Equipment

Personal protective equipment (PPE) will be worn during these activities to prevent on-site personnel from being injured by the safety hazards posed by the site and/or the activities being performed. In addition, chemical protective clothing will be worn to prevent direct dermal contact with the site's chemical contaminants. The following table describes the PPE and chemical protective clothing to be worn for general site activities and for certain specific tasks.

7.1 Personal Protective Equipment

PPE Item	General	Sample Collection	Sample Processing
Hard Hat	1 & 2	✓	✓
Traffic Vests	2	2	2
Steel Toed Safety Shoes	1	✓	✓
Safety Glasses with Sideshields	1	✓	✓
Goggles or Faceshield		3	3
Hearing Protection		4	4
Tyvek Coveralls		5	5
Nitrile Gloves	5	5	5
Ivy Block® or Ivy Screen® barrier cream	5 & 6	5 & 6	5 & 6
Type III Personal Floatation Device	7	7	7
Chest Waders		8	

✓ Required PPE

1 All employees must comply with United States Department of Defense safety requirements.

2 Traffic vests and hardhats are required when working within twenty feet of any public road or any private road with active traffic.

3 Goggles and/of face shields will be worn when splash hazards are present while conducting work.

4 Hearing protection should be worn around equipment if normal conversation cannot be understood.

5 Tyvek coveralls and Nitrile gloves are only required of those that are likely to come in direct contact with potentially contaminated sediments and water. Tyvek coveralls and Nitrile gloves will be worn to protect workers from poison ivy and poison oak when contact cannot be avoided.

6 Ivy Block® or Ivy Screen® barrier cream should be worn on exposed skin where there is a potential for exposure to poison ivy or oak.

7 Personal Floatation Devices are required when work is conducted in water three feet or greater in depth.

8 If sample collection involves entering shallow waters, employees will wear chest waders to avoid contact with potentially contaminated sediments and water.

7.2 Respiratory Protection

Although not likely, respiratory protection as described below will be required if worker breathing zone PID concentrations are sustained above the action levels in the following table.

Task	Action Limit	Respiratory Protection	Level
All tasks involving potential exposure to contaminated soils and/or groundwater	25 ppm as Isobutylene for 1 minute	Half or full face mask respirator with combination organic vapor/HEPA cartridges	C
	50 ppm as Isobutylene	Full face respirator with organic vapor/HEPA cartridges	C
	100 ppm as isobutylene	Supplied air respirator	B

Respiratory protection (half or full face mask respirator with combination organic vapor/HEPA cartridges) should also be donned if odors become objectionable at any time or if respiratory tract irritation is noticed.

All employees who are expected to don respiratory protection must have successfully passed a qualitative or quantitative fit-test within the past year for the brand, model and size respirator they plan to don.

If worn, respirators will be cleaned after each use with respirator wipe pads and will be stored in plastic bags after cleaning. Respirators will be thoroughly cleaned using disinfectant material within one week following any respirator use. Refer to the cleaning instructions provided with the respirator or specified by Appendix B-2 to the OSHA regulations at 29 CFR 1910.134.

7.3 Other Safety Equipment

The following additional safety items will be available at the site:

- Throwable flotation device
- Portable, hand-held eyewash bottles
- First aid kit
- Type A-B-C fire extinguisher
- Portable phones

8.0 Site Control

To prevent both exposure of unprotected personnel and migration of contamination due to tracking by personnel or equipment, hazardous work areas will be clearly identified and decontamination procedures will be required for personnel and equipment leaving those areas.

8.1 Designation of Zones

ENSR designates work areas or zones as suggested in the "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities," NIOSH/OSHA/USCG/EPA, November 1985. They recommend that the areas surrounding each of the work areas to be divided into three zones:

- Exclusion or "Hot" Zone
- Contamination Reduction Zone
- Support Zone

8.1.1 Exclusion Zone

An exclusion zone will be established around the drilling areas. The perimeter of the exclusion zone will be marked with caution tape or indicated by traffic cones so that employees, visitors, and client or host employer personnel are aware of the work being conducted.

All ENSR and contractor personnel entering these work areas must wear the prescribed level of protective equipment.

8.1.2 Contamination Reduction Zone

A decontamination zone will be established adjacent to each work area. Personnel will remove contaminated gloves and other disposable items in this area and place them in a plastic bag until they can be properly disposed of.

8.1.3 Support Zone

At this site the support zone will include the area outside of the exclusion zone.

8.1.4 Site Access Control

The public will be restricted from the project site and monitoring well locations (during monitoring) by fences, barricade tape, traffic cones, and/or signs.

8.1.5 Parking and Staging Areas

Parking will be restricted to areas that have been cleared of tall grass and combustible material. Vehicles parked on the public streets will be marked with cones both in front of and behind the vehicle.

8.1.6 Pedestrian Walkways

Pathways within the work areas will be kept clear of obstructions. Public pathways will be clearly marked to provide access to the business onsite and protect the public from the hazards of the project.

8.2 General Site Safety Practices

The following measures are designed to augment the specific health and safety guidelines provided in this plan.

- The "buddy system" will be used at all times by all field personnel. No one is to perform field work alone. Standby team member must be intimately familiar with the procedures for initiating an emergency response.
- Eating, drinking, chewing gum or tobacco, smoking or any practice that increases the probability of hand-to-mouth transfer and ingestion of materials is prohibited in the immediate work area and the decontamination zone.
- Eating, chewing gum or tobacco, smoking or any practice that increases the probability of hand-to-mouth transfer and ingestion of materials is prohibited in the immediate work area and the decontamination zone. Water and Ice may be consumed in all areas to prevent heat stress but precautions must be taken to prevent contamination of the water and ice.
- Smoking is prohibited in all work areas. Matches and lighters are not allowed in these areas.
- Hands and face must be thoroughly washed upon leaving the work area and before eating, drinking or any other activities.
- Beards or other facial hair that interfere with respirator fit are prohibited.
- The use of alcohol or illicit drugs is prohibited during the conduct of field operations.
- All equipment must be decontaminated or properly discarded before leaving the site in accordance with the project work plan.
- Parking and pedestrian areas will be established and communicated to all workers.

9.0 Decontamination

9.1 Personal Decontamination

Proper decontamination is required of all personnel before leaving the site. Decontamination will occur within the contamination reduction zone.

Regardless of the type of decontamination system required, a container of potable water and liquid soap should be made available so employees can wash their hands and face before leaving the site for lunch or for the day.

9.2 PPE Decontamination

Disposable PPE, such as Tyvek coveralls, gloves, etc. will be removed in the decon zone and placed in garbage bags. Final disposal of contaminated PPE will be in accordance with the work plan.

If worn, respirators will be cleaned after each use with respirator wipe pads and will be stored upright in plastic bags. Respirators will be thoroughly cleaned using disinfectant material within one week following any respirator use. Refer to the cleaning instructions provided with the respirator or specified by Appendix B-2 to the OSHA regulations at 29 CFR 1910.134.

9.3 Equipment Decontamination

Equipment will be decontaminated prior to being moved to other locations. Decontamination procedures will be specified by the Project Manager.

10.0 Medical Monitoring and Training Requirements

Each worker subject to this HASP shall have copies of documentation that the requirements for training, medical surveillance, and respirator use are current. Copies of these documents shall be made available to ENSR or ENSR's client upon request.

10.1 Medical Monitoring

All personnel performing activities covered by this HASP must be active participants in a medical monitoring program that complies with 29 CFR 1910.120(f). Each individual must have completed an annual surveillance examination and/or an initial baseline examination within the last year prior to performing any work on the site covered by this HASP.

10.2 Health and Safety Training

10.2.1 HAZWOPER

All personnel performing activities covered by this HASP must have completed the appropriate training requirements specified in 29 CFR 1910.120 (e). Each individual must have completed an annual 8-hour refresher training course and/or initial 40-hour training course within the last year prior to performing any work on the sites covered by this HASP.

10.2.2 Pre-Entry Briefing

Prior to the commencement of on-site activities, a pre-entry briefing will be conducted by the SSO to review the specific requirements of this HASP. Attendance of the pre-entry meeting is mandatory for all personnel covered by this HASP and must be documented on the attendance form provided in Attachment C. HASP sign-off sheets should also be collected at the time of the pre-entry briefing. All documentation should be maintained in the project file.

The pre-entry briefing must be completed for each new employee before they begin work at the site. Short safety refresher meetings will be conducted, as needed, throughout the duration of the project.

11.0 Emergency Response

OSHA defines emergency response as any "response effort by employees from outside the immediate release area or by other designated responders (i.e., mutual-aid groups, local fire departments, etc.) to an occurrence which results, or is likely to result in an uncontrolled release of a hazardous substance." According to ENSR policy, ENSR personnel shall not participate in any emergency response where there are potential safety or health hazards (i.e., fire, explosion, or chemical exposure). ENSR response actions will be limited to evacuation and medical/first aid as described within this section below. As such this section is written to comply with the requirements of 29 CFR 1910.38 (a).

The basic elements of an emergency evacuation plan include:

- employee training,
- alarm systems,
- escape routes,
- escape procedures,
- critical operations or equipment,
- rescue and medical duty assignments,
- designation of responsible parties,
- emergency reporting procedures and
- methods to account for all employees after evacuation.

11.1 Employee Training

Employees must be instructed in the site-specific aspects of emergency evacuation. On-site refresher or update training is required anytime escape routes or procedures are modified or personnel assignments are changed.

11.2 Alarm System/Emergency Signals

An emergency communication system must be in effect at all sites. The simplest and most and effective emergency communication system in many situations will be direct verbal communications. Each site must be assessed at the time of initial site activity and periodically as the work progresses. Verbal communications must be supplemented anytime voices can not be clearly perceived above ambient noise levels (i.e., noise from heavy equipment; drilling rigs, backhoes, etc.) and anytime a clear line-of-sight can not be easily maintained amongst all ENSR personnel because of distance, terrain or other obstructions.

Verbal communications will be adequate to warn employees of hazards associated with the immediate work area. The property is occupied but ENSR may not have access to facility phones. Therefore, ENSR will bring a portable phone to the site to ensure that communications with local emergency responders is maintained, when necessary.

11.3 Escape Routes and Procedures

The escape route from the site and an emergency muster point will be determined and provided to all workers during the project mobilization.

Prior to mobilizing to a new project area, the Site Safety Officer or his designee will confirm that the escape routes are clear and lead to a safe area.

11.4 Employee Accounting Method

The SSO is responsible for identifying all ENSR personnel on-site at all times. ENSR and its subcontract employees will notify the SSO when they enter and leave the site. The SSO will account for all ENSR and its subcontract employees following an evacuation.

11.5 Injuries and Illnesses

The phone numbers of the police and fire departments, ambulance service, local hospital, and ENSR representatives are provided in the emergency reference sheet on page 1. This sheet will be posted in the site vehicle.

11.5.1 First Aid

Minor injuries will be treated on site using materials from the first aid kit or other local sources. All cuts and abrasions will be cleaned with potable water and a clean dressing applied. The injured employee will be evaluated at the end of the work day and the following day when the employee arrives at the project site to determine whether the wound has started the healing process. The wound will be protected from contamination during the project activities.

11.5.2 Professional Treatment

In the event an injury or illness requires more than first aid treatment, the SSO will accompany the injured person to the medical facility and will remain with the person until release or admittance is determined. The escort will relay all appropriate medical information to the on-site project manager and the RHSM.

If the injured employee can be moved from the accident area, he or she will be brought to the CRZ where their PPE will be removed. If the person is suffering from a back or neck injury the person will not be moved and the requirements for decontamination do not apply. The SSO must familiarize the responding emergency personnel about the nature of the site and the injury. If the responder feels that the PPE can be cut away from the injured person's body, this will be done on-site. If this not feasible, decontamination will be performed after the injured person has been stabilized.

11.6 Designation of responsible parties

The SSO is responsible for initiating emergency response. In the event the SSO can not fulfill this duty, the alternate SSO will take charge.

11.7 Emergency Response Drills

For projects with durations of greater than four days on site, the SSO will initiate an evacuation drill during the first five days and shall repeat the drills at least quarterly. Deficiencies noted during the drills will be documented as a Near Loss, a Root Cause Analysis conducted and corrective actions initiated.

Emergency Response drills and subsequent personnel briefings on evacuation procedures will be documented in the safety briefing agenda or briefing notes.

11.8 Incident Reporting and Investigation

Any incident (other than minor first aid treatment) resulting in injury, illness or property damage requires an Incident investigation and report. The investigation should be conducted as soon as emergency conditions are under control. The purpose of the investigation is not to attribute blame but to determine the pertinent facts so that repeat or similar occurrences can be avoided. An ENSR Incident investigation form is presented in Attachment D of this HASP. The injured ENSR employee's supervisor, the ENSR Project Manager, and the RHSM should be notified immediately of the injury.

If a subcontractor employee is injured, they are required to notify the ENSR SSO. Once the incident is under control, the subcontractor will submit a copy of their company's Incident investigation report to the ENSR SSO.

Attachment A - Health and Safety Plan Review Form

Health and Safety Plan Acceptance Form

Sediment Sampling and Site Investigation
Washington Navy Yard
Anacostia River
Washington, D.C.

I have reviewed a copy of the Health and Safety Plan prepared for the above-referenced site and activities. I have read and understood its contents and I agree that I will abide by its requirements.

Name: _____

Signature: _____

Date: _____

Representing: _____

Attachment B - Job Safety Analysis

Job Safety Analysis

ENSR | AECOM

JSA Type: ☒ Investigation ☐ O&M ☐Office ☐ Construction	☒ New ☐ Revised	**Date**: 9/19/07

Work Activity: Sediment Sampling in the Anacostia River and sediment processing on shore.

Personal Protective Equipment (PPE): Minimum PPE is Level D including: hard hat, safety glasses, steel-toed boots, long-sleeved shirts, high visibility safety vest, hearing protection as needed, and gloves as needed (type dependent on job-specific requirements). Hip Waders, Life Vests, USGS approved personnel floatation devices.

Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
Global Site Work Safety (applies to all activities) and **1.) Arrive on site** **2.) Use GPS to verify coordinate accuracy** **3.) Check water depth using staff gauges**	• Drowning **(See HASP Sections 5.2 and 5.11)**	• Use life vest when within 10 feet of water (as needed). • Have life preservers or flotation devices available for supervising personnel, instrument operator, person in the river, and sentinels. • Personnel in river will be required to wear personal flotation devices (USCG approved PFDs), if in "belly boats". • When a boat is used for sampling, the personnel in the boat will use USCG approved PFDs. In addition, the boat and engine should be inspected prior to use to ensure that safety equipment (i.e. fire extinguisher etc.) is in good condition. • Safe wading depth will be considered to be 3.0 feet. Additionally, the safe wading depth will be adjusted (decreased) based upon the depth, location and flow/force of the river. • Be aware of river conditions; leave the area if flash flooding or inclement weather conditions present hazards to personnel. • Use buddy system, have sentinel(s) to watch person in river. • Ensure that personnel working on water do not have a fear of water and that they can swim. • Avoid entanglement in the ropes, cables and other anchoring systems that are used to anchor the booms in the river. • Use radios to maintain contact with supervisor and safety officer. • In times of heavy rain and/or high water within the river, personnel will not be allowed along the banks of the river without a safety line and/or a floatation device.	• flash flooding or inclement weather conditions present hazards to personnel • PFDs are not available to everyone on boat • boat or engine is found to be unsafe and/or safety equipment is missing such as a fire extinguisher

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Job Hazard Analysis

ENSR

JSA Type: ☒ Investigation ☐ O&M ☐ Office ☐ Construction	☒ New ☐ Revised	Date: 9/19/07

Work Activity: Sediment Sampling in the Anacostia River and sediment processing on shore.

Personal Protective Equipment (PPE): Minimum PPE is Level D including: hard hat, safety glasses, steel-toed boots, long-sleeved shirts, high visibility safety vest, hearing protection as needed, and gloves as needed (type dependent on job-specific requirements). Hip Waders, Life Vests, USGS approved personnel floatation devices.

Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
(Continued) Global Site Work Safety (applies to all activities) and **1.) Arrive on site** **2.) Use GPS to verify coordinate accuracy** **3.) Check water depth using staff gauges**	• Hand injury **(See HASP Section 5.5)**	• Wear chemically protective gloves when in contact with contaminated material. • Use shears rather than utility knives to cut tubing or other materials. • Wear leather or heat-resistant gloves while performing manual work. • Avoid touching hot surfaces without proper protective equipment. • Identify and avoid pinch points • Do not grasp steel hoist cables during hoisting. • Use only appropriate tools for the task.	• If major injury occurs or proper PPE not available
	• Slip, Trip, and Fall on same level (FS) or to lower level (FL) **(See HASP Section 5.9)**	• Maintain a clean work area and good housekeeping practices by drying wet surfaces, cleaning up muddy areas, and keeping unnecessary equipment and supplies out of walkways. • Inspect tread on steel-toed boots for signs of wear and replace as necessary • When carrying field equipment maintain clear view of footing • Wear steel-toed boots that extend over the ankle • Never run while on the job site • Use backpacks for moving gear around the site to keep hands free. • Be aware of slippery conditions. • No work after dusk.	• If major injury occurs or unable to perform work safely
	• Lifting – Back and Foot injury **(See HASP Section 5.1)**	• Follow standard safe lifting practices. • Wear proper PPE, i.e., steel-toed shoes with metatarsals. • Use Mechanical lifting devices when ever possible. • Ensure path is clear prior to lifting and moving materials. • Ensure proper rigging.	• Do not attempt lift if the load is too heavy or worker is not capable
(Continued) Global Site Work Safety (applies to all activities) and **1.) Arrive on site** **2.) Use GPS to verify coordinate**	Damaged Tools or Improper Use of Tools • Injury • Property damage **(See HASP Section 5.5.2)**	• Training of personnel. • Inspection and maintenance of equipment. • Use of proper tools for the work being performed.	• If major injury occurs or unable to perform work safely

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Job Hazard Analysis

ENSR

JSA Type: ☒ Investigation ☐ O&M ☐Office ☐ Construction	☒ New ☐ Revised	**Date**: 9/19/07

Work Activity: Sediment Sampling in the Anacostia River and sediment processing on shore.

Personal Protective Equipment (PPE): Minimum PPE is Level D including: hard hat, safety glasses, steel-toed boots, long-sleeved shirts, high visibility safety vest, hearing protection as needed, and gloves as needed (type dependent on job-specific requirements). Hip Waders, Life Vests, USGS approved personnel floatation devices.

Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
accuracy **3.) Check water depth using staff gauges**	• Contact with (CW) Hazardous Plants, Insects, and Animals **(See HASP Sections 5.6 and 5.8)**	• Survey the surroundings before any activity for any hazardous plants and animals detailed in Section 5.6 and 5.8 of the HASP especially dogs, venomous insects, bees, and snakes • Use bug repellant sprays • Be aware of any coworkers with any insect bite allergies • Wear proper PPE, i.e., long sleeved shirt and long pants • Personnel to be trained in the proper procedures to be followed in the event that an animal exhibits abnormal behavior. • Seek first aid for any bites or stings (insect, snake, or otherwise) • Provide medications to the SHSO for any sting/bite allergies you may have	• If major injury occurs or unable to perform work safely
	• CW Chemicals **(See HASP Section 4.4)**	• Always show precaution, follow protocol, and were proper PPE including gloves and safety glasses while handling chemicals. • Keep portable eyewash kits nearby.	• If major injury occurs or proper PPE not available
	• Exposure (E) to Toxic Substances **(See HASP Section 4.4)**	• All workers will be trained in expected site-specific hazards prior to beginning work on-site. • Standards and safe work practices will be developed for any newly discovered toxic hazards that are determined to be present at the site	• If major injury occurs or proper PPE not available
	• Poor Lighting	• All workers should have flashlight available with spare batteries • Two lines of communication should be open – cell phone and pager	• If unable to see properly • If no lines of communication exist
	• Mechanical Hazards – Pinch Points/Sharp edges/Impalement **(See HASP Sections 5.2, 5.3, and 5.5)**	• Care should be taken when working around the boats and sampling equipment. Each of these items feature sharp/hard edges that present cutting, scraping, and impalement hazards.	• If major injury occurs or unable to perform work safely

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Job Hazard Analysis

ENSR

JSA Type: ☒ Investigation ☐ O&M ☐Office ☐ Construction	☒ New ☐ Revised	**Date**: 9/19/07

Work Activity: Sediment Sampling in the Anacostia River and sediment processing on shore.

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Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
(Continued) Global Site Work Safety (applies to all activities) and **1.) Arrive on site** **2.) Use GPS to verify coordinate accuracy** **3.) Check water depth using staff gauges**	• E to Lightning Strikes **(See HASP Section 5.12.1)**	• Fieldwork shall not be conducted when lightning can be seen or thunder heard from the work area. When lightning and/or thunder occur, employees are to cease work, perform emergency equipment shut down as needed, and then seek shelter. • Minimize contact with ground and keep body parts touching ground close together.	• During any lightning or thunder
	• E to Thunderstorms, Hurricanes **(See HASP Section 5.12.1)**	• Listen to radio announcements for updates • Visually track threatening weather, cease outdoor activities if necessary • Seek shelter.	• During any lightning or thunder
	• Heat Stress (HS) **(See HASP Section 5.12.2)**	• Monitor self and other workers when ambient temperature exceed 85°F • Wear appropriate clothing • Consume sufficient quantities of water/electrolytes to avoid dehydration • Monitor yourself and co-workers for signs of heat stress • Take frequent breaks and take shelter to cool-off if feeling signs of heat stress	• If any signs of heat stress are observed

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Job Hazard Analysis

ENSR

JSA Type: ☒ Investigation ☐ O&M ☐ Office ☐ Construction	☒ New ☐ Revised	Date: 9/19/07

Work Activity: Sediment Sampling in the Anacostia River and sediment processing on shore.

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Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
(Continued) Global Site Work Safety (applies to all activities) and **1.) Arrive on site** **2.) Use GPS to verify coordinate accuracy** **3.) Check water depth using staff gauges**	• Cold Stress (CS) **(See HASP Section 5.12.3)**	• Wear adequate insulating clothing to maintain core temperatures above 36ºC • At air temperatures of 2ºC (35.6ºF) or less, if immersed in water or clothing becomes wet, immediately change clothing and get treated for hypothermia. • Cover metal handles of tools and control bars by thermal insulating material at temperatures below -1ºC (30ºF). • Protect hands with mittens if the air temperature is -17.5ºC (9ºF) or less. • Consume warm sweet drinks and soups to provide caloric intake and fluid volume. Limit the intake of coffee because of the diuretic and circulatory effects. • Work under constant protective observation (buddy system or supervision) at or below -12ºC (10ºF) ECT. • Monitor yourself and co-workers for signs of cold stress • Take frequent breaks and take shelter to warm if feeling signs of cold stress. Avoid sweaty conditions. • Minimize standing or sitting still.	• If any signs of cold stress are observed
4.) Equipment Mobilization/Setup/ Maintenance	• Overexertion (O) when lifting supplies/equipment **(See HASP Section 5.1)**	• Use equipment whenever possible. • Procure help when lifting awkward loads or materials that weigh greater than 60 lbs. • Use proper lifting techniques.	• Do not attempt to lift if the load is too heavy or worker is not capable
	• CW flying or broken glass when opening packages	• Open packages with care and wear appropriate PPE.	• If major injury occurs or unable to perform work safely
	• CW with chemicals inside broken containers and while instrument calibration **(See HASP Sections 4.4)**	• See Global site work safety.	• If major injury occurs or unable to perform work safely

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Job Hazard Analysis

ENSR

JSA Type: ☒ Investigation ☐ O&M ☐ Office ☐ Construction	☒ New ☐ Revised	Date: 9/19/07

Work Activity: Sediment Sampling in the Anacostia River and sediment processing on shore.

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Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
(Continued) **4.) Equipment Mobilization/Setup/ Maintenance**	• Fire Ignition while refilling generator fuel tank (if used) and or vehicle/boat, CW with fire **(See HASP Section 5.2.1)**	• Monitor generator usage; use care while refilling generator/vehicles with gas/diesel. • Do not leave vehicles idling on dry grass. • Maintain fire extinguisher in vehicle with current inspection tags. • For refueling, take boat gas tank to service station, keep on ground to reduce explosion hazard from sparking (esp. pickup bed liners), and fill tank at service station.	• If major injury occurs or unable to perform work safely
	• FL (from boat) **(See Global site work safety and HASP Sections 5.2 and 5.9)**	• Limit walking on elevated surface • Clean mud from boots prior to walking on boat • Only subcontractor company personnel to remove equipment off of boat.	• If major injury occurs or unable to perform work safely
5.) Sediment sampling in shallow waters	E to toxic substances • Dermal contact with contaminated media • Ingestion of contaminated media **(See Global site work safety and HASP Section 4.4)**	• Be alert during sampling to avoid splashing. • Wear proper PPE including protective gloves, protective coveralls, and safety glasses. • On exposure, rinse immediately with fresh water • Compliance with SOIs/SOPs regarding the collection of samples. • Training of personnel. • Practice good personal hygiene and implementation of decontamination procedures using disinfectant.	• If major injury occurs or proper PPE not available
	• CW Chemicals **(See HASP Section 4.4)**	• See Global site work safety.	• If major injury occurs or proper PPE not available
	• Drowning **(See HASP Sections 5.2 and 5.11)**	• See Global site work safety. • **NEVER** enter the river where the water is deeper than 3 feet.	• If major injury occurs or unable to perform work safely
	• O by repeated motion	• Take breaks during sampling and sample homogenization. • Maintain good ergonomics.	• If major injury occurs or unable to perform work safely

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Job Hazard Analysis

ENSR

JSA Type: ☒ Investigation ☐ O&M ☐ Office ☐ Construction	☒ New ☐ Revised	Date: 9/19/07

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Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
Sediment sampling in shallow waters (Continued)	• FS and FL due to Non Stabilized Surfaces and Weak or Narrow Embankments **(See Global site work safety and HASP Sections 5.9)**	• Avoid embankment edges. • Avoid positioning personnel downhill of equipment on embankments • Personnel to be cognizant of potential collapse of embankments. • Personnel to be cognizant of loose slopes. • Avoid wading in areas with deep water and/or swift currents (use PFDs if wading in moving water greater than 3 feet deep). • Use buddy system to provide assistance to personnel in water.	• If major injury occurs or unable to perform work safely
	• Slip and trip **(See Global site work safety and HASP Sections 5.9)**	• When working in river, wear appropriate footwear (waders) and gloves. • Be aware of the presence/potential of underwater debris (sharp edges, rebar, concrete, etc.)	• If major injury occurs or unable to perform work safely
	• SB tools/equipment **(See Global site work safety and HASP Section 5.5.2)**	• Care should be taken while collecting samples using a spade or similar sample. • Maintain eye contact with boat operator and other boat drivers (if present). • Employ buddy system.	• If major injury occurs or unable to perform work safely
	• CW electrical energy **(See HASP Section 5.5.2)**	• Use operable GFCIs for any hand tool. • Keep any electrical equipment/tool away from wet surfaces and pond water body.	• If major injury occurs or unable to perform work safely
	• CW Hazardous Plants, Insects, and Animals **(See Global site work safety and HASP Section 5.6 and 5.8)**	• Whenever possible, work in pairs. • Stay in communication (cell phone) with co-workers.	• If major injury occurs or unable to perform work safely
6.) Sediment sampling when in boat	• FL off boat, Drowning, Man Overboard **(See Global site work safety and HASP Sections 5.2 and 5.11)**	• Wear PFD at all times while on the deck of the vessel. • Be aware of your location at all times. • Be aware of trip hazards. • Hold handrails when getting on and off boat. • Do not perform tight maneuvers at high speed. • Orient boat perpendicular to waves to prevent excess rocking. • Conduct a safety briefing on boat hazards prior to sampling activities.	• If major injury occurs or unable to perform work safely

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Job Hazard Analysis

ENSR

JSA Type: ☒ Investigation ☐ O&M ☐ Office ☐ Construction	☒ New ☐ Revised	Date: 9/19/07

Work Activity: Sediment Sampling in the Anacostia River and sediment processing on shore.

Personal Protective Equipment (PPE): Minimum PPE is Level D including: hard hat, safety glasses, steel-toed boots, long-sleeved shirts, high visibility safety vest, hearing protection as needed, and gloves as needed (type dependent on job-specific requirements). Hip Waders, Life Vests, USGS approved personnel floatation devices.

Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
	• Boat Capsizing **(See HASP Sections 5.2 and 5.11)**	• Do not perform tight maneuvers at high speed. • Distribute weight in boat uniformly and avoid sudden shifts to one side of boat. • Be aware of changing weather conditions.	• If major injury occurs or unable to perform work safely
	• Use of Boats **(See HASP Sections 5.2 and 5.11)**	• Ensure that personnel working from or in boats are familiar and competent with boat operation and boating safety. • Operator to complete Boating Safety Operator's Checklist prior to launching boat. • Boating Safety Maintenance Checklist and Health and Safety Representative's Checklist to be completed at a minimum of once a week.	• If major injury occurs or unable to perform work safely
	E to toxic substances • Dermal contact with contaminated media • Ingestion of contaminated media **(See Global site work safety and HASP Sections 4.4)**	• Be alert during sampling to avoid splashing. • Wear proper PPE including protective gloves, protective coveralls, and safety glasses. • On exposure, rinse immediately with fresh water • Compliance with SOIs/SOPs regarding the collection of samples.	• If major injury occurs or proper PPE not available
(Continued) **Sediment sampling when in boat**	• SB sampling equipment aboard the vessel **(See HASP Section 5.2 and 5.5.2)**	• Wear a hard hat at all times on the vessel. • Obey the instructions of the vessel captain. • Keep proper clearance from equipment. • Never approach equipment without establishing eye contact with operator. • Establish protocol for hand and arm signals.	• If major injury occurs or unable to perform work safely
	• Slip, Trip, and FS on wet or oily surfaces and over equipment **(See Global site work safety and HASP Sections 5.9)**	• Wear appropriate slip-resistant boots with a steel toe. • Only the necessary personnel should be in the area of operation. • Always return equipment to proper storage location on the vessel. • Store coolers out of the walkway • Coil up anchor ropes when not in use.	• If major injury occurs or unable to perform work safely

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Job Hazard Analysis

ENSR

JSA Type: ☒ Investigation ☐ O&M ☐ Office ☐ Construction	☒ New ☐ Revised	Date: 9/19/07

Work Activity: Sediment Sampling in the Anacostia River and sediment processing on shore.

Personal Protective Equipment (PPE): Minimum PPE is Level D including: hard hat, safety glasses, steel-toed boots, long-sleeved shirts, high visibility safety vest, hearing protection as needed, and gloves as needed (type dependent on job-specific requirements). Hip Waders, Life Vests, USGS approved personnel floatation devices.

Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
	• CW Chemicals **(See HASP Section 4.4)**	• See Global site work safety.	• If major injury occurs or proper PPE not available
7.) Sample handling, packaging, and processing	• CW electrical energy **(See HASP Section 5.5.2)**	• Use operable GFCIs for any hand tool. • Keep any electrical equipment/tool away from wet surfaces and pond water body. • Inspect tools for frayed cords, damaged parts, etc. at the start of each day.	• If major injury occurs or unable to perform work safely
	• Caught between (CB) drill-mounted paddles used for sample homogenization **(See HASP Section 5.5)**	• Wear proper PPE including protective gloves, protective coveralls, and safety glasses. • Always keep attention focused on work when using the paddles. • Review tool instructions and safety features at the start of each day.	• If major injury occurs or unable to perform work safely
	• O by repeated motion	• Take breaks during sample preparation. • Maintain good ergonomics.	• If major injury occurs or unable to perform work safely
(Continued) **Sample handling, packaging, and processing**	• Chemical Hazards - spills **(See HASP Section 4.4)**	• Exercise proper placement, handling, and storage of the chemical preservatives used during the sampling event. • Read associated MSDS • Ensure that the chemical/contaminated material is stored in a secondary containment device so that an unscheduled release of the chemical/contaminated material cannot occur.	• If major injury occurs or proper PPE not available

	• E to toxic substances
	  • Dermal contact with contaminated media
	  • Ingestion of contaminated media
	(See Global site work safety and HASP Section 4.4)
		• Be alert during sample preparation to avoid splashing.
		• Wear proper PPE including protective gloves, protective coveralls, and safety glasses.
		• On exposure, rinse immediately with fresh water
		• Compliance with SOIs/SOPs regarding the collection of samples.
		• Training of personnel.
		• Practice good personal hygiene and implementation of decontamination procedures using disinfectant.
			• If major injury occurs or proper PPE not available

	• CW chemicals like acid and basic preservatives **(See HASP Section 4.4)**	• See Global site work safety.	• If major injury occurs or proper PPE not available

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Job Hazard Analysis

ENSR

JSA Type: ☒ Investigation ☐ O&M ☐ Office ☐ Construction	☒ New ☐ Revised	Date: 9/19/07

Work Activity: Sediment Sampling in the Anacostia River and sediment processing on shore.

Personal Protective Equipment (PPE): Minimum PPE is Level D including: hard hat, safety glasses, steel-toed boots, long-sleeved shirts, high visibility safety vest, hearing protection as needed, and gloves as needed (type dependent on job-specific requirements). Hip Waders, Life Vests, USGS approved personnel floatation devices.

Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
8.) Decontamination of equipment	• E and CW with airborne mists or vapors **(See HASP Section 4.4)**	• See Global site work safety.	• If major injury occurs or proper PPE not available
	• Chemical Hazards - spills **(See HASP Section 4.4)**	• Exercise proper placement, handling, and storage of the chemical solutions used during decontamination. • Read associated MSDS • Ensure that decontamination is carried out in a secondary containment device so that an unscheduled release of the contaminated decon water cannot occur.	• If major injury occurs or proper PPE not available
(Continued) Decontamination of equipment	E to toxic substances • Dermal contact with contaminated media • Ingestion of contaminated media **(See Global site work safety and HASP Section 4.4)**	• Be alert during decontamination to avoid splashing. • Wear proper PPE including protective gloves, protective coveralls, and safety glasses. • On exposure, rinse immediately with fresh water • Training of personnel.	• If major injury occurs or proper PPE not available
9.) Store excess material (sediment, etc.)	• Chemical Hazards – spills and leaks **(See HASP Section 4.4)**	• Inspect drums and the buckets used to transfer material into them for integrity and contaminant prior to adding material for disposal. • Store drums in a secure area; clearly label all drums. • Wear appropriate PPE. • Empty the bucket carefully so as to minimize splashing of wet materials.	• If major injury occurs or proper PPE not available
	• CB pinch points **(See HASP Sections 5.3 and 5.5)**	• When opening and closing drum lids, be aware of pinch points. • Wear proper PPE including protective gloves, protective coveralls, and safety glasses. • Always keep attention focused on work.	• If major injury occurs or unable to perform work safely
	• Lifting – Back and Foot injury **(See Global site work safety and HASP Section 5.1)**	• Get assistance when lifting or moving drums. Use drum dolly if necessary. • When transferring excess material into the drums, lift the bucket with the legs not the back and limit the amount of material in each transfer. • Hold the bucket used to transfer the excess material at both the top and the bottom.	• Do not attempt lift if the load is too heavy or worker is not capable

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Job Hazard Analysis

ENSR

JSA Type: ☒ Investigation ☐ O&M ☐Office ☐ Construction	☒ New ☐ Revised	**Date**: 9/19/07

Work Activity: Sediment Sampling in the Anacostia River and sediment processing on shore.

Personal Protective Equipment (PPE): Minimum PPE is Level D including: hard hat, safety glasses, steel-toed boots, long-sleeved shirts, high visibility safety vest, hearing protection as needed, and gloves as needed (type dependent on job-specific requirements). Hip Waders, Life Vests, USGS approved personnel floatation devices.

Development Team	Position/Title	Reviewed By	Position/Title	Date
Helen Jones	Geochemist	Kathleen McDonough	Environmental Engineer	9/21/07

❶ Job Steps[1]	❷ Potential Hazards[2]	❸ Critical Actions[3]	STOP Stop Work Criteria
10.) On-shore support	• FL into the water when loading/unloading equipment and supplies onto/off the vessel **(See Global site work safety and HASP Sections 5.1 and 5.11)**	• Wear Coast Guard-approved life jackets at all times while on peers, docks, or close to the shoreline. • Hold handrails when getting on and off boat.	• If major injury occurs or unable to perform work safely
	• CW Hazardous Plants, Insects, Animals, and aggressive humans **(See Global site work safety and HASP Section 5.6 and 5.8)**	• Whenever possible, work in pairs. Stay in communication (cell phone) with co-workers.	• If major injury occurs or unable to perform work safely
11.) Post-sampling	• Cross-contamination **(See HASP Section 9.0)**	Wash your hands prior to touching your food or other "clean" materials that may be tainted by what is on your hands	• If major injury occurs or proper PPE not available

1 – Target number of job steps: six to ten

2 – Codes for Potential Hazards:

Caught Between (CBT)	Contacted By (CB)	Caught On (CO)	Fall To Below (FB)	Overexertion (O)	Struck Against (SA)
Caught In (CI)	Contact With (CW)	Exposure (E)	Fall - Same Level (FS)	Release To (R)	Struck By (SB)

3 – Types of Critical Actions: Administrative Controls, Engineering Controls, PPE, and/or Safe Work Practice / SOP

Attachment C - Pre-Entry Briefing Attendance Form

Health and Safety Pre-Entry Briefing Attendance Form

Sediment Sampling and Site Investigation
Washington Navy Yard
Anacostia River
Washington, D.C.

Conducted by:		**Date Performed:**	
Topics Discussed:	1. Review of the content of the HASP (Required)		
	2.		
	3.		
	4.		

Printed Name	Signature	Representing

Attachment D - Supervisor's Incident Investigation Report

Supervisor's Incident Investigation Report

Injured Employee _____ Job Title _____

Home Office _____ Division/Department _____

Date/Time of Incident _____

Location of Incident _____

Witnesses to the Incident _____

Injury Incurred? _____ Nature of Injury _____

_____

Engaged in What Task When Injured? _____

_____

Will Lost Time Occur? _____ How Long? _____ Date Lost Time Began _____

Were Other Persons Involved/Injured? _____

_____

How Did the Incident Occur? _____

_____

_____

_____

_____

What Could Be Done to Prevent Recurrence of the Incident? _____

_____

_____

What Actions Have You Taken Thus Far to Prevent Recurrence? _____

_____

Supervisor's Signature _____ Title _____ Date _____

Reviewer's Signature _____ Title _____ Date _____

Note: If the space provided on this form is insufficient, provide additional information on a separate page and attach. The completed Incident investigation report must be submitted to the Regional Health and Safety Manager within two days of the occurrence of the Incident.

Attachment E - Material Safety Data Sheets

.....Alconox

SPI Supplies Division
Structure Probe, Inc.
P.O. Box 656 West Chester, PA 19381-0656 USA
Phone: *1-(610)-436-5400* ***Fax:*** *1-(610)-436-5755*
E-mail: *spi3spi@2spi.com*
WWW: http://www.2spi.com
Manufacturer's CAGE: 1P573

Material Safety Data Sheet

SPI #01200-AB and #01200A-AB Alconox® Powdered Detergent

Section 1: Identification




```
Date Effective..........  November 14, 2005
                          (most recent revision)

Chemical Name/Synonyms...  On Label:  Alconox®

Chemical Family.........  Anionic powdered detergent

Emergencies
Contacting CHEMTREC:

24 Hour Emergency Use Only #'s...
Worldwide phone: 1-(703)-527-3887
Worldwide FAX:   1-(703)-741-6090
Toll-free phone: 1-(800)-424-9300 USA only

Product or Trade Name....  SPI #01200-AB and #01200A-AB
                           Alconox® Powdered Detergent

CAS #...................  Not applicable

Chemical Formula.........  Not applicable
```

Section 2 Composition

Component Name	CAS #	OSHA	OSHA	ACGIH	ACGIH

No hazardous ingredients in Alconox Powdered Detergent as defined by the OSHA Standard and Hazardous Substance List 29 CFR 1910 Subpart Z.

Hazardous Material Information System USA	**Health**	**0**	**National Fire Protection Association USA**	
	Fire Hazard	**0**		
	Reactivity	**0**		
	Personal Protection			

NFPA (National Fire Protection Association) Rating (Scale 0-4):
HEALTH=0 FLAMMABILITY=0 REACTIVITY=0 OTHER=0 Not known

Section 3: Hazard Identification

Routes of entry
Inhalation? Yes
Skin? No
Ingestion? Yes

Health Hazards (Acute and chronic):
Inhalation of powder may prove locally irritating to mucous membranes. Ingestion may cause discomfort and/or diarrhea. Eye contact may prove irritating.

Carcinogenicity:
NTP? No
IARC Monographs? No
OSHA Regulated? No

Section 4: First Aid Measures

Signs and Symptoms of Exposure:
Exposure may irritate mucous membranes. May cause sneezing.

Medical conditions generally aggravated by exposure:

Not established. Unnecessary exposure to this product or any industrial chemical should be avoided. Respiratory conditions may be aggravated by powder if air borne.

Emergency and First Aid Procedures:

Eyes: Immediately flush eyes with copious amounts of water for minimum 15 minutes. Call physician.

Skin: Flush with plenty of water.

Ingestion: Drink large quantities of water or milk. Do not induce vomiting. If vomiting occurs re-administer fluids. See a physician for discomfort.

Section 5: Fire Fighting Measures

NFPA Rating: Not known

Extinguishing Media

Suitable/Not suitable:

SMALL FIRE: Use DRY chemical powder, water, foam, carbon dioxide

LARGE FIRE: Use extinguishing media suitable for the surrounding materials.

Special firefighting procedures:
Self-contained positive pressure breathing apparatus and protective clothing should be worn when fighting fires involving chemicals.

Unusual Fire/Explosion Hazards: None

Hazardous thermal decomposition products: None known.

Protection of fire fighters: No special measures are required.

Flammable Limits:
LEL: No data
UEL: No data

Section 6: Accidental Release Measures

Personal precautions: No special precautions

Environmental Precautions and Clean Up Methods:
Material foams profusely. Recover as much as possible and flush remainder to sewer. Material is biodegradable.

Section 7: Handling and Storage

Material should be stored in a dry area to prevent caking.

Section 8: Exposure Controls and Personal Protection

Engineering controls: Normal ventilation is normally required when handling or using this product. Avoid conditions that could produce dusting.

Personal Protective Equipment

Respiratory system: Dust mask recommended but not required.

Skin and body: Laboratory coat recommended but not required.

Hands: Impervious gloves recommended

Eyes: Goggles are recommended, especially when handling solutions irrespective of what they might be.

Other: Wash hands before eating, drinking, or smoking.

Section 9: Physical and Chemical Properties

Physical State and Appearance: White powder interspersed with cream colored flakes.

Odor: None

Boiling Point: Not applicable

Melting Point: Not applicable

Density (water = 1): Not applicable

Solubility: Appreciable, to 10% at ambient conditions.

Octanol/water partition coefficient: Not available

pH: Not known

Flash Point: None

Flammability: Non-flammable

Autoignition temperature: Not applicable

Section 10: Stability and Reactivity

Chemical Stability: The product is stable

Hazardous polymerization: Will not occur

Conditions to Avoid: None

Hazardous Products of Deposition: May release CO_2 on burning.

Reactions with Air and Water:
Does not react with air, water or other common materials.

Section 11: Toxicological Information

Summary: Not considered to be toxic to humans or animals.

Skin Effects: Can be locally irritating

Eye Irritation: Can be irritating to the eyes

Inhalation: Dust can be irritating to mucous membranes

Sensitization: Not known

Chronic toxicity: There is no known effect from the chronic exposure to this product.

Section 12: Ecological Information

Exotoxicity: Not know but it is expected to be low because the material is biodegradable.

Environmental Fate: It is biodegradable.

Bioaccumulation: Not expected to occur (because the material is biodegradable).

Section 13: Disposal Considerations

This material is NOT classified as a hazardous material by RCRA. Use only licensed transporters and permitted disposal facilities and conform to all laws.

Recycle to process, if possible.

Germany water class: VCI WGK: No products were found.

Methods of disposal; waste of residues; contaminated packaging:

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

Proper Shipping Name: Non-Regulated, No dangerous cargo

DOT Hazard Class: Non-Regulated, No dangerous cargo

UN/NA ID: Non-Regulated, No dangerous cargo

Packing Group: Not Applicable

Labels: Not Regulated

Marine Pollutant: No

NAER Guidebook: Not Regulated

DOT Status: Not Regulated

Land-Road/Railway:
ADR/RID Class: No dangerous cargo

Sea:
IMDG Class: No dangerous cargo

Air:
IATA-DGR Class: No dangerous cargo

Section 15: Regulatory Information

TSCA: All components of this product are listed on the TSCA 8(b) inventory. If identified components of this product are listed under the TSCA 12(b) Export Notification Rule, they will be listed below.

TSCA 12(b) Component Listed under TSCA Section

SARA Title 3: Section 313 Information/Emissions Reporting (**40 CFR 372**):

Component Reporting Threshold

SARA-Section 311/312:

No components present in this product are subject to the reporting requirements of this statute.

CERCLA Hazardous Substances and their Reportable Quantities:

Component Reportable Quantity

EU Regulations: Risk Phrases: This product is not classified according to the EU regulations.

Safety Phrases: Not applicable

Contains: Not applicable

California Prop. 65:
Proposition 65 requires manufacturers or distributors of consumer products into the State of California to provide a warning statement if the product contains ingredients for which the State has found to cause cancer, birth defects or other reproductive harm. If this product contains an ingredient listed by the State of California to cause cancer or reproductive toxicity, it will be listed below:

None found

Section 16: Other Information

Disclaimer of Liability:

Caution! Do not use SPI Supplies products or materials in applications involving implantation within the body; direct or indirect contact with the blood pathway; contact with bone, tissue, tissue fluid, or blood; or prolonged contact with mucous membranes. Products offered by SPI Supplies are not designed or manufactured for use in implantation in the human body or in contact with internal body fluids or tissues. SPI Supplies will not provide to customers making devices for such applications any notice, certification, or information necessary for such medical device use required by US FDA (Food and Drug Administration) regulation or any other statute. SPI Supplies and Structure Probe, Inc. make no representation, promise, express warranty or implied warranty concerning the suitability of these materials for use in implantation in the human body or in contact with internal body tissues of fluids.

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Thursday February 22, 2007

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Worldwide Distributors, Representatives, and Agents

…..Isobutylene Calibration Gas

MATERIAL SAFETY DATA SHEET

Prepared to U.S. OSHA, CMA, ANSI and Canadian WHMIS Standards

PART I *What is the material and what do I need to know in an emergency?*

1. PRODUCT IDENTIFICATION

CHEMICAL NAME; CLASS: **NON-FLAMMABLE GAS MIXTURE**

- Document Number: 002103

PRODUCT USE: For general analytical/synthetic chemical uses.

SUPPLIER/MANUFACTURER'S NAME: AIRGAS INC.

ADDRESS: 259 North Radnor-Chester Road
Suite 100
Radnor, PA 19087-5283

BUSINESS PHONE: 1-610-687-5253

EMERGENCY PHONE: 1-800-949-7937

International: 1-423-479-0293

DATE OF PREPARATION: April 22, 2001

2. COMPOSITION and INFORMATION ON INGREDIENTS

CHEMICAL NAME	CAS #	mole %	EXPOSURE LIMITS IN AIR					
			ACGIH		OSHA		NIOSH	OTHER
			TLV ppm	STEL ppm	PEL ppm	STEL ppm	IDLH ppm	ppm
Isobutylene	115-11-7	1 ppm - 1.7%	There are no specific exposure limits for Isobutylene. Isobutylene is a simple asphyxiant (SA). Oxygen levels should be maintained above 19.5%.					
Air	25635-88-5	Balance	There are no specific exposure limits applicable to Air.					
Air is a mixture of gases. The primary components of air, and the approximate concentration of each component, are listed below								
Nitrogen	7727-37-9	79%	There are no specific exposure limits for Nitrogen. Nitrogen is a simple asphyxiant (SA). Oxygen levels should be maintained above 19.5%.					
Oxygen	7782-44-7	21%	There are no specific exposure limits for Oxygen					

NE = Not Established. See Section 16 for Definitions of Terms Used.

NOTE (1): ALL WHMIS required information is included in appropriate sections based on the ANSI Z400.1-1998 format. This product has been classified in accordance with the hazard criteria of the CPR and the MSDS contains all the information required by the CPR.

3. HAZARD IDENTIFICATION

EMERGENCY OVERVIEW: This product is a colorless, odorless, non-flammable gas. The main health hazards associated with releases of this gas are related to the high pressure within the cylinder. Air, the main component of this product, is generally considered non-flammable, however, Air will support combustion. The flammable component of this gas mixture is below the LEL. A cylinder rupture hazard exists when this product, which is under pressure, is subjected to heat or flames. Emergency responders must wear personal protective equipment appropriate for the situation to which they are responding.

SYMPTOMS OF OVER-EXPOSURE BY ROUTE OF EXPOSURE: The most significant route of over-exposure for air is by inhalation at elevated or reduced pressure.

INHALATION: This product is non-toxic. Air, the main component of this product, is necessary for life.

OTHER POTENTIAL HEALTH EFFECTS: Contact with rapidly expanding gases (which are released under high pressure) may cause frostbite. Symptoms of frostbite include change in skin color to white or grayish-yellow. The pain after contact with liquid can quickly subside.

HEALTH EFFECTS OR RISKS FROM EXPOSURE: An Explanation in **Lay Terms**. Over-exposure to this product may cause the following health effects:

ACUTE: The most significant hazards associated with compressed air is the pressure hazard. Contact with rapidly expanding gases (which are released under high pressure) may cause frostbite. Symptoms of frostbite include change in skin color to white or grayish-yellow. The pain after contact with liquid can quickly subside.

CHRONIC: There are currently no known adverse health effects associated with chronic exposure to this gas.

TARGET ORGANS: ACUTE: Respiratory system under ambient low pressure conditions. Central nervous system under ambient high pressure conditions. CHRONIC: None expected.

PART II *What should I do if a hazardous situation occurs?*

4. FIRST-AID MEASURES

RESCUERS SHOULD NOT ATTEMPT TO RETRIEVE VICTIMS OF EXPOSURE TO THIS PRODUCT WITHOUT ADEQUATE PERSONAL PROTECTIVE EQUIPMENT. At a minimum, Self-Contained Breathing Apparatus equipment should be worn.

Victim(s) must be taken for medical attention. Rescuers should be taken for medical attention, if necessary. Take copy of label and MSDS to physician or other health professional with victim(s). Remove victim(s) to fresh air, as quickly as possible. In case of eye contact which leads to irritation, immediately flush eyes with copious amounts of water for at least 15 minutes. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Only trained personnel should administer supplemental oxygen.

In case of frostbite, place the frostbitten part in warm water. DO NOT USE HOT WATER. If warm water is not available, or is impractical to use, wrap the affected parts gently in blankets. Alternatively, if the fingers or

hands are frostbitten, place the affected area in the armpit. Encourage victim to gently exercise the affected part while being warmed. Seek immediate medical attention.
MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE: Acute or chronic respiratory conditions, as well as disorders involving the "Target Organs", as listed in Section 3 (Hazard Information), may be aggravated by overexposure to the components of this product.
RECOMMENDATIONS TO PHYSICIANS: Administer oxygen as soon as possible, following exposure.

5. FIRE-FIGHTING MEASURES

FLASH POINT: Not applicable.
AUTOIGNITION TEMPERATURE: Not applicable.
FLAMMABLE LIMITS (in air by volume, %):
Lower (LEL): Not applicable.
Upper (UEL): Not applicable.

5. FIRE-FIGHTING MEASURES (Continued)

FIRE EXTINGUISHING MATERIALS: Non-flammable gas. Use extinguishing media appropriate for surrounding fire.
UNUSUAL FIRE AND EXPLOSION HAZARDS: When involved in a fire, this material may decompose and produce toxic gases including carbon monoxide and carbon dioxide. Additionally, when involved in fire, the cylinders may rupture.
Explosion Sensitivity to Mechanical Impact: Not Sensitive.
Explosion Sensitivity to Static Discharge: Not Sensitive.
SPECIAL FIRE-FIGHTING PROCEDURES: Structural firefighters must wear Self-Contained Breathing Apparatus and full protective equipment. Move fire-exposed cylinders from area, if it can be done without risk to fire-fighters. Withdraw immediately in case of rising sounds from venting pressure relief devices or any discoloration of tanks or cylinders due to a fire.

6. ACCIDENTAL RELEASE MEASURES

SPILL AND LEAK RESPONSE: Uncontrolled releases should be responded to by trained personnel using pre-planned procedures. Proper protective equipment should be used. In case of a release, clear the affected area, protect people, and respond with trained personnel. Minimum Personal Protective Equipment should be **Level D: safety glasses, and mechanically-resistant gloves. Level B, which includes the use of Self-Contained Breathing Apparatus, should be worn when oxygen levels are below 19.5% or are unknown.** Locate and seal the source of the leaking gas. If this does not stop the release (or if it is not possible to reach the valve), allow the gas to release in place or remove it to a safe area and allow the gas to be released there.

PART III *How can I prevent hazardous situations from occurring?*

7. HANDLING and STORAGE

WORK PRACTICES AND HYGIENE PRACTICES: Do not eat or drink while handling chemicals.
STORAGE AND HANDLING PRACTICES: Cylinders should be stored in dry, well-ventilated areas away from sources of heat. Compressed gases can present significant safety hazards. Store containers away from heavily trafficked areas and emergency exits.

SPECIAL PRECAUTIONS FOR HANDLING GAS CYLINDERS: Protect cylinders against physical damage. Store in cool, dry, well-ventilated, fireproof area, away from flammable or combustible materials and corrosive atmospheres. Store away from heat and ignition sources and out of direct sunlight. Do not store near elevators, corridors or loading docks. Do not allow area where cylinders are stored to exceed 52°C (125°F). Isolate from incompatible materials including flammable materials (see Section 10, Stability and Reactivity), which can burn violently. Use only storage containers and equipment (pipes, valves, fittings to relieve pressure, etc.) designed for the storage of Air. Do not store containers where they can come into contact with moisture. Cylinders should be stored upright and be firmly secured to prevent falling or being knocked over. Cylinders can be stored in the open, but in such cases, should be protected against extremes of weather and from the dampness of the ground to prevent rusting. Never tamper with pressure relief devices in valves and cylinders. The following rules are applicable to situations in which cylinders are being used:

Before Use: Move cylinders with a suitable hand-truck. Do not drag, slide or roll cylinders. Do not drop cylinders or permit them to strike each other. Secure cylinders firmly. Leave the valve protection cap in-place until cylinder is ready for use.

During Use: Use designated CGA fittings and other support equipment. Do not use adapters. Do not heat cylinder by any means to increase the discharge rate of the product from the cylinder. Use check valve or trap in discharge line to prevent hazardous backflow into the cylinder. Do not use oils or grease on gas-handling fittings or equipment.

After Use: Close main cylinder valve. Replace valve protection cap. Mark empty cylinders "EMPTY".

NOTE: Use only DOT or ASME code containers. Earth-ground and bond all lines and equipment associated with this product. Close valve after each use and when empty. Cylinders must not be recharged except by or with the consent of owner. For additional information refer to the Compressed Gas Association Pamphlet P-1, *Safe Handling of Compressed Gases in Containers*. Additionally, refer to CGA Bulletin SB-2 "*Oxygen Deficient Atmospheres*".

7. HANDLING and STORAGE (Continued)

PROTECTIVE PRACTICES DURING MAINTENANCE OF CONTAMINATED EQUIPMENT: Follow practices indicated in Section 6 (Accidental Release Measures). Make certain application equipment is locked and tagged out safely. Purge gas handling equipment with inert gas (i.e. nitrogen) before attempting repairs. Always use product in areas where adequate ventilation is provided.

8. EXPOSURE CONTROLS - PERSONAL PROTECTION

VENTILATION AND ENGINEERING CONTROLS: Use with adequate ventilation.

RESPIRATORY PROTECTION: Maintain Oxygen levels above 19.5% in the workplace. If respiratory protection is needed, use only protection authorized in the U.S. Federal OSHA Standard (29 CFR 1910.134), applicable U.S. State regulations, or the Canadian CSA Standard Z94.4-93 and applicable standards of Canadian Provinces. Oxygen levels below 19.5% are considered IDLH by OSHA. In such atmospheres, use of a full facepiece pressure/demand SCBA or a full facepiece, supplied air respirator with auxiliary self-contained air supply is required under OSHA's Respiratory Protection Standard (1910.134-1998).

EYE PROTECTION: Splash goggles, face-shields or safety glasses. If necessary, refer to U.S. OSHA 29 CFR 1910.133, or Canadian Standards.

HAND PROTECTION: Wear mechanically-resistant gloves when handling cylinders of this product. If necessary, refer to U.S. OSHA 29 CFR 1910.138 or appropriate Standards of Canada.

BODY PROTECTION: Use body protection appropriate for task. If a hazard of injury to the feet exists due to falling objects, rolling objects, where objects may pierce the soles of the feet or where employee's feet may be exposed to electrical hazards, use foot protection, as described in U.S. OSHA 29 CFR.

9. PHYSICAL and CHEMICAL PROPERTIES

The following information is for **Air**, the main component of this product , unless otherwise stated:

RELATIVE VAPOR DENSITY: 1 EVAPORATION RATE (nBuAc = 1): Not applicable.
SPECIFIC GRAVITY: Not applicable. FREEZING POINT: -216.2°C (-357.2°F)
SOLUBILITY IN WATER: 1.49% (v/v) BOILING POINT @ 1 atmos: -194.3°C(-317.8°F)
VAPOR PRESSURE, mmHg @ 20°C:. pH: Not applicable.
EXPANSION RATIO: Not applicable. VAPOR PRESSURE: Not applicable.
SPECIFIC VOLUME: 13.3 ft_3/lb; (0.833 m_3/kg) ODOR THRESHOLD: Not applicable.
COEFFICIENT WATER/OIL DISTRIBUTION: Not applicable.

The following information is pertinent to this gas mixture:
APPEARANCE, ODOR AND COLOR: This product is a colorless, odorless gas.
HOW TO DETECT THIS SUBSTANCE (warning properties): There are no distinctive properties to this product. In terms of leak detection, fittings and joints can be painted with a soap solution to detect leaks, which will be indicated by a bubble formation.

10. STABILITY and REACTIVITY

STABILITY: Normally stable.
DECOMPOSITION PRODUCTS: None known.
MATERIALS WITH WHICH SUBSTANCE IS INCOMPATIBLE. Air (the main component of this product) is not compatible with fuels, in that air will support combustion. The Isobutylene component of this mixture is incompatible with Strong oxidizers (e.g., chlorine, bromine pentafluoride, oxygen, oxygen difluoride, and nitrogen trifluoride).
HAZARDOUS POLYMERIZATION: Will not occur.
CONDITIONS TO AVOID: Contact with incompatible materials and exposure to heat, sparks and other sources of ignition. Cylinders exposed to high temperatures or direct flame can rupture or burst.

PART III *How can I prevent hazardous situations from occurring?*

11. TOXICOLOGICAL INFORMATION

TOXICITY DATA: The following toxicology data are for the components of this gas mixture present at a level greater than 1 mole %:

ISOBUTYLENE:

LC50 (Inhalation-Rat) 620 gm/m3/4 hours LC50 (Inhalation-Mouse) 415 gm/m3/2 hours

SUSPECTED CANCER AGENT: No component of this gas mixture is found on the following lists: FEDERAL OSHA Z LIST, NTP, CAL/OSHA, IARC, and therefore is not considered to be, nor suspected to be, cancer causing agents by these agencies.
IRRITANCY OF PRODUCT: Contact with rapidly expanding gases can cause frostbite and damage to exposed skin and eyes.
SENSITIZATION OF PRODUCT: No component of this product is a skin or respiratory sensitizer.
REPRODUCTIVE TOXICITY INFORMATION: Listed below is information concerning the effects of this product and its components on the human reproductive system.
Mutagenicity: This product is not reported to cause mutagenic effects in humans.
Embryotoxicity: This product is not reported to cause embryotoxic effects in humans.
Teratogenicity: This product is not reported to cause teratogenic effects in humans.

Reproductive Toxicity: This product is not reported to cause adverse reproductive effects in humans.
A mutagen is a chemical which causes permanent changes to genetic material (DNA) such that the changes will propagate through generation lines. An embryotoxin is a chemical which causes damage to a developing embryo (i.e. within the first eight weeks of pregnancy in humans), but the damage does not propagate across generational lines. A teratogen is a chemical which causes damage to a developing fetus, but the damage does not propagate across generational lines. A reproductive toxin is any substance which interferes in any way with
the reproductive process.
BIOLOGICAL EXPOSURE INDICES: Biological Exposure Indices (BEIs) have been determined for the components of this product are as follows:

12. ECOLOGICAL INFORMATION

ENVIRONMENTAL STABILITY: This gas will be dissipated rapidly in well-ventilated areas.
EFFECT OF MATERIAL ON PLANTS or ANIMALS: No adverse effect is anticipated to occur to plant-life, except for frost produced in the presence of rapidly expanding gases.
EFFECT OF CHEMICAL ON AQUATIC LIFE: No evidence of an adverse effect of this product on aquatic life is currently available.

13. DISPOSAL CONSIDERATIONS

PREPARING WASTES FOR DISPOSAL: Product removed from cylinder must be disposed of in accordance with appropriate U.S. Federal, State and local regulations or with regulations of Canada and its Provinces. Return cylinders with residual product to Airgas, Inc. Do not dispose of locally.

14. TRANSPORTATION INFORMATION

THIS GAS MIXTURE IS HAZARDOUS AS DEFINED BY 49 CFR 172.101 BY THE U.S. DEPARTMENT OF TRANSPORTATION.

PROPER SHIPPING NAME: Compressed gases, n.o.s. (Air, Isobutylene)
HAZARD CLASS NUMBER and DESCRIPTION: 2.2 (Compressed Gas)
UN IDENTIFICATION NUMBER: UN 1956
PACKING GROUP: Not Applicable
DOT LABEL(S) REQUIRED: Compressed Gas
NORTH AMERICAN EMERGENCY RESPONSE GUIDEBOOK NUMBER (2000): 126

TRANSPORT CANADA, TRANSPORTATION OF DANGEROUS GOODS REGULATIONS: This gas mixture is considered as dangerous goods, per regulations of Transport Canada. Use the above information for the preparation of Canadian Shipments.

15. REGULATORY INFORMATION

ADDITIONAL U.S. REGULATIONS:

U.S. SARA REPORTING REQUIREMENTS: The components of this gas mixture are not subject to the reporting requirements of Sections 302, 304 and 313 of Title III of the Superfund Amendments and Reauthorization Act.

U.S. SARA THRESHOLD PLANNING QUANTITY: There are no specific Threshold Planning Quantities for this material. The default Federal MSDS submission and inventory requirement filing threshold of 10,000 lb (4,540 kg) may apply, per 40 CFR 370.20.

U.S. CERCLA REPORTABLE QUANTITY (RQ): Not applicable.

U.S. TSCA INVENTORY STATUS: The components of this product are listed on the TSCA Inventory.

OTHER U.S. FEDERAL REGULATIONS: Not applicable.

U.S. STATE REGULATORY INFORMATION: The components of this gas mixture are covered under specific State regulations, as denoted below:

Alaska - Designated Toxic and Hazardous Substances: None.
California - Permissible Exposure Limits for Chemical Contaminants: None.
Florida - Substance List: Isobutylene. **Illinois - Toxic Substance List:** None.
Kansas - Section 302/313 List: None.
Minnesota - List of Hazardous Substances: Isobutylene.
Massachusetts - Substance List: None.
Missouri - Employer Information/Toxic Substance List: None.
New Jersey - Right to Know Hazardous Substance List: Isobutylene.
North Dakota - List of Hazardous Chemicals, Reportable Quantities: None.
Pennsylvania - Hazardous Substance List: Isobutylene.
Rhode Island - Hazardous Substance List: None.
Texas - Hazardous Substance List: None.
West Virginia - Hazardous Substance List: None.
Wisconsin - Toxic and Hazardous Substances: None.

CALIFORNIA SAFE DRINKING WATER AND TOXIC ENFORCEMENT ACT (PROPOSITION 65): No component of this product is on the California Proposition 65 Lists.

LABELING: **CAUTION:** HIGH PRESSURE GAS.
MAY ACCELERATE COMBUSTION.
Keep oil and grease away.
Use equipment rated for cylinder pressure.
Close valve after each use and when empty.
Use in accordance with the Material Safety Data Sheet.

FIRST-AID: **IF INHALED**, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
IN CASE OF FROSTBITE, obtain immediate medial attention.
DO NOT REMOVE THIS PRODUCT LABEL.

ADDITIONAL CANADIAN REGULATIONS:
CANADIAN DSL INVENTORY: The components of this product are listed on the DSL Inventory.
OTHER CANADIAN REGULATIONS: Not applicable.
CANADIAN ENVIRONMENTAL PROTECTION ACT (CEPA) PRIORITIES SUBSTANCES LISTS: The components of this product are not on the CEPA Priorities Substances Lists.
CANADIAN WHMIS SYMBOLS: **Class A:** Compressed Gases

16. OTHER INFORMATION

PREPARED BY: CHEMICAL SAFETY ASSOCIATES, Inc.
9163 Chesapeake Drive, San Diego, CA 92123-1002
858/565-0302

The information contained herein is based on data considered accurate. However, no warranty is expressed or implied regarding the accuracy of these data or the results to be obtained from the use thereof. AirGas, Inc. assumes no responsibility for injury to the vendee or third persons proximately caused by the material if reasonable safety procedures are not adhered to as stipulated in the data sheet. Additionally, AirGas, Inc. assumes no responsibility for injury to vendee or third persons proximately caused by abnormal use of the material even if reasonable safety procedures are followed. Furthermore, vendee assumes the risk in his use of the material.

DEFINITIONS OF TERMS

A large number of abbreviations and acronyms appear on a MSDS. Some of these which are commonly used include the following:
CAS #: This is the Chemical Abstract Service Number which uniquely identifies each constituent.
EXPOSURE LIMITS IN AIR:
ACGIH - American Conference of Governmental Industrial Hygienists, a professional association which establishes exposure limits. **TLV -** Threshold Limit Value - an airborne concentration of a substance which represents conditions under which it is generally believed that nearly all workers may be repeatedly exposed without adverse effect. The duration must be considered, including the 8-hour Time Weighted Average **(TWA)**, the 15-minute Short Term Exposure Limit, and the instantaneous Ceiling Level **(C)**. Skin absorption effects must also be considered.

OSHA - U.S. Occupational Safety and Health Administration. **PEL -** Permissible Exposure Limit - This exposure value means exactly the same as a TLV, except that it is enforceable by OSHA. The OSHA Permissible Exposure Limits are based in the 1989 PELs and the June, 1993 Air Contaminants Rule (Federal Register: 58: 35338-35351 and 58: 40191). Both the current PELs and the vacated PELs are indicated. The phrase, "Vacated 1989 PEL," is placed next to the PEL which was vacated by Court Order.

IDLH - Immediately Dangerous to Life and Health **-** This level represents a concentration from which one can escape within 30- minutes without suffering escape-preventing or permanent injury. **The DFG - MAK** is the Republic of Germany's Maximum Exposure Level, similar to the U.S. PEL. **NIOSH** is the National Institute of Occupational Safety and Health, which is the research arm of the U.S. **O**ccupational **S**afety and **H**ealth

Administration (**OSHA**). NIOSH issues exposure guidelines called **R**ecommended **E**xposure **L**evels (**REL**s). When no exposure guidelines are established, an entry of **NE** is made for reference.

HAZARD RATINGS:
HAZARDOUS MATERIALS IDENTIFICATION SYSTEM: Health Hazard: **0** (minimal acute or chronic exposure hazard); **1** (slight acute or chronic exposure hazard); **2** (moderate acute or significant chronic exposure hazard); **3** (severe acute exposure hazard; onetime overexposure can result in permanent injury and may be fatal); **4** (extreme acute exposure hazard; onetime overexposure can be fatal). Flammability Hazard: **0** (minimal hazard); **1** (materials that require substantial pre-heating before burning); **2** (combustible liquid or solids; liquids with a flash point of 38-93 C [100-200 F]); **3** (Class IB and IC flammable liquids with flash points below 38 C [100 F]); **4** (Class IA flammable liquids with flash points below 23 C [73 F] and boiling points below 38 C [100 F]. Reactivity Hazard: **0** (normally stable); 1 (material that can become unstable at elevated temperatures or which can react slightly with water); **2** (materials that are unstable but do not detonate or which can react violently with water); **3** (materials that can detonate when initiated or which can react explosively with water); **4** (materials that can detonate at normal temperatures or pressures).
PERSONAL PROTECTIVE EQUIPMENT CODES: B: Gloves and goggles; **C:** Gloves, goggles, rubber apron (appropriate body protection); **D:** Gloves, goggles, faceshield; rubber apron (appropriate body protection);. **X:** Special attention should be given to PPE Selection.

NATIONAL FIRE PROTECTION ASSOCIATION: Health Hazard: **0** (material that on exposure under fire conditions would offer no hazard beyond that of ordinary combustible materials); **1** (materials that on exposure under fire conditions could cause irritation or minor residual injury); **2** (materials that on intense or continued exposure under fire conditions could cause temporary incapacitation or possible residual injury); **3** (materials that can on short exposure could cause serious temporary or residual injury); **4** (materials that under very short exposure could cause death or major residual injury). Flammability Hazard and Reactivity Hazard: Refer to definitions for “Hazardous Materials Identification System”.

FLAMMABILITY LIMITS IN AIR:
Much of the information related to fire and explosion is derived from the National Fire Protection Association (**NFPA**). Flash Point – Minimum temperature at which a liquid gives off sufficient vapors to form an ignitable mixture with air. Autoignition Temperature: The minimum temperature required to initiate combustion in air with no other source of ignition. LEL - the lowest percent of vapor in air, by volume, that will explode or ignite in the presence of an ignition source. UEL – the highest percent of vapor in air, by volume, that will explode or ignite in the presence of an ignition source.

TOXICOLOGICAL INFORMATION:
Possible health hazards as derived from human data, animal studies, or from the results of studies with similar compounds are presented. Definitions of some terms used in this section are: **LD50** - Lethal Dose (solids & liquids) which kills 50% of the exposed animals; **LC50** – Lethal Concentration (gases) which kills 50% of the exposed animals; **ppm** concentration expressed in parts of material per million parts of air or water; **mg/m3** concentration expressed in weight of substance per volume of air; **mg/kg** quantity of material, by weight, administered to a test subject, based on their body weight in kg. Data from several sources are used to evaluate the cancer-causing potential of the material. The sources are: **IARC** - the International Agency for Research on Cancer; **NTP** - the National Toxicology Program, **RTECS** - the Registry of Toxic Effects of Chemical Substances, **OSHA** and **CAL/OSHA**. IARC and NTP rate chemicals on a scale of decreasing

potential to cause human cancer with rankings from 1 to 4. Subrankings (2A, 2B, etc.) are also used. Other measures of toxicity include **TDLo**, the lowest dose to cause a symptom and **TCLo** the lowest concentration to cause a symptom; **TDo**, **LDLo**, and **LDo**, or **TC**, **TCo**, **LCLo**, and **LCo**, the lowest dose (or concentration) to cause lethal or toxic effects. **BEI** - Biological Exposure Indices, represent the levels of determinants which are most likely to be observed in specimens collected from a healthy worker who has been exposed to chemicals to the same extent as a worker with inhalation exposure to the TLV. Ecological Information: EC is the effect concentration in water.

REGULATORY INFORMATION:

This section explains the impact of various laws and regulations on the material. **EPA** is the U.S. Environmental Protection Agency. **WHMIS** is the Canadian Workplace Hazardous Materials Information System. **DOT** and **TC** are the U.S. Department of Transportation and the Transport Canada, respectively. Superfund Amendments and Reauthorization Act **(SARA)**; the Canadian Domestic/Non-Domestic Substances List **(DSL/NDSL)**; the U.S. Toxic Substance Control Act **(TSCA)**; Marine Pollutant status according to the **DOT**; the Comprehensive Environmental Response, Compensation, and Liability Act **(CERCLA or Superfund)**; and various state regulations.

MATERIAL SAFETY DATA SHEET: SIMPLE GREEN®

I. PRODUCT & COMPANY INFORMATION

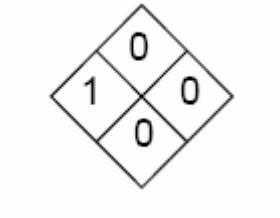
PRODUCT NAME: SIMPLE GREEN® CLEANER / DEGREASER Page 1 of 4
COMPANY NAME: SUNSHINE MAKERS, INC. Version No. 1006
15922 Pacific Coast Highway Issue Date: March, 1999
Huntington Harbour, CA 92649 USA
Telephone: 800-228-0709 x 562-795-6000
Fax: 562-592-3034
Website: www.simplegreen.com
For 24-hour emergency, call Chem-Tel, Inc.: 800-255-3924
USE OF PRODUCT: An all purpose cleaner and degreaser used undiluted or diluted in water for direct, spray,and dip tank procedures.

II. INGREDIENT INFORMATION

The only ingredient of Simple Green® with established exposure limits is undiluted 2-butoxyethanol (<6%) (Butyl Cellosolve; CAS No. 111-76-2): the OSHA PEL and ACGIH TLV is 25 ppm (skin). Note, however, that Butyl Cellosolve is only one of the raw material ingredients that undergo processing and dilution during the manufacture of Simple Green®. Upon completion of the manufacturing process, Simple Green® does not possess the occupational health risks associated with exposure to undiluted Butyl Cellosolve. Verification of this is contained in the independent test results detailed under “Toxicological Information” on Page 3 of this MSDS.
The Butyl Cellosolve in Simple Green® is part of a chemical category (glycol ethers) regulated by the Emergency Planning and Community Right-to-Know Act (SARA, Title III, section 313); therefore, a reporting requirement exists. Based upon chemical analysis, Simple Green® contains no known EPA priority pollutants, heavy metals, or chemicals listed under RCRA, CERCLA, or CWA. Analysis by TCLP (Toxicity Characteristic Leaching Procedure) according to RCRA revealed no toxic organic or inorganic constituents. All components of Simple Green® are listed on the TSCA Chemical Substance Inventory.

III. HAZARDS IDENTIFICATION

UN Number: Not required
Dangerous Goods Class: Nonhazardous
Hazard Rating (NFPA/HMIS) Rating Scale
Health = 1* Reactivity = 0 0 = minimal 1 = slight
Fire = 0 Special = 0 2 = moderate 3 = serious
4 = severe
*Mild eye irritant, non-mutagenic and non-carcinogenic. **None of the ingredients in Simple Green® are regulated or listed as potential cancer agents by Federal OSHA, NTP, or IARC.**



IV. FIRST AID MEASURES

SYMPTOMS OF OVEREXPOSURE AND FIRST AID TREATMENT

Eye contact: Reddening may develop. Immediately rinse the eye with large quantities of cool water; continue 10-15 minutes or until the material has been removed; be sure to remove contact lenses, if present, and to lift upper and lower lids during rinsing. Get medical attention if irritation persists.

Skin contact: Minimal effects, if any; rinse skin with water, rinse shoes and launder clothing before reuse. Reversible reddening may occur in some dermal-sensitive users; thoroughly rinse area and get medical attention if reaction persists.
Swallowing: Essentially non-toxic. Give several glasses of water to dilute; do not induce vomiting. If stomach upset occurs, consult physician.
Inhalation: Non-toxic. Exposures to concentrate-mist may cause mild irritation of nasal passages or throat; remove to fresh air. Get medical attention if irritation persists.

V. FIRE FIGHTING MEASURES

Simple Green® is stable, not flammable, and will not burn.
Flash Point/Auto-Ignition: Not flammable.
Flammability Limits: Not flammable.
Extinguishing Media: Not flammable/nonexplosive. No special procedures required.
Special Fire Fighting Procedures: None required.

VI. ACCIDENTAL RELEASE MEASURES

Recover usable material by convenient method; residual may be removed by wipe or wet mop. If necessary, unrecoverable material may be washed to drain with large quantities of water.

VII. HANDLING, STORAGE & TRANSPORT INFORMATION

No special precautions are required. **This product is non-hazardous for storage and transport according to the U.S. Department of Transportation Regulations.** Simple Green® requires no special labeling or placarding to meet U.S. Department of Transportation requirements.
UN Number: Not required
Dangerous Goods Class: Nonhazardous

VIII. EXPOSURE CONTROLS

Exposure Limits: The Simple Green® formulation presents no health hazards to the user when used according to label directions for its intended purposes. Mild skin and eye irritation is possible (please see Eye contact and Skin contact in Section IV.).
Ventilation: No special ventilation is required during use.
Human Health Effects or Risks from Exposure: Adverse effects on human health are not expected from Simple Green®, based upon twenty years of use without reported adverse health incidence in diverse population groups, including extensive use by inmates of U.S. Federal prisons in cleaning operations. Simple Green® is a mild eye irritant; mucous membranes may become irritated by concentrate-mist. Simple Green® is not likely to irritate the skin in the majority of users. Repeated daily application to the skin without rinsing, or continuous contact of Simple Green® on the skin may lead to temporary, but reversible, irritation.
Medical Conditions Aggravated by Exposure: No aggravation of existing medical conditions is expected; dermal-sensitive users may react to dermal contact by Simple Green®.

IX. PERSONAL PROTECTION

Precautionary Measures: No special requirements under normal use conditions.
Eye Protection: Caution, including reasonable eye protection, should always be used to avoid eye contact where splashing may occur.
Skin Protection: No special precautions required; rinse completely from skin after contact.
Respiratory Protection: No special precautions required.
Work and Hygienic Practices: No special requirements. Wash or rinse hands before touching eyes or contact lenses.

X. PHYSICAL AND CHEMICAL PROPERTIES

Appearance/odor: Translucent green liquid with characteristic sassafras odor.
Specific Gravity: 1.0257 **Vapor Pressure:** 17 mm Hg @ 20 °C; 22 mm Hg @ 25 °C
pH of concentrate: 9.5 **Vapor Density:** 1.3 (air = 1)
Evaporation: >1 (butyl acetate = 1) **Density:** 8.5 lbs./gallon
Boiling Point: 110 °C (231 °F)
Freezing Point: -9 °C (16 °F) If product freezes, it will reconstitute without loss of efficacy when brought back to room
temperature and agitated.

VOC Composite Partial Pressure: 0.006 mm Hg @ 20 °C
Volatile Organic Compounds (VOCs): 7.96 g/L per ASTM Method 3960-90. Per California AQMD's VOC test method, product must be diluted at least 2 parts of water to 1 part Simple Green® in order to meet SCAQMD Rule 1171 & Rule 1122 and BAAQMD Regulation 8-16 VOC requirements for solvent cleaning operations.
Water Solubility: Completely soluble in water. The higher salt concentrations in marine ecosystems will lead to complexes with Simple Green® that may become visible at ratios above one part Simple Green® to 99 parts seawater.
Ash Content: At 600 °F: 1.86% by weight.
Nutrient Content: Nitrogen: <1.0% by weight (fusion and qualitative test for ammonia).
Phosphorus: 0.3% by formula.
Sulfur: 0.6% by weight (barium chloride precipitation method).
Detection: Simple Green® has a characteristic sassafras odor that is not indicative of any hazardous situation.

XI. STABILITY AND REACTIVITY INFORMATION

Nonreactive. Simple Green® is stable, even under fire conditions, and will not react with water or oxidizers. Hazardous polymerization will not occur.

XII. TOXICOLOGICAL INFORMATION

Nonhuman Toxicity

Acute Mortality Studies:
Oral LD_{50} (rat): >5.0 g/kg body weight
Dermal LD_{50} (rabbit): >2.0 g/kg body weight
Dermal Irritation: Only mild, but reversible, irritation was found in a standard 72-hr test on rabbits. A value of 0.2 (non-irritating) was found on a scale of 8.
Eye Irritation: With or without rinsing with water, the irritation scores in rabbits at 24 hours did not exceed 15 (mild irritant) on a scale of 110.
Subchronic dermal effects: No adverse effects, except reversible dermal irritation, were found in rabbits exposed to Simple Green® (up to 2.0 g/kg/day for 13 weeks) applied to the skin of 25 males and 25 females. Only female body weight gain was affected. Detailed microscopic examination of all major tissues showed no adverse changes.
Fertility Assessment by Continuous Breeding: The Simple Green® formulation had no adverse effect on fertility and reproduction in CD-1 mice with continuous administration for 18 weeks, and had no adverse effect on the reproductive performance of their offspring.

XIII. BIODEGRADABILITY AND ENVIRONMENTAL TOXICITY INFORMATION

Biodegradability:

Simple Green® is readily decomposed by naturally occurring microorganisms. The biological oxygen demand (BOD), as a percentage of the chemical oxygen demand (COD), after 4, 7, and 11 days was 56%, 60%, and 70%, respectively. Per OECD Closed Bottle Test, Simple Green® meets OECD and EPA recommendations for ready biodegradability. In a standard biodegradation test with soils from three different countries, Butyl Cellosolve reached 50% degradation in 6 to 23 days, depending upon soil type, and exceeded the rate of degradation for glucose which was used as a control for comparison.

Environmental Toxicity Information:

Simple Green® is considered practically non-toxic per EPA's aquatic toxicity scale. Simple Green® is non-lethal to any of the marine and estuarine test animals listed in the following table at concentrations below 200 mg/L (0.02%). This table shows the Simple Green® concentrations that are likely to be lethal to 50% of the exposed organisms.

	LC_{50} in mg/L (ppm)	
	48-hour	**96-hour**
Marine Fish:		
Mud minnow (Fundulus heteroclitus)	1690	1574
Whitebait (Galaxias maculatus)	210	210
Marine/Estuarine Invertebrates:		
Brine Shrimp (Artemia salina)	610	399

Grass Shrimp (Palaemonetes pugio) 270 220
Green-lipped Mussel (Perna canaliculus) 220 220
Mud Snail (Potamopyrgus estuarinus) 410 350

XIV. DISPOSAL CONSIDERATIONS

Simple Green® is fully water soluble and biodegradable and will not harm sewage-treatment microorganisms if disposal by sewer or drain is necessary. Dispose of in accordance with all applicable local, state, and federal laws.

XV. OTHER INFORMATION

Containers: Simple Green® residues can be completely removed by rinsing with water; the container may be recycled or applied to other uses.
Electrical Wiring Polyimide insulated wiring is not affected by exposure to Simple Green®. After immersion in Simple Compatibility: Green® for 14 days at 74°F, the 61 cm piece of polyimide insulated wire passed a one minute dielectric proof test at 2500 volts (ASTM D-149).
Contact Point: Sunshine Makers, Inc., Research and Development Division: 562-795-6000.

*** NOTICE ***

All information appearing herein is based upon data obtained by the manufacturer and recognized technical sources. Judgments as to the suitability of information herein for purchaser's purposes are necessarily purchaser's responsibility. Therefore, although reasonable care has been taken in the preparation of this information, Sunshine Makers, Inc. or its distributors extends no warranties, makes no representations and assumes no responsibility as to the suitability of such information for application to purchaser's intended purposes or for consequences of its use.

Appendix E: Field Documentation Forms

Waypoint # 87

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-01
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-01
RETEC Project Name:	ESTCP	**Time of Collection:**	1015
Date Sample Collection:	11/7/07	**Sampling Personnel:**	HA, AJ, AM
Weather:	Sunny, 50s	**Sampling Personnel:**	Rob, Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.4180
Longitude (W):	76° 59.3067

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	8.6		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	12.27	**Temperature (°C):**	12.19
Salinity (ppm):	0.300	**Salinity (ppm):**	0.303
Dissolved Oxygen (mg/L):	43.1	**Dissolved Oxygen (mg/L):**	43.1
Conductivity (µmhos/cm):	0.227	**Conductivity (µmhos/cm):**	0.229
pH:	7.75	**pH:**	7.66
Turbidity (NTU):	20.1	**Turbidity (NTU):**	287.6
ORP (mv):	100.9	**ORP (mv):**	97.9
Field Instrument Calibration Completed:			

Other Observations or Notes

East of USACE dock off north shoreline of river

brown silt → soft, thick organic layer - leaves, twigs, shells (snail)

no odor, no sheen

Waypoint # 88

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-02
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-02
RETEC Project Name:	ESTCP	**Time of Collection:**	1025
Date Sample Collection:	11/7/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	50s, sunny	**Sampling Personnel:**	Rob; Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3876
Longitude (W):	76° 59.3627

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	9.5		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 12.16		**Temperature (°C):** 12.27	
Salinity (ppm): 0.301		**Salinity (ppm):** 0.301	
Dissolved Oxygen (mg/L): 44.1		**Dissolved Oxygen (mg/L):** 42.0	
Conductivity (µmhos/cm): 0.227		**Conductivity (µmhos/cm):** 0.228	
pH: 7.73		**pH:** 7.64	
Turbidity (NTU): 19.6		**Turbidity (NTU):** 230.7	
ORP (mv): 103.3		**ORP (mv):** 106.0	
Field Instrument Calibration Completed:			

Other Observations or Notes

West of USACE dock off N. Shore of river
HC odor, HC sheen, gray staining, in area of former Washington gas MGP

dark brown silty clay, trace sand (very fine grain), suspected black product, some gravel, moderate sheen, moderate HC like odor

Waypoint #89

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:** AR-03	
RETEC Project Number:	09000-422-100	**Sediment Sample:** AR-03	
RETEC Project Name:	ESTCP	**Time of Collection:** 1032	
Date Sample Collection: 11/7/07		**Sampling Personnel:** HA, HJ, AM	
Weather: 50s, sunny		**Sampling Personnel:** Rob, Wayne	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3748
Longitude (W):	76° 59.3851

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	4		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 11.27		**Temperature (°C):** 12.42	
Salinity (ppm): 0.320		**Salinity (ppm):** 0.342	
Dissolved Oxygen (mg/L): 44.1		**Dissolved Oxygen (mg/L):** 42.0	
Conductivity (µmhos/cm): 0.246		**Conductivity (µmhos/cm):** 0.247	
pH: 7.71		**pH:** 7.65	
Turbidity (NTU): 29.0		**Turbidity (NTU):** 30.9	
ORP (mv): 111.3		**ORP (mv):** 112.1	
Field Instrument Calibration Completed:			

Other Observations or Notes

West of AR-02 near outfall, in middle of conservation dock -> 2 buckets
Took pictures of outfall

med gravel, dark brown, little sand, trace large pebbles
visible suspected product
trace sheen, slight HC-like odor

Waypoint #97

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-04
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-04
RETEC Project Name:	ESTCP	**Time of Collection:**	1315
Date Sample Collection:	11/7/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	Partly Cloudy, 50s	**Sampling Personnel:**	Rob, Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3146
Longitude (W):	76° 59.4671

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	8.4		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	12.05	**Temperature (°C):**	12.09
Salinity (ppm):	0.306	**Salinity (ppm):**	0.309
Dissolved Oxygen (mg/L):	43.1	**Dissolved Oxygen (mg/L):**	42.0
Conductivity (µmhos/cm):	0.231	**Conductivity (µmhos/cm):**	0.233
pH:	7.89	**pH:**	7.78
Turbidity (NTU):	21.7	**Turbidity (NTU):**	39.4
ORP (mv):	179.0	**ORP (mv):**	17.8
Field Instrument Calibration Completed:			

Other Observations or Notes

Near outfall on west side of 11th St. bridge
Slight HCL odor

brown silt, trace organics, no sheen

Waypoint #98

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	Station Identification:	AR-05
RETEC Project Number:	09000-422-100	Sediment Sample:	AR-05
RETEC Project Name:	ESTCP	Time of Collection:	1330
Date Sample Collection:	11/7/07	Sampling Personnel:	HA, HJ, AM
Weather:	Partly Cloudy, 50s	Sampling Personnel:	Rob i Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.2674
Longitude (W):	76° 59.5119

Photographic Record

Photographic Description:		Photographic Description:	
Photographic Description:		Photographic Description:	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	11.5	
Type of Sediment Sampler Used:	Ponar	

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.91	Temperature (°C):	11.88
Salinity (ppm):	0.309	Salinity (ppm):	0.315
Dissolved Oxygen (mg/L):	44.1	Dissolved Oxygen (mg/L):	42.0
Conductivity (µmhos/cm):	0.232	Conductivity (µmhos/cm):	0.236
pH:	7.74	pH:	7.67
Turbidity (NTU):	20.3	Turbidity (NTU):	47.0
ORP (mv):	60.7	ORP (mv):	65.6
Field Instrument Calibration Completed:			

Other Observations or Notes

East of Navy yard campus building a couple hundred feet from north shore of river.

brown silt, organic layer - leaves, twigs
no odor, no sheen

Waypoint # 105

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	Station Identification: AR-06	
RETEC Project Number:	09000-422-100	Sediment Sample: AR-06	
RETEC Project Name:	ESTCP	Time of Collection: 0955	
Date Sample Collection: 11/18/07		Sampling Personnel: HA, HJ, AM	
Weather: 40 ish, Sunny		Sampling Personnel: Rob, Wayne Reynolds Spencer	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.2365
Longitude (W):	76° 59.5727

Photographic Record

Photographic Description:		Photographic Description:	
Photographic Description:		Photographic Description:	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	13	
Type of Sediment Sampler Used:	Ponar	

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 11.80		Temperature (°C): 11.74	
Salinity (ppm): 0.304		Salinity (ppm): 0.306	
Dissolved Oxygen (mg/L): 43.1		Dissolved Oxygen (mg/L): 41.0	
Conductivity (µmhos/cm): 0.227		Conductivity (µmhos/cm): 0.228	
pH: 7.96		pH: 7.89	
Turbidity (NTU): 17.1		Turbidity (NTU): 56.2	
ORP (mv): 217.3		ORP (mv): 215.9	
Field Instrument Calibration Completed:			

Other Observations or Notes

brown silt, some organics - leaves, twigs, trace sheen, slight HC-like odor

Waypoint #106

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-07
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-07
RETEC Project Name:	ESTCP	**Time of Collection:**	1002
Date Sample Collection:	11/08/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	40s, sunny	**Sampling Personnel:**	RR, WS

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.2632
Longitude (W):	76° 59.1614

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	13.6		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.86	**Temperature (°C):**	11.85
Salinity (ppm):	0.305	**Salinity (ppm):**	0.313
Dissolved Oxygen (mg/L):	43.1	**Dissolved Oxygen (mg/L):**	41.0
Conductivity (µmhos/cm):	0.228	**Conductivity (µmhos/cm):**	0.237
pH:	7.88	**pH:**	7.74
Turbidity (NTU):	19.2	**Turbidity (NTU):**	1305.6
ORP (mv):	208.8	**ORP (mv):**	-13.5
Field Instrument Calibration Completed:			

Other Observations or Notes

brown silt w/ organic layer, moderate HC-like odor, no sheen

Waypoint #102

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:** AR-08	
RETEC Project Number:	09000-422-100	**Sediment Sample:** AR-08	
RETEC Project Name:	ESTCP	**Time of Collection:** 0900	
Date Sample Collection: 11/8/07		**Sampling Personnel:** HA, HS, AM	
Weather: Sunny, 36s		**Sampling Personnel:** Rob, Wayne	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.2834
Longitude (W):	76° 59.7122

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	15.4		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	12.09	**Temperature (°C):**	11.95
Salinity (ppm):	0.309	**Salinity (ppm):**	0.305
Dissolved Oxygen (mg/L):	44.1	**Dissolved Oxygen (mg/L):**	42.0
Conductivity (µmhos/cm):	0.230	**Conductivity (µmhos/cm):**	0.229
pH:	8.10	**pH:**	7.98
Turbidity (NTU):	16.8	**Turbidity (NTU):**	20.4
ORP (mv):	213.3	**ORP (mv):**	217.9
Field Instrument Calibration Completed:			

Other Observations or Notes

brown silt, slight HC-like odor, trace organic material, no sheen, snail shells

Waypoint # 104

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-09
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-09
RETEC Project Name:	ESTCP	**Time of Collection:**	0916
Date Sample Collection:	11/8/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	Sunny, 30s	**Sampling Personnel:**	Rob, Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.2961
Longitude (W):	76° 59.7036

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	16		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.95	**Temperature (°C):**	11.96
Salinity (ppm):	0.305	**Salinity (ppm):**	0.305
Dissolved Oxygen (mg/L):	43.1	**Dissolved Oxygen (mg/L):**	42.0
Conductivity (µmhos/cm):	0.229	**Conductivity (µmhos/cm):**	0.229
pH:	7.93	**pH:**	7.86
Turbidity (NTU):	16.7	**Turbidity (NTU):**	21.2
ORP (mv):	211.3	**ORP (mv):**	211.2
Field Instrument Calibration Completed:			

Other Observations or Notes

Opposite side of Pier 1 from originally proposed location. Unable to get in to AR-09 location due to boom.

brown silt, dense org layer - leaves twigs sticks
visible black product/staining, slight NG-like odor, no sheen

Waypoint #103

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-10
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-10
RETEC Project Name:	ESTCP	**Time of Collection:**	0910
Date Sample Collection:	11/8/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	30s, sunny	**Sampling Personnel:**	Rob: Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.2905
Longitude (W):	76° 59.7567

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	17.5		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 11.95		**Temperature (°C):** 12.00	
Salinity (ppm): 0.305		**Salinity (ppm):** 0.305	
Dissolved Oxygen (mg/L): 44.1		**Dissolved Oxygen (mg/L):** 42.0	
Conductivity (µmhos/cm): 0.229		**Conductivity (µmhos/cm):** 0.229	
pH: 8.02		**pH:** 7.94	
Turbidity (NTU): 64.3		**Turbidity (NTU):** 20.2	
ORP (mv): 212.2		**ORP (mv):** 211.9	
Field Instrument Calibration Completed:			

Other Observations or Notes

Adjacent to end of Pier 1

brown silt, organic layer (leaves, sticks) visible black staining
slight HC-like odor, no sheen

N 38° 52.3183
W 76° 59.7587

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:** AR-11	Waypoint # 66
RETEC Project Number:	09000-422-100	**Sediment Sample:** AR-11	
RETEC Project Name:	ESTCP	**Time of Collection:** 1021	
Date Sample Collection:	11106107	**Sampling Personnel:** HA, HJ	AM
Weather:	Breezy, 40s, sunny	**Sampling Personnel:**	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3183
Longitude (W):	76° 59.7587

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	13.7		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	13.73	**Temperature (°C):**	13.70
Salinity (ppm): mS/cm³	0.297	**Salinity (ppm):**	0.294
Dissolved Oxygen (mg/L):	5.14	**Dissolved Oxygen (mg/L):**	38.0
Conductivity (~~µmhos/cm~~): MS/cm	~~152.2~~ 0.232	**Conductivity (µmhos/cm):**	0.232
pH:	8.05	**pH:**	7.83
Turbidity (NTU):	27.5	**Turbidity (NTU):**	26.3
ORP (mv):	152.2	**ORP (mv):**	160.1
Field Instrument Calibration Completed:	Came calibrated		

Other Observations or Notes

Next to dock, pier 2
Creosote treated wood
A lot of garbage under the pier

Brown silt w/ organic matter (leaves, sticks)
No HC-like odor, no sheen

Waypoint # 99

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-12
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-12
RETEC Project Name:	ESTCP	**Time of Collection:**	0847
Date Sample Collection:	11/8/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	Sunny, 30s	**Sampling Personnel:**	Rob; Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.2905
Longitude (W):	76° 59.8303

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	19.8		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.99	**Temperature (°C):**	12.06
Salinity (ppm):	0.310	**Salinity (ppm):**	0.305
Dissolved Oxygen (mg/L):	45.1	**Dissolved Oxygen (mg/L):**	43.1
Conductivity (µmhos/cm):	0.234	**Conductivity (µmhos/cm):**	0.229
pH:	8.83	**pH:**	8.54
Turbidity (NTU):	15.0	**Turbidity (NTU):**	17.2
ORP (mv):	216.7	**ORP (mv):**	215.9
Field Instrument Calibration Completed:			

Other Observations or Notes

Adjacent to stern of destroyer

brown silt, organic material - leaves, twigs

slight HC-like odor, no sheen

Waypoint #67

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:** AR-13	
RETEC Project Number:	09000-422-100	**Sediment Sample:** AR-13	
RETEC Project Name:	ESTCP	**Time of Collection:** 1047	
Date Sample Collection:	11/06/07	**Sampling Personnel:** HA, HJ, AM	
Weather: Sunny, breezy, 40s		**Sampling Personnel:**	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3336
Longitude (W):	76° 59.8676

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	13.4		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	13.77	**Temperature (°C):**	13.74
Salinity (ppm):	0.297	**Salinity (ppm):**	0.297
Dissolved Oxygen (mg/L):	6.76	**Dissolved Oxygen (mg/L):**	5.29
Conductivity (µmhos/cm):	0.233	**Conductivity (µmhos/cm):**	0.233
pH:	7.87	**pH:**	7.81
Turbidity (NTU):	23.5	**Turbidity (NTU):**	32.5
ORP (mv):	159.5	**ORP (mv):**	155.4
Field Instrument Calibration Completed:			

Other Observations or Notes

Plastic
Directly next to dock
No odor

silt, dark brown, heavy organic debris, twigs, leaves, shells
no HC-like odor,
no sheen

Waypoint # 68

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR14
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR14
RETEC Project Name:	ESTCP	**Time of Collection:**	1101
Date Sample Collection:	11/06/07	**Sampling Personnel:**	HA, HJ, AM
Weather: Sunny, breezy, 40s		**Sampling Personnel:**	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3201
Longitude (W):	76° 59.8713

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	17.6		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	13.75	**Temperature (°C):**	13.72
Salinity (ppm):	0.296	**Salinity (ppm):**	0.295
Dissolved Oxygen (mg/L):	10.7	**Dissolved Oxygen (mg/L):**	36.9
Conductivity (µmhos/cm):	0.233	**Conductivity (µmhos/cm):**	0.231
pH:	7.90	**pH:**	7.81
Turbidity (NTU):	23.9	**Turbidity (NTU):**	35.4
ORP (mv):	117.0	**ORP (mv):**	116.9
Field Instrument Calibration Completed:			

Other Observations or Notes

30 ft. out from dock
Further out from dock than figure shows

silt, dark brown, some organic debris, twigs, leaves, shells
trace HC-like odor, trace suspected black product by org. material
no sheen

Waypoint # 69

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:** AR-15	
RETEC Project Number:	09000-422-100	**Sediment Sample:** AR-15	
RETEC Project Name:	ESTCP	**Time of Collection:** 1116	
Date Sample Collection:	111607	**Sampling Personnel:** HA, HJ, AM	
Weather: 40s, sunny, breezy		**Sampling Personnel:** Rob & Wayne	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3486
Longitude (W):	76° 59.8691

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	13.8		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 13.84		**Temperature (°C):** 13.78	
Salinity (ppm): 0.300		**Salinity (ppm):** 0.301	
Dissolved Oxygen (mg/L): 7.34		**Dissolved Oxygen (mg/L):** 5.22	
Conductivity (µmhos/cm): 0.257		**Conductivity (µmhos/cm):** 0.236	
pH: 7.88		**pH:** 7.67	
Turbidity (NTU): 24.3		**Turbidity (NTU):** 1321.7	
ORP (mv): 127.9		**ORP (mv):** −84.6	
Field Instrument Calibration Completed:			

Other Observations or Notes

Right next to sea wall

dark brown silty clay moderate organic matter (sticks)
some trash - plastic, no sheen
slight HC-like odor trace suspected product

Waypoint # 73

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-16
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-16
RETEC Project Name:	ESTCP	**Time of Collection:**	1153
Date Sample Collection:	11/06/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	40s, Sunny, breezy	**Sampling Personnel:**	Rob & Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	~~38° 52.3415~~	38° 52.3816
Longitude (W):	~~76° 59.9079~~	76° 59.9360

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	14		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 13.97	~~13.83~~	**Temperature (°C):** 13.69	
Salinity (ppm): 0.302	~~0.295~~	**Salinity (ppm):** 0.295	
Dissolved Oxygen (mg/L): 5.26	~~39.8~~	**Dissolved Oxygen (mg/L):** 5.20	
Conductivity (µmhos/cm): 0.239	~~0.235~~	**Conductivity (µmhos/cm):** 0.231	
pH: 7.70	~~7.91~~	**pH:** 7.62	
Turbidity (NTU): 22.8	~~23.7~~	**Turbidity (NTU):** 29.6	
ORP (mv): 91.1	~~78.7~~	**ORP (mv):** 94.2	
Field Instrument Calibration Completed:			

Other Observations or Notes

No sample recovery
Moved to area further off sea wall
Tied to buoy & went off that for sample

silty clay, ~~little~~ lots of organic matter (sticks & leaves)
no HC-like odor, no sheen
dark brown, some suspected product

Waypoint # 77

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	Station Identification: AR-17	
RETEC Project Number:	09000-422-100	Sediment Sample: AR-17	
RETEC Project Name:	ESTCP	Time of Collection: 1505	
Date Sample Collection: 11/06/07		Sampling Personnel: HA, JH, and AM	
Weather: Sunny, breezy, 40s		Sampling Personnel: Rob & Wayne	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3636
Longitude (W):	76° 59.9417

Photographic Record

Photographic Description:		Photographic Description:	
Photographic Description:		Photographic Description:	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	19.1		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	13.69	Temperature (°C):	13.73
Salinity (ppm):	0.296	Salinity (ppm):	0.296
Dissolved Oxygen (mg/L):	39.0	Dissolved Oxygen (mg/L):	38.6
Conductivity (µmhos/cm):	0.233	Conductivity (µmhos/cm):	0.232
pH:	7.96	pH:	7.89
Turbidity (NTU):	25.6	Turbidity (NTU):	27.9
ORP (mv):	185.2	ORP (mv):	185.2
Field Instrument Calibration Completed:			

Other Observations or Notes

Directly off sea wall | against sea wall about 70 ft. west of outfall

brown silt, some organic matter (twigs, leaves, snail shells) plastic

trace org. odor, no sheen, suspected trace product - black staining

Waypoint #70

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:** AR-17	
RETEC Project Number:	09000-422-100	**Sediment Sample:** AR-17	
RETEC Project Name:	ESTCP	**Time of Collection:** 1125	
Date Sample Collection: 11/06/07		**Sampling Personnel:** HA, HJ, AM	
Weather: 40s, sunny, breezy		**Sampling Personnel:** Rob ; Wayne	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	CSO

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3553
Longitude (W):	76° 59.9011

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	9.2		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 13.98		**Temperature (°C):** 13.90	
Salinity (ppm): 0.299		**Salinity (ppm):** 0.300	
Dissolved Oxygen (mg/L): ~~0.2~~ 39.0		**Dissolved Oxygen (mg/L):** 38.0	
Conductivity (µmhos/cm): 0.238		**Conductivity (µmhos/cm):** 0.236	
pH: 7.66		**pH:** 7.60	
Turbidity (NTU): 28.5		**Turbidity (NTU):** 46.6	
ORP (mv): 21.1		**ORP (mv):** 29.9	
Field Instrument Calibration Completed:			

Other Observations or Notes

Right next to sea wall
Moved 15 ft off original location due to poor sample recovery. - still no recovery
Moved along sea wall towards AR-15 ~~38° 52.3553~~ (HA)

Waypoint #76

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	Station Identification:	AR-18
RETEC Project Number:	09000-422-100	Sediment Sample:	AR-18
RETEC Project Name:	ESTCP	Time of Collection:	1446
Date Sample Collection:	11/6/07	Sampling Personnel:	HA, HI, AM
Weather:	Sunny, breezy, 40s	Sampling Personnel:	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3411
Longitude (W):	76° 59.9679

Photographic Record

Photographic Description:		Photographic Description:	
Photographic Description:		Photographic Description:	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	19.3		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	13.67	Temperature (°C):	13.77
Salinity (ppm):	0.298	Salinity (ppm):	0.298
Dissolved Oxygen (mg/L):	40.0	Dissolved Oxygen (mg/L):	38.0
Conductivity (µmhos/cm):	0.234	Conductivity (µmhos/cm):	0.233
pH:	8.14	pH:	7.96
Turbidity (NTU):	22.2	Turbidity (NTU):	27.6
ORP (mv):	178.1	ORP (mv):	179.2
Field Instrument Calibration Completed:			

Other Observations or Notes

~100 ft off sea wall & 100 ft. west of buoy

brown silt + clay

slight HC-like odor, no sheen, trace organics,

Waypoint #75

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:** AR-19	
RETEC Project Number:	09000-422-100	**Sediment Sample:** AR-19	
RETEC Project Name:	ESTCP	**Time of Collection:** 1424	
Date Sample Collection: 11/06/07		**Sampling Personnel:** HA, HJ, AM	
Weather: 40s, sunny, breezy		**Sampling Personnel:** Rob, Wayne	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.355~~2~~
Longitude (W):	77° 00.028~~1~~

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	19.5		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 13.70		**Temperature (°C):**	13.65
Salinity (ppm): 0.295		**Salinity (ppm):**	0.294
Dissolved Oxygen (mg/L): 40.0		**Dissolved Oxygen (mg/L):**	38.6
Conductivity (µmhos/cm): 0.232		**Conductivity (µmhos/cm):**	0.238
pH: 8.27		**pH:**	8.03
Turbidity (NTU): 24.4		**Turbidity (NTU):**	55.7
ORP (mv): 172.9		**ORP (mv):**	153.8
Field Instrument Calibration Completed:			

Other Observations or Notes

Approximately 46 ft. off sea wall, to south of wall

dark brown silt, some suspected product
no HC-like odor, no sheen

Waypoint #82

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-20
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-20
RETEC Project Name:	ESTCP	**Time of Collection:**	0902
Date Sample Collection:	11/7/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	Sunny, 50s	**Sampling Personnel:**	Rob, Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.8016
Longitude (W):	76° 58.2947

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	9.2		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.56	**Temperature (°C):**	11.58
Salinity (ppm):	0.320	**Salinity (ppm):**	0.321
Dissolved Oxygen (mg/L):	42.0	**Dissolved Oxygen (mg/L):**	41.0
Conductivity (µmhos/cm):	0.238	**Conductivity (µmhos/cm):**	0.239
pH:	8.25	**pH:**	8.09
Turbidity (NTU):	22.4	**Turbidity (NTU):**	22.6
ORP (mv):	204.6	**ORP (mv):**	207.3
Field Instrument Calibration Completed:			

Other Observations or Notes

Middle of train bridge to the east

brown silt, dense organic layer - leaves, twigs
no HC-like odor, no sheen

Waypoint # 84

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	Station Identification:	AR-21
RETEC Project Number:	09000-422-100	Sediment Sample:	AR-21
RETEC Project Name:	ESTCP	Time of Collection:	0920
Date Sample Collection:	11/7/07	Sampling Personnel:	HA, HJ, AM
Weather:	Sunny, 50s	Sampling Personnel:	Rob, Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.6276
Longitude (W):	76° 58.8315

Photographic Record

Photographic Description:		Photographic Description:	
Photographic Description:		Photographic Description:	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	8.7		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.63	Temperature (°C):	11.86
Salinity (ppm):	0.306	Salinity (ppm):	0.306
Dissolved Oxygen (mg/L):	43.1	Dissolved Oxygen (mg/L):	41.0
Conductivity (µmhos/cm):	0.229	Conductivity (µmhos/cm):	0.228
pH:	7.88	pH:	7.67
Turbidity (NTU):	20.3	Turbidity (NTU):	180.5
ORP (mv):	204.9	ORP (mv):	73.4
Field Instrument Calibration Completed:			

Other Observations or Notes

East
West of bridge on north shore ~~off~~ of river
Near old outfall

slight HC-like odor, dense organic layer (peat, leaves, twigs), shells
brown silt, trace black staining, no sheen

Waypoint # 95

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-22
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-22
RETEC Project Name:	ESTCP	**Time of Collection:**	1134
Date Sample Collection:	11/7/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	50s, partly cloudy	**Sampling Personnel:**	Rob; Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3260
Longitude (W):	76° 59.4362

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	10		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.92	**Temperature (°C):**	12.02
Salinity (ppm):	0.304	**Salinity (ppm):**	0.308
Dissolved Oxygen (mg/L):	45.1	**Dissolved Oxygen (mg/L):**	41.0
Conductivity (µmhos/cm):	0.229	**Conductivity (µmhos/cm):**	0.232
pH:	7.78	**pH:**	7.70
Turbidity (NTU):	21.2	**Turbidity (NTU):**	137.6
ORP (mv):	151.4	**ORP (mv):**	150.8
Field Instrument Calibration Completed:			

Other Observations or Notes

brown silt (very fine), lots of organics (sticks, leaves)
no sheen, no odor, some trash

Waypoint # 96

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	Station Identification:	AR-23
RETEC Project Number:	09000-422-100	Sediment Sample:	AR-23
RETEC Project Name:	ESTCP	Time of Collection:	1300
Date Sample Collection:	11/7/07	Sampling Personnel:	HA, HJ, Am
Weather:	Partly cloudy, 50s	Sampling Personnel:	Rob, Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3102
Longitude (W):	76° 59.4329

Photographic Record

Photographic Description:		Photographic Description:	
Photographic Description:		Photographic Description:	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	12.5		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.92	Temperature (°C):	11.91
Salinity (ppm):	0.313	Salinity (ppm):	0.319
Dissolved Oxygen (mg/L):	44.1	Dissolved Oxygen (mg/L):	42.0
Conductivity (µmhos/cm):	0.235	Conductivity (µmhos/cm):	0.239
pH:	7.95	pH:	7.86
Turbidity (NTU):	19.1	Turbidity (NTU):	29.1
ORP (mv):	185.5	ORP (mv):	184.7
Field Instrument Calibration Completed:			

Other Observations or Notes

Under 11th Street bridge
Sheen on water in bucket

brown silt, trace organics - leaves, roots
no odor, no sheen

Waypoint # 89

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-24
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-24
RETEC Project Name:	ESTCP	**Time of Collection:**	0802
Date Sample Collection:	11/07/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	Sunny, 40s	**Sampling Personnel:**	Rob & Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.1553
Longitude (W):	76° 59.7653

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	18.7'		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	12.60	**Temperature (°C):**	12.68
Salinity (ppm):	0.306	**Salinity (ppm):**	0.305
Dissolved Oxygen (mg/L):	42.0	**Dissolved Oxygen (mg/L):**	42.0
Conductivity (µmhos/cm):	0.234	**Conductivity (µmhos/cm):**	0.233
pH:	8.31	**pH:**	8.19
Turbidity (NTU):	16.8	**Turbidity (NTU):**	18.0
ORP (mv):	164.5	**ORP (mv):**	168.6
Field Instrument Calibration Completed:			

Other Observations or Notes

Across river from experimental vessel a couple hundred feet from southern shore line

brown silt, slight HC-like odor, trace org. material - leaves, sticks.

visible black staining no sheen

Waypoint # 81

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-25
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-25
RETEC Project Name:	ESTCP	**Time of Collection:**	0816
Date Sample Collection:	11/7/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	Sunny, 40s	**Sampling Personnel:**	Rob & Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.1936
Longitude (W):	77° 00.2612

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	22.2		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 13.05		**Temperature (°C):** 13.16	
Salinity (ppm): 6.387		**Salinity (ppm):** 0.327	
Dissolved Oxygen (mg/L): 42.1		**Dissolved Oxygen (mg/L):** 42.10	
Conductivity (µmhos/cm): 0.261		**Conductivity (µmhos/cm):** 0.252	
pH: 8.16		**pH:** 7.99	
Turbidity (NTU): 15.9		**Turbidity (NTU):** 16.8	
ORP (mv): 183.7		**ORP (mv):** 185.2	
Field Instrument Calibration Completed:			

Other Observations or Notes

Across river from new Nationals stadium / brick building

brown silt, some suspected product (black)
slight HC-like odor, no sheen
trace organic material

Waypoint #78

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-26
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-26
RETEC Project Name:	ESTCP	**Time of Collection:**	1520
Date Sample Collection:	11/6/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	Sunny, breezy, 40s	**Sampling Personnel:**	Rob ; Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3346
Longitude (W):	76° 59.9135

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	16.8		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	13.71	**Temperature (°C):**	13.74
Salinity (ppm):	0.298	**Salinity (ppm):**	0.296
Dissolved Oxygen (mg/L):	40.0	**Dissolved Oxygen (mg/L):**	39.0
Conductivity (µmhos/cm):	0.234	**Conductivity (µmhos/cm):**	0.232
pH:	7.95	**pH:**	7.84
Turbidity (NTU):	25.7	**Turbidity (NTU):**	31.8
ORP (mv):	209.2	**ORP (mv):**	207.1
Field Instrument Calibration Completed:			

Other Observations or Notes

400 feet of buoy to the east / NE
Sheen on surface of water in sample bucket
Slight HCL odor

coarse sand, some silt, fine pebbles, brown, dense organics
trace large org. debris - sticks leaves, cobbles
suspected black product, trace sheen

Waypoint # 83

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	Station Identification:	AR-27
RETEC Project Number:	09000-422-100	Sediment Sample:	AR-27
RETEC Project Name:	ESTCP	Time of Collection:	0913
Date Sample Collection:		Sampling Personnel:	HA, HJ, AM
Weather:		Sampling Personnel:	Rob & Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.6513
Longitude (W):	76° 58.5209

Photographic Record

Photographic Description:		Photographic Description:	
Photographic Description:		Photographic Description:	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	5.5	
Type of Sediment Sampler Used:	Ponar	

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.75	Temperature (°C):	11.76
Salinity (ppm):	0.310	Salinity (ppm):	0.310
Dissolved Oxygen (mg/L):	41.0	Dissolved Oxygen (mg/L):	41.0
Conductivity (µmhos/cm):	0.232	Conductivity (µmhos/cm):	0.232
pH:	7.98	pH:	7.84
Turbidity (NTU):	21.2	Turbidity (NTU):	330.2
ORP (mv):	206.8	ORP (mv):	207.1
Field Instrument Calibration Completed:			

Other Observations or Notes

Approximately half way between AR-20 and AR-21 near marina

brown silt w/ little fine grained sand, no HC-like odor, no sheen

some org. material - leaves, sticks no sheen

Waypoint #85

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:** AR-28	
RETEC Project Number:	09000-422-100	**Sediment Sample:** AR-28	
RETEC Project Name:	ESTCP	**Time of Collection:** 1002	
Date Sample Collection:	111707	**Sampling Personnel:** HA, HJ, AM	
Weather:	50s, sunny	**Sampling Personnel:** Rob, Wayne	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.4841
Longitude (W):	76° 59.1255

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	10.9		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.69	**Temperature (°C):**	11.90
Salinity (ppm):	0.309	**Salinity (ppm):**	0.310
Dissolved Oxygen (mg/L):	44.1	**Dissolved Oxygen (mg/L):**	41.0
Conductivity (µmhos/cm):	0.232	**Conductivity (µmhos/cm):**	0.232
pH:	7.96	**pH:**	7.84
Turbidity (NTU):	20.6	**Turbidity (NTU):**	56.7
ORP (mv):	106.2	**ORP (mv):**	110.2
Field Instrument Calibration Completed:			

Other Observations or Notes

~1000 ft east of AR-01 near eastern power boat club.

brown silt, organic layer - leaves, twigs, no odor, no sheen

Waypoint # 86

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-29
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-29
RETEC Project Name:	ESTCP	**Time of Collection:**	1010
Date Sample Collection:	11/7/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	50s, sunny	**Sampling Personnel:**	Rob & Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.4515
Longitude (W):	76° 59.2358

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	11.6		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.75	**Temperature (°C):**	11.98
Salinity (ppm):	0.305	**Salinity (ppm):**	0.307
Dissolved Oxygen (mg/L):	44.1	**Dissolved Oxygen (mg/L):**	42.6
Conductivity (µmhos/cm):	0.229	**Conductivity (µmhos/cm):**	0.230
pH:	7.89	**pH:**	7.75
Turbidity (NTU):	21.6	**Turbidity (NTU):**	130.4
ORP (mv):	110.8	**ORP (mv):**	110.0
Field Instrument Calibration Completed:			

Other Observations or Notes

Approximately half way between AR-28 and AR-01 off N shore of river

brown silt, dense organic matter - leaves, twigs, shells
no HC-like odor, no sheen

Waypoint #90

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-30
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-30
RETEC Project Name:	ESTCP	**Time of Collection:**	1110
Date Sample Collection:	11/7/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	Sunny, 50s	**Sampling Personnel:**	Rob, Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3579
Longitude (W):	76° 59.2817

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	9.4		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.97	**Temperature (°C):**	11.98
Salinity (ppm):	0.308	**Salinity (ppm):**	0.309
Dissolved Oxygen (mg/L):	43.1	**Dissolved Oxygen (mg/L):**	41.0
Conductivity (µmhos/cm):	0.230	**Conductivity (µmhos/cm):**	0.233
pH:	7.92	**pH:**	7.85
Turbidity (NTU):	20.3	**Turbidity (NTU):**	28.7
ORP (mv):	169.4	**ORP (mv):**	160.0
Field Instrument Calibration Completed:			

Other Observations or Notes

Approximately half way between AR-01 & AR-02 in middle of river

brown clay - silty, trace organics - leaves, twigs
slight HC-like odor, no sheen

Waypoint # 91

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	Station Identification: AR-31	
RETEC Project Number:	09000-422-100	Sediment Sample: AR-31	
RETEC Project Name:	ESTCP	Time of Collection: 1125	
Date Sample Collection: 11/7/07		Sampling Personnel: HA, HJ, Am	
Weather: 50s, partly cloudy		Sampling Personnel: Rob, Wayne	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52. 3467
Longitude (W):	76° 59. 3343

Photographic Record

Photographic Description: P2		Photographic Description:	
Photographic Description:		Photographic Description:	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	13.7		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	11.91	Temperature (°C): 11.88	
Salinity (ppm):	0.308	Salinity (ppm): 0.312	
Dissolved Oxygen (mg/L):	43.1	Dissolved Oxygen (mg/L): 42.0	
Conductivity (µmhos/cm):	0.231	Conductivity (µmhos/cm): 0.234	
pH:	7.82	pH: 7.73	
Turbidity (NTU):	17.6	Turbidity (NTU): 24.4	
ORP (mv):	156.0	ORP (mv): 156.1	
Field Instrument Calibration Completed:			

Other Observations or Notes

~Half way between AR-02 and AR-03 about half way across the rivers.

brown silt, no odor, no sheen, trace fine grain sand

Waypoint # 100

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-32
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-32
RETEC Project Name:	ESTCP	**Time of Collection:**	0855
Date Sample Collection:	11/8/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	Sunny, 30s	**Sampling Personnel:**	Rob & Wayne

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.2429
Longitude (W):	76° 59.8043

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	17.5		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	12.05	**Temperature (°C):**	12.14
Salinity (ppm):	0.309	**Salinity (ppm):**	0.312
Dissolved Oxygen (mg/L):	45.1	**Dissolved Oxygen (mg/L):**	42.0
Conductivity (µmhos/cm):	0.233	**Conductivity (µmhos/cm):**	0.236
pH:	8.44	**pH:**	8.23
Turbidity (NTU):	16.1	**Turbidity (NTU):**	30.9
ORP (mv):	213.8	**ORP (mv):**	215.1
Field Instrument Calibration Completed:			

Other Observations or Notes

≈ 280 ft. South of stern of destroyer

brown silt, no organics, slight HC-like odor, no sheen, trace black staining /ps suspected product

Waypoint # 107

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:** AR-33	
RETEC Project Number:	09000-422-100	**Sediment Sample:** AR-33	
RETEC Project Name:	ESTCP	**Time of Collection:** 1009	
Date Sample Collection: 11/08/07		**Sampling Personnel:** HA, HJ, AM	
Weather: 40s, sunny		**Sampling Personnel:** RR, WS	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.2709
Longitude (W):	76° 59.8772

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	18.0		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 12.08		**Temperature (°C):** 11.98	
Salinity (ppm): 0.306		**Salinity (ppm):** 0.305	
Dissolved Oxygen (mg/L): 44.1		**Dissolved Oxygen (mg/L):** 41.0	
Conductivity (µmhos/cm): 0.230		**Conductivity (µmhos/cm):** 0.230	
pH: 7.70		**pH:** 7.64 ~~8.68~~ 7.64	
Turbidity (NTU): 14.8		**Turbidity (NTU):** 24.8	
ORP (mv): 54.5		**ORP (mv):** 61.0	
Field Instrument Calibration Completed:			

Other Observations or Notes

Approximately 350-400 off stern of ship to the ~~east~~ west

brown silt, slight HC-like odor, organic layer (leaves + twigs)
no sheen

Waypoint # 108

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:**	AR-34
RETEC Project Number:	09000-422-100	**Sediment Sample:**	AR-34
RETEC Project Name:	ESTCP	**Time of Collection:**	1013
Date Sample Collection:	11/08/07	**Sampling Personnel:**	HA, HJ, AM
Weather:	40s, sunny	**Sampling Personnel:**	RR, WS

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.2773
Longitude (W):	76° 59.9542

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	18.1		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)			Top Measurement (1 ft above sediment)	
Temperature (°C):	12.15		**Temperature (°C):**	12.06
Salinity (ppm):	0.309		**Salinity (ppm):**	0.306
Dissolved Oxygen (mg/L):	44.1		**Dissolved Oxygen (mg/L):**	42.0
Conductivity (µmhos/cm):	0.233		**Conductivity (µmhos/cm):**	0.230
pH:	7.75		**pH:**	7.66
Turbidity (NTU):	15.2		**Turbidity (NTU):**	22.2
ORP (mv):	74.6		**ORP (mv):**	79.6
Field Instrument Calibration Completed:				

Other Observations or Notes

South of buoy.

brown silt, slight HC-like odor, no sheen

Waypoint # 109

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	**Station Identification:** AR-35	
RETEC Project Number:	09000-422-100	**Sediment Sample:** AR-35	
RETEC Project Name:	ESTCP	**Time of Collection:** 1022	
Date Sample Collection: 11/08/07		**Sampling Personnel:** HA, HJ, AM	
Weather: 40s, sunny		**Sampling Personnel:** RB, WS	

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3484
Longitude (W):	77° 00.0015

Photographic Record

Photographic Description:		**Photographic Description:**	
Photographic Description:		**Photographic Description:**	

Field Measurements

Depth of Water (feet to nearest 0.1ft) 18.8		
Type of Sediment Sampler Used: Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C): 12.19		**Temperature (°C):** 12.12	
Salinity (ppm): 0.309		**Salinity (ppm):** 0.366	
Dissolved Oxygen (mg/L): 43.1		**Dissolved Oxygen (mg/L):** 41.0	
Conductivity (µmhos/cm): 0.234		**Conductivity (µmhos/cm):** 0.236	
pH: 7.71		**pH:** 7.64	
Turbidity (NTU): 14.5		**Turbidity (NTU):** 22.9	
ORP (mv): 92.3		**ORP (mv):** 95.4	
Field Instrument Calibration Completed:			

Other Observations or Notes

Between AR-18 and AR-19

brown silt, no odor, no sheen

Waypoint #110

SEDIMENT SAMPLING FIELD FORM

Sample Site:	Anacostia River	Station Identification:	AR-36
RETEC Project Number:	09000-422-100	Sediment Sample:	AR-36
RETEC Project Name:	ESTCP	Time of Collection:	1028
Date Sample Collection:	11/08/07	Sampling Personnel:	HA, HJ, AM
Weather:	40s, sunny	Sampling Personnel:	BR, WS

Study Area Observations

Depositional and Erosional Areas:	
Zones of Sediment Transport:	
Currents or Tides:	
On Shore Adjacent Facilities:	
Adjacent Storm Water Outfalls:	

GPS Station Coordinates (Degrees, Decimal Minutes NAD83)

Latitude (N):	38° 52.3416
Longitude (W):	77° 00.0925

Photographic Record

Photographic Description:		Photographic Description:	
Photographic Description:		Photographic Description:	

Field Measurements

Depth of Water (feet to nearest 0.1ft)	18.0		
Type of Sediment Sampler Used:	Ponar		

Water Quality Measurements

Top Measurement (1 ft below water surface)		Top Measurement (1 ft above sediment)	
Temperature (°C):	12.16	Temperature (°C):	12.16
Salinity (ppm):	0.310	Salinity (ppm):	0.307
Dissolved Oxygen (mg/L):	44.1	Dissolved Oxygen (mg/L):	42.0
Conductivity (µmhos/cm):	0.234	Conductivity (µmhos/cm):	0.232
pH:	7.76	pH:	7.66
Turbidity (NTU):	14.8	Turbidity (NTU):	19.1
ORP (mv):	101.2	ORP (mv):	103.2
Field Instrument Calibration Completed:			

Other Observations or Notes

South of Navy yard inlet, west of AR-19 ~400 ft

brown silt, trace organics, slight HC-like odor, no sheen

Appendix F: Chain of Custody Forms

Chain of Custody Record

№ 0331

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St.	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: FedEx
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: UMBC
Fax: 607-277-9057	

Analysis Requested: Carbon

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR12	11/8/07	0847	Sed	1		
AR32		0855				
AR08		0900				
AR10		0910				
AR09		0916				
AR06		0955				
AR07		1002				
AR33		1009				
AR34		1013				
AR35		1022				
AR36		1028				

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen A. Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level	Turnaround
Level I ☐	Routine ☐
Level II ☐	24 Hour ☐
Level III ☐	1 Week ☐
Other ☐	Other _____

Sample Receipt	
Total # Containers Received?	
COC Seals Present?	
COC Seals Intact?	
Received Containers Intact?	
Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

№ 0333

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: FedEx
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: ~~FedEx~~ ERDC
Fax: 607-277-9057	

Analysis Requested: H, 97 Hg

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR12	11/8/07	0847	Sed	2		
AR32		0855				
AR08		0900				
AR10		0910				
AR09		0916				
AR06		0955				
AR07		1002				
AR33		1009				
AR34		1013				
AR35		1022				
AR36		1028				

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen A. Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround		Sample Receipt	
Level I	☐	Routine	☐	Total # Containers Received?	
Level II	☐	24 Hour	☐	COC Seals Present?	
Level III	☐	1 Week	☐	COC Seals Intact?	
Other	☐	Other _____		Received Containers Intact?	
				Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

№ 0330

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St.	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: FedEx
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: EERC
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR12	11/8/07	0847	Sed	2		
AR32		0855				
AR08		0900				
AR10		0910				
AR09		0916				
AR06		0955				
AR07		1002				
AR33		1009				
AR34		1013				
AR35		1022				
AR36		1028				

Analysis Requested: Soxhlet PAH; Pore water PAH; SOC; TOC; DOC

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen A. Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround		Sample Receipt	
Level I	☐	Routine	☐	Total # Containers Received?	
Level II	☐	24 Hour	☐	COC Seals Present?	
Level III	☐	1 Week	☐	COC Seals Intact?	
Other	☐	Other _____		Received Containers Intact?	
				Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

№ 0332

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: FedEx
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: Test America
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Analysis Requested: grain size, conventionals, PIT, Solids	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR12	11/8/07	0847	Sed	2		Please hold until further notice	
AR32		0855					
AR08		0900					
AR10		0910					
AR09		0916					
AR06		0955					
AR07		1002					
AR33		1009					
AR34		1013					
AR35		1022					
AR36		1028					

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen A. Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround		Sample Receipt	
Level I	☐	Routine	☐	Total # Containers Received?	
Level II	☐	24 Hour	☐	COC Seals Present?	
Level III	☐	1 Week	☐	COC Seals Intact?	
Other	☐	Other _____		Received Containers Intact?	
				Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

No 0326

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jon
Address: 1001 W. Seneca St	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: Fed Ex
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: UMBC
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR24	11/7/07	0802	Sed	1		
AR25		0816				
AR20		0902				
AR27		0913				
AR21		0920				
AR28		1002				
AR29		1010				
AR01		1015				
AR02		1025				
AR03		1032				
AR30		1110				
AR31		1125				
AR22		1134				
AR23		1300				
AR04		1315				
AR05		1330				

Analysis Requested: Chlordane

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen A. Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround		Sample Receipt	
				Total # Containers Received?	
Level I	☐	Routine	☐	COC Seals Present?	
Level II	☐	24 Hour	☐	COC Seals Intact?	
Level III	☐	1 Week	☐	Received Containers Intact?	
Other	☐	Other _____		Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

№ 0328

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: FedEx
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: ERDC
Fax: 607-277-9057	

Analysis Requested: 17. Cations

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR24	11/7/07	0802	Sed	2		
AR25		0816				
AR20		0902				
AR27		0913				
AR21		0920				
AR28		1002				
AR29		1010				
AR01		1015				
AR02		1025				
AR03		1032				
AR30		1110				
AR31		1125				
AR22		1134				
AR23		1300				
AR04		1315				
AR05		1330				

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen A. Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround	
Level I	☐	Routine	☐
Level II	☐	24 Hour	☐
Level III	☐	1 Week	☐
Other	☐	Other _____	

Sample Receipt	
Total # Containers Received?	
COC Seals Present?	
COC Seals Intact?	
Received Containers Intact?	
Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

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www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St Suite 204 Ithaca NY 14850	Sampler (Print Name): H. Albert
	Shipment Method: FedEx
	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: EERC
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #:_____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Analysis Requested: Sorbent PAH / Porewater PAH / TOC / SOC / DOC	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR24	11/7/07	0802	sed	2			
AR25		0816					
AR20		0902					
AR27		0913					
AR21		0920					
AR28		1002					
AR29		1010					
AR01		1015					
AR02		1025					
AR03		1032					
AR30		1110					
AR31		1125					
AR22		1134					
AR23		1300					
AR04		1315					
AR05		1330					

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Helen d. Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround	
Level I	☐	Routine	☐
Level II	☐	24 Hour	☐
Level III	☐	1 Week	☐
Other	☐	Other _____	

Sample Receipt	
Total # Containers Received?	
COC Seals Present?	
COC Seals Intact?	
Received Containers Intact?	
Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

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Address: 1001 W. Seneca St	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: FedEx
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: Test America
Fax: 607-277-9057	

Page 1 of ____

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Analysis Requested	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR24	11/7/07	0802	Sed	2		please hold until further contact	
AR25		0816					
AR20		0902					
AR27		0913					
AR21		0920					
AR28		1002					
AR29		1010					
AR01		1015					
AR02		1025					
AR03		1032					
AR30		1110					
AR31		1125					
AR22		1134					
AR23		1300					
AR04		1315					
AR05		1330					

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Helen A. Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround		Sample Receipt	
				Total # Containers Received?	
Level I	☐	Routine	☐	COC Seals Present?	
Level II	☐	24 Hour	☐	COC Seals Intact?	
Level III	☐	1 Week	☐	Received Containers Intact?	
Other	☐	Other _____		Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

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Address: 1001 W. Seneca St.	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: FedEx
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: UMBC
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #:

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Analysis Requested: Cyber analysis	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR11	11/6/07	1021	Sed	1			
AR13		1047					
AR14		1101					
AR15		1116					
AR16		1153					
AR17		1505					
AR18		1446					
AR19		1424					
AR20		1520					

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Helen A Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround		Sample Receipt	
				Total # Containers Received?	
Level I	☐	Routine	☐	COC Seals Present?	
Level II	☐	24 Hour	☐	COC Seals Intact?	
Level III	☐	1 Week	☐	Received Containers Intact?	
Other	☐	Other _____		Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

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RETEC

Project Name: Anacostia River	Project Number: 09080-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: FedEx
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: ERSC
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Analysis Requested: Hg	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR11	11/6/07	1021	Sed	2			
AR13		1047					
AR14		1101					
AR15		1116					
AR16		1153					
AR17		1505					
AR18		1446					
AR19		1424					
AR26		1520					

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen A. Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround		Sample Receipt	
Level I	☐	Routine	☐	Total # Containers Received?	
Level II	☐	24 Hour	☐	COC Seals Present?	
Level III	☐	1 Week	☐	COC Seals Intact?	
Other	☐	Other _____		Received Containers Intact?	
				Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

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Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: Fed Ex
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: EERC
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Analysis Requested: Soxhlet PAH	Pore Water PAH	TOC	SOC	DOC	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR11	11/6/07	1021	sed	2							
AR13		1047									
AR14		1101									
AR15		1116									
AR16		1153									
AR17		1505									
AR18		1446									
AR19		1424									
AR26		1520									

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround		Sample Receipt	
				Total # Containers Received?	
Level I	☐	Routine	☐	COC Seals Present?	
Level II	☐	24 Hour	☐	COC Seals Intact?	
Level III	☐	1 Week	☐	Received Containers Intact?	
Other	☐	Other _____		Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

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Project Name: Anacostia River	Project Number: 09000-422-100
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Address: 1001 W. Seneca St	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: Fed Ex
Ithaca NY 14850	Airbill Number:
Phone: (607-277-5716	Laboratory Receiving: Test America
Fax: (607-277-9057	

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Analysis Requested: Grain Size	Ammonia	Soil pH	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR11	11/6/07	1021	Sed	2					
AR13		1047							
AR14		1101							
AR15		1116							
AR16		1153							
AR17		1505							
AR18		1446							
AR19		1424							
AR26		1520							

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen A. Jones			
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround		Sample Receipt	
Level I	☐	Routine	☐	Total # Containers Received?	
Level II	☐	24 Hour	☐	COC Seals Present?	
Level III	☐	1 Week	☐	COC Seals Intact?	
Other	☐	Other _____		Received Containers Intact?	
				Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Appendix G: Quality Assurance Project Plan (QAPP)

Quality Assurance Project Plan (QAPP)

Draft Demonstration Plan

The Determination of Sediment Polycyclic Aromatic Hydrocarbon (PAH) Bioavailability using Supercritical Fluid Extraction (SFE) and Ultra-Trace Porewater (UTP) analysis (ER-0709)

Washington Navy Yard – The Anacostia River



October 15, 2007

Table of Contents

1 Purpose 1-1

2 Project Description 2-1
2.1 Analytical Parameters 2-1

3 Project Organization and Responsibility 3-1
3.1 RETEC Project Manager 3-1
3.2 Project Geologist 3-1
3.3 Lead Field Scientist 3-2
3.4 Project Quality Assurance Manager 3-2
3.5 Laboratory Project Managers 3-2
3.6 Laboratory Quality Assurance Managers 3-2
3.7 Laboratory Managers (Commercial Laboratories) 3-3
3.8 Laboratory Director (Professional Research Scientist) 3-3

4 Data Quality Objectives for Measurement Data 4-1
4.1 Data Quality Levels 4-1
4.1.1 Field Testing Methods – Level I 4-1
4.1.2 Field Analysis Methods – Level II 4-1
4.1.3 Laboratory Methods – Level III 4-1
4.1.4 Laboratory Methods – Level IV 4-2
4.1.5 Laboratory Methods – Level V 4-2
4.2 Quality Control Parameters 4-2

5 Sampling Procedures 5-1

6 Sample Custody 6-1
6.1 Field Sample Custody Documentation 6-1
6.2 Laboratory Sample Custody Documentation 6-1

7 Analytical Procedures 7-1

8 Calibration Procedures and Frequency 8-1
8.1 Field Instrumentation 8-1
8.1.1 pH Meter 8-1
8.1.2 Conductivity and Salinity Meter 8-1
8.1.3 Dissolved Oxygen 8-1
8.1.4 Turbidimeter 8-1
8.1.5 Temperature 8-2
8.2 Laboratory Instrumentation Calibration 8-2

Table of Contents

9 Data Reduction, Assessment, and Reporting 9-1
9.1 Data Reduction 9-1
9.2 Data Assessment 9-2
9.2.1 Field Data Verification 9-2
9.2.2 Laboratory Peer Review 9-2
9.2.3 Independent Third-Party Data Validation 9-3
9.3 Data Reporting 9-3
9.3.1 Laboratory Reports 9-3
9.3.2 Data Reports and Document Retention 9-4
9.3.3 Electronic Data Deliverables 9-5

10 Quality Control Procedures 10-1
10.1 Field QC Checks 10-1
10.2 Internal Laboratory Organic and Inorganic QC Checks 10-1
10.3 Sediment Toxicity Test Quality Control 10-1

11 Performance and Systems Audits 11-1
11.1 Performance Audits 11-1
11.1.1 Laboratory Performance Audits 11-1
11.1.2 Field Performance Audits 11-1
11.2 Systems Audits 11-1
11.2.1 Laboratory Systems Audits 11-1
11.2.2 Field Systems Audits 11-1

12 Assessment Procedures for Data Usability 12-1
12.1 Precision 12-1
12.2 Accuracy 12-1
12.3 Representativeness 12-1
12.4 Completeness 12-2
12.5 Comparability (Method Compliance) 12-2
12.6 Criteria for Determining Data Usability 12-2

13 Preventive Maintenance 13-1

14 Corrective Action 14-1
14.1 Laboratory Corrective Action 14-1
14.2 Field Corrective Action 14-1

15 QA Reports to Management 15-1
15.1 Laboratory Quality Assurance Reports to Management 15-1
15.2 Project Quality Assurance Reports to Management 15-1

List of Appendices

Appendix 1: Analytical Methodology

Appendix 2: Sample Handling Requirements

Appendix 3: Quality Control and Detection Limits

1 Purpose

Data generated for the evaluation of the toxicity and bioavailability of PAHs must be technically sound and legally defensible, and supported by defined and verified limits of confidence. This document specifies the quality control and quality assurance procedures to ensure the generation of valid data for the evaluation of toxicity and bioavailability of PAHs. The following quality assurance project plan (QAPP) procedures are equivalent to those specified in the U.S. EPA's QA/R-5 "EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations" and its promulgated updates.

It is the responsibility of all personnel involved in this investigation to perform and document the required procedures designated within this document. This QAPP sets forth the data collection procedures and data evaluation process, which will ensure that appropriate levels of data quality are obtained throughout the PAH Toxicity and Bioavailability Evaluation Program.

At the completion of data collection, data evaluation is to be conducted and data compiled and statistically evaluated. The primary goal of this project is to evaluate potential toxicity of sediments to aquatic life and improve predictions of PAH bioavailability and toxicity to benthic aquatic organisms.

2 Project Description

This project is to assess the bioavailability and toxicity of the polycyclic aromatic hydrocarbons (PAHs) in aquatic sediments as part of the ESTCP project The Determination of Sediment Polycyclic Aromatic Hydrocarbon (PAH) Bioavailability using Ultra-Trace Pore water (UTP) analysis (ER-0709). This project is designed to build upon the information and database for freshwater sediments that has been initiated by Niagara Mohawk Power Corporation (NMPC), Alcoa, and the Northeast Gas Association (NGA).

The primary goal of this project is to evaluate the toxicity of aquatic sediments for remedial decision-making and to evaluate the feasibility of estimating PAH bioavailability as a means to improve predictions of PAH toxicity to benthic aquatic organisms.

The following parameters have been designated for baseline measurement and on-going laboratory analysis. A complete list of the laboratory analyses and method references is presented in Appendix 1, Table A1-1.

2.1 Analytical Parameters

- Total sediment Parent and Alkylated PAH Determinations by GC/MS
- Pore Water Parent and Alkylated PAH Determinations by GC/MS
- Total and Soot Organic Carbon
- Grain Size Distribution
- Ammonia Nitrogen
- Acid-Soluble Sulfides
- Amphipod (*Hyallela azteca*) 28-Day Chronic Toxicity Test
- Surface Water Field Tests: pH, temperature, conductivity, dissolved oxygen (DO), and turbidity

The exact number, locations, and rationale for each sample and analytical parameters selected are provided in the main text of the dem/val plan.

3 Project Organization and Responsibility

This section details the responsibilities of key personnel with respect to sediment sample collection, sample analysis, data reporting and validation, and record keeping in support of the PAH Bioavailability Evaluation.

3.1 RETEC Project Manager

The Project Manager will be the primary point of contact and will have primary responsibility for technical, financial, and scheduling matters for the PAH Bioavailabilty Evaluation. Duties will include:

- Procuring and supervising subcontractor services, including reviewing of subcontract work and approving of subcontract invoices;
- Assigning duties to the project staff and orientating the staff to the needs and requirements of the project as they relate to the project objectives;
- Preparing schedules for the completion of each investigation phase;
- Reviewing all major project deliverables for technical accuracy and completeness;
- Closing out the project; and
- Maintaining the project files.

3.2 Project Geologist

The Project Geologist will act as the field team leader and be responsible for the supervision of field surveys, collection and shipping of environmental samples for analytical testing, and peer review of field data. Duties will include:

- Coordinating field related activities with the Project Manager;
- Supervising the collection of samples and providing for their proper documentation, handling, and shipment;
- Overseeing the field sampling operations to verify that the sampling team members adhere to the QAPP and/or the field sampling Standard Operating Procedures (SOPs), as required; and
- Preparing, certifying, and reporting field investigation data.

3.3 Lead Field Scientist

The Lead Field Scientist will be responsible for field activities and field record keeping, including:

- Coordinating field related activities with the Project Geologist;
- Collecting samples and providing for their proper documentation, handling, and shipment; and
- Preparing and reporting field sample collection records and field measurement data.

3.4 Project Quality Assurance Manager

The Project Quality Assurance Manager will be responsible for quality assurance oversight of the project including:

- Serving as a resource for information and data quality objectives for the project;
- Serving as a liaison between the field and data interpretation personnel and the laboratories; and
- Performing third-party validation for the project data.

3.5 Laboratory Project Managers

The Laboratory Project Managers, associated with detailed chemical characterization and toxicity testing are responsible for all facets of laboratory analysis and reporting, including the following:

- Conducting an initial review of the data to provide a data package case narrative that describes any nonconformance and its impact on data quality;
- Validating of the laboratory data per the laboratory QA/QC plan;
- Reviewing the data package to ensure data is transcribed correctly and that a complete and correct data package is reported to the user, and
- Serving as liaison between the laboratory and project personnel.

3.6 Laboratory Quality Assurance Managers

The Laboratory Quality Assurance (QA) Managers associated with the chemical and toxicity testing laboratories are responsible for adherence to the laboratory QA objectives

as defined within this QAPP. The Laboratory QA Managers act independently of the personnel performing analyses. Responsibilities include:

- Writing, maintaining, and implementing laboratory analytical and administrative SOPs;
- Conducting laboratory performance and systems audits on a scheduled basis; and
- Initiating system corrective action and verifying that the corrective action appropriately resolves the problem(s).

3.7 Laboratory Managers (Commercial Laboratories)

The Laboratory Managers associated with commercial laboratories are ultimately responsible for all aspects of the analytical services provided for this project. The responsibilities of the Laboratory Managers include:

- Maintaining adequate staff and instrumentation to support this project;
- Directing manpower and resources to support project data quality objectives;
- Reviewing and releasing laboratory data reports; and
- Coordinating and managing laboratory activities.

3.8 Laboratory Director (Professional Research Scientist)

The Laboratory Director of the Energy & Environmental Research Center of the University of North Dakota is a professional research scientist. As such, the director performs the analyses and is responsible for all aspects of quality assurance, quality control, and data reporting.

4 Data Quality Objectives for Measurement Data

Data quality objectives are qualitative and quantitative statements to ensure that data of known and appropriate quality are obtained during the investigation. The quality of the data must be sufficient to fulfill the overall objective of the specific investigation activity. These objectives will be further defined in the work plan. The work plan will further identify the constituents of interest, required limits of detection, intended use of the data, and associated data deliverables. The detection and quality control limits for the organic and inorganic parameters are presented in Appendix 3 of this QAPP.

4.1 Data Quality Levels

There are several analytical levels of data quality available to achieve the data quality objectives. These levels are typically designated as follows:

- **Level I** field screening or analysis using portable instruments, calibrated to non-compound specific standards;
- **Level II** field analysis using portable instruments, calibrated to specific compounds;
- **Level III** EPA OSWER Recommended Performance-Based Methodologies such as those outlined in U. S. EPA SW-846;
- **Level IV** US EPA CLP Routine Analytical Services (RAS) methods; and,
- **Level V** Other internationally recognized and/or non-standard analytical methods.

The PAH Toxicity and Bioavailability Evaluation will utilize levels I, III, and V as described above.

4.1.1 Field Testing Methods – Level I

A YSI field meter or equivalent will be used to measure pH, temperature, salinity, conductivity, and dissolved oxygen (DO), and a Lamott 2020 meter or equivalent will be used to measure turbidity, of surface water samples in the field.

4.1.2 Field Analysis Methods – Level II

No level II procedures are planned for the PAH Toxicity and Bioavailability Evaluation.

4.1.3 Laboratory Methods – Level III

Level III data documents the precision, accuracy, and defensibility of the data. A higher degree of quality control is integral to this level of testing. Standardized toxicity and chemical analyses will be conducted on screened sediment samples, including pH, grain

size, ammonia, percent solids, and dissolved TOC. The method descriptions, modifications, and references are presented in Appendix 1 of this QAPP.

4.1.4 Laboratory Methods – Level IV

No level IV procedures are planned for the PAH Toxicity and Bioavailability Evaluation.

4.1.5 Laboratory Methods – Level V

The investigative nature of this Evaluation requires the use of several non-standard analytical procedures developed and validated in a university research laboratory setting.

The following detailed, non-standard analyses will be performed.

- Total PAHs consisting of 34 parent and alkylated compounds for each chosen sediment using GC/MS technology in selected ion monitoring (SIM) mode.

- Pore water samples will be generated in the laboratory from the selected sediment samples and analyzed for PAHs following centrifugation, flocculation, and extraction using solid-phase microextraction and GC/MS SIM analysis.

The method descriptions and published literature references are presented in Appendix 1 of this QAPP.

4.2 Quality Control Parameters

For the organic and inorganic analyses, the basic quantitative criteria used to evaluate data quality (precision and accuracy) for the sediment and pore water samples are presented in Appendix 3, Tables A3-3 and A3-4.

Toxicity laboratory internal laboratory QC checks will be performed as described in the laboratory's standard operating procedures to ensure that measurement systems are in-control and that daily activities that affect the quality of the final data are carried out in accordance with the overall quality assurance program. The toxicity testing QC checks are summarized in Section 10.3

5 Sampling Procedures

For each major measurement parameter and measurement system, a description of the sampling procedures to be used shall be provided in the work plan. The following specific information is outlined in the work plan:

- Techniques and guidelines for the selection of sampling sites;
- Procedures for specific sampling techniques and field activities to be used;
- Outline of sampling program operations;
- Description of containers, procedures, holding times, reagents, etc., used for sample collection, preservation, transport, and storage;
- Procedures for field and laboratory chain-of-custody (COC); and
- Procedures, forms, and notebooks to be used to record sample history, sampling conditions, and analyses to be performed.

Appendix 2 of this QAPP lists the general container, preservation, and holding time requirements for the analyses to be performed. Section 6.0 of this QAPP outlines the general field and laboratory sample custody procedures.

6 Sample Custody

The primary objective of sample custody is to create an accurate, written, verifiable record, which can be used to trace the possession and handling of the samples from the moment of collection through data analysis and reporting. A sample is under a person's custody if:

1. It is in that person's possession;

2. It is in that person's view, after being in that person's possession;

3. It was in that person's possession and subsequently was placed in a secure area by that person; or

4. It is in a designated secure area.

6.1 Field Sample Custody Documentation

Field samples shall be maintained so that sample integrity is not jeopardized. Documentation records of sample custody shall be maintained. The Project Geologist and Lead Field Scientist will be responsible for the care and custody of the samples from collection until they are properly transferred or dispatched. A completed COC record will always be included in the sample-shipping container. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the COC record.

6.2 Laboratory Sample Custody Documentation

Upon arrival at the laboratory, samples will be logged-in by the sample receiving department utilizing either a hardcopy or a computerized sample log-in format. The laboratory shall log-in the samples and assign tests according to the following procedures, at a minimum. The laboratory shall contact the Project Manager as soon as possible should any deficiency be noted.

1. The sample-receiving group will first examine whether the shipping container is intact or broken.

2. The sample-receiving group will open the shipping container upon receipt and inspect the contents to determine if any sample containers have been damaged during shipping. For samples to be shipped on ice at 4° C ± 2° C, upon receipt by the laboratory, the temperature of the sample containers will be measured and the temperature recorded on the COC.

3. If samples have been damaged during shipment, the remaining samples shall be carefully examined to determine whether they were affected. It will be noted on

the COC that specific samples were damaged or affected and whether the samples were removed from the sampling program. The Project Manager will determine the appropriate course of action for any sample considered damaged or suspect.

4. The sample-receiving group will compare the samples received against those listed on the COC and verify the sample identification information, and verify that holding times have not been exceeded. The required holding times for sample analyses for this project are presented on Appendix 2 of this document.

5. The sample-receiving group will then sign and date the COC and attach any courier bill to the COC.

Upon verification of sample receipt at the laboratory, the sample-receiving group will assign a unique laboratory identification number to the sample for entry into their laboratory tracking system. This allows for a uniform format for easier sample tracking. Each laboratory has a unique format for assigning identification numbers.

Once samples have been logged-in and transferred to the proper storage areas, the laboratory will be responsible for the protection of sample integrity and implementation and documentation of the appropriate level of internal COC program, as specified in the laboratory quality assurance manual, or research laboratory practice and policy.

7 Analytical Procedures

A laboratory, capable of providing reliable data that meets the data quality objectives stated in the site-specific work plan, shall perform all analyses. The specific analytical procedures and the modifications required are described in Appendix 1. Where applicable, analyses shall be performed using the following U. S. EPA–approved and/or nationally-recognized analytical references:

- "Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), U. S. EPA Office of Solid Waste and Emergency Response, Third Edition," 1992, and subsequent updates;

- American Society for Testing Materials (ASTM), "Soil and Rock", Volume 04.08, Philadelphia , PA, 1994;

- "Standard Methods for the Examination of Water and Wastewater," 19th edition, Eaton, A. D. Clesceri, L.S. Greenberg, A. E. American Water Works Association, Water Pollution Control Federation, American public Health Association: Washington D. C., 1995;

- "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates," second edition, U. S. EPA Office of Research and Development, Duluth, MN, EPA 600/R-99/064, March 2000;

- U. S. EPA Contract Laboratory Program Statement of Work (CLP SOW), OLM04.2/4.3 and ILM05.2; and

- "Method for Chemical Analysis of Water and Wastes," U. S. EPA EPA-600/4-79-020, revised March 1983.

Several analyses require analytical methods outside the scope to the references cited above. The non-standard testing procedures are described in Appendix 1 and in the following published research literature:

- Total Organic Carbon (TOC)

  "PAH Release during Water Desorption, Supercritical Carbon Dioxide Extraction, and Field Bioremediation," Hawthorne, Steven B., et al., Environmental Science & Technology, Vol. 35, No. 22, 2001.

- Soot Organic Carbon (SOC)

"Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability," Gustofson, Orjan, et al., Environmental Science & Technology, Vol. 31, No. 1, 1997.

- Total PAH Measurements of Sediments

  "Procedures for the Derivation of ESBs for the Protection of Benthic Organisms: PAH Mixtures," EPA/600/R-02/013, Office of Research and Development, Washington D. C., 2003.

  "Measurement of "Total" PAH Concentrations and Toxic Units Used for Sediment Risk Assessment at Manufactured Gas Plant Sites," Hawthorne, Steven B., et al., 2005b, Energy & Environmental Research Center, University of North Dakota, Grand Forks, ND. Environmental Toxicology & Chemistry, Accepted for publication July 20, 2005.

  "Sampling and Analytical Methods of the National Status and Trends Program. Mussel Watch Project: 1993-1996," U. S. Department of Commerce, 1998, Update, NOAA Technical Memorandum NOS ORCA 130, National Oceanic and Atmospheric Administration, Silver Spring, MD.

- Pore water PAH Determinations

  "Solid-Phase Microextraction Measurement of Parent and Alkyl Polycyclic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values," Hawthorne, Steven B., et al., 2005a, Environmental Science & Technology, 39: 2795-2803.

  "Measurement of "Total" PAH Concentrations and Toxic Units Used for Sediment Risk Assessment at Manufactured Gas Plant Sites," Hawthorne, Steven B., et al., 2005b, Energy & Environmental Research Center, University of North Dakota, Grand Forks, ND. Environmental Toxicology & Chemistry, Accepted for publication July 20, 2005.

  "Sampling and Analytical Methods of the National Status and Trends Program. Mussel Watch Project: 1993-1996," U. S. Department of Commerce, 1998, Update, NOAA Technical Memorandum NOS ORCA 130, National Oceanic and Atmospheric Administration, Silver Spring, MD.

The Project Manager shall ensure that laboratories generating data in support of the PAH Bioavailability Evaluation maintain the relevant government regulatory accreditations, certifications, and/or registrations to perform the required analyses.

8 Calibration Procedures and Frequency

All field and laboratory equipment will be calibrated prior to use to ensure optimal operating conditions, according to the applicable SOPs or published literature references. The applicable quality control requirements will be defined within the field and laboratory quality assurance manuals and/or SOPs. Research laboratories will utilize the procedures in the published literature references in Appendix 1.

8.1 Field Instrumentation

All field instrumentation shall be calibrated or verified under laboratory conditions prior to field use utilizing manufacturer's recommended operating procedures. Daily performance checks shall be conducted prior to the start of each sampling day. Whether individual or multi-parameter measurement instrument systems are used, calibrations must be recorded in a field-sampling logbook or on a field instrument calibration sheet.

8.1.1 pH Meter

The initial calibration is performed with two standard buffer solutions, typically having pH 4.0 and pH 10.0. The calibration is checked initially and after every ten samples, and at the end of the day, with the pH 7.0 buffer. Should a field check against the 7.0 buffer fall outside normal acceptance criteria; the meter is returned to the vendor for service and recalibration.

8.1.2 Conductivity and Salinity Meter

The meter is checked prior to use with an outside calibration standard provided by the vendor or manufacturer. If the standard is not within 10% of the true value, the instrument is not in calibration; the meter is returned to the vendor for service and recalibration.

8.1.3 Dissolved Oxygen

The dissolved oxygen probe is calibrated against saturated moist air within the laboratory and corrected for current ambient barometric and temperature conditions. Since these conditions present a variable in the field, no further action is taken until the unit is returned to the laboratory where a post- calibration is performed.

8.1.4 Turbidimeter

The turbidimeter is calibrated daily against known gel standards (1, 10 and 100 NTU) and checked periodically against a gel standard (1 NTU) contained within the field kit. Should a field check against the gel standard fall outside normal acceptance criteria, the meter is returned to the vendor for service and recalibration.

8.1.5 Temperature

The temperature is generated by thermocouple response within the unit and is not user-adjustable. Should the check against a mercury thermometer fall outside normal acceptance criteria, the meter is returned to the vendor for service and recalibration.

8.2 Laboratory Instrumentation Calibration

Calibrations will be established as per instrument manufacturers' instructions or as specified by the specific analytical method or published literature references. The laboratory quality assurance manual and all analytical SOPs and/or literature references will be retained on file by the Laboratory Project Manager.

9 Data Reduction, Assessment, and Reporting

Data reduction, evaluation, transfer, and support are essential functions in summarizing information to support conclusions. It is essential that these processes are performed accurately and, in the case of data reduction, accepted statistical techniques are used. The procedures to be used and information necessary to meet project requirements are outlined below.

9.1 Data Reduction

For most analyses, data reduction involves the comparison of samples to a standard reference curve. Samples (or extracts) must be analyzed within the concentration range of the calibration curve. Sometimes, by diluting the sample to bring that constituent of highest concentration within the concentration range of the curve, other constituents of interest may be diluted out of the reportable concentration range. Typically, these data are reported as "not detected" at an elevated detection limit. For this project, all constituents of interest must be analyzed and reported within an appropriate concentration range to report the detected concentrations of all constituents of interest, or to report them as not detected at the practical quantitation limit (PQL) of an undiluted sample. This may require that the laboratory prepare, analyze, and report the results from more than one dilution. Non-detected values above the PQL of the analytical method are unacceptable unless due to matrix interference, or to prevent saturation of the detector. Also, if a constituent concentration is not detected at the PQL, the laboratory must compare the raw data to the calculated method detection limit (MDL). For standard tests, detected concentrations between the MDL and PQL will be reported as estimated (i.e., "J") concentrations and will be qualified as such by the laboratory. For the non-standard PAH analyses, concentrations below the lowest calibration standard with a signal to noise ratio of 3:1 or greater, will be reported as estimated (i.e., "J") concentrations.

When estimating the average response peak in alkyl PAH clusters, the final average peak may exceed the calibration range (i.e., qualified "E"). The PQL for parent and alkyl PAHs is defined as one-thirty-fourth of a Toxic Unit. For all PAH measurements, the determination as to whether a "J," or an "E" qualifier should be reported is based on the parent compound calibration (adjusted for the difference in relative response factor) where the alkylated daughter PAH was not in the calibration standard mix.

Results from analyses that do not make use of a standard curve are calculated by the appropriate formula given in the method, taking the number of significant figures into account.

The objective of the toxicity testing is to compare growth and survival (H. azteca) between site and reference sediments with great enough statistical power (i.e., $\geq$ 0.8) to render the results scientifically defensible. Data will be analyzed for normality and homogeneity of variances. If the data are normal with equal variance then the data will

be analyzed by ANOVA followed by means comparisons to the reference response. If the data are not normal or unequal variances are observed, data will be transformed. If data transformation fails to render the data normal or with homogenous variance then an ANOVA on ranks will be conducted followed by a means comparison of site and reference sediment responses. Survival data will be arc-sine square root transformed prior to analysis.

9.2 Data Assessment

The quality of all data will be assessed prior to its intended end use. Data are assessed on three levels. First, during the field operations, the Project Geologist will assess field measurements at the time of collection by verifying the use of standard operating procedures for the sampling effort and evaluating field quality control checks. Second, all laboratory analytical records and final results will undergo a peer review by the laboratory section supervisor or qualified designee, other than the analyst who performed the analysis under review. Third, the final data package will be validated by a third-party (i.e., Project Quality Assurance Manager) who is independent of the laboratory using U. S. EPA guidelines and method specifications. Problems with the data report(s) such as omissions or transcription errors are resolved with the laboratory prior to submittal of the final data validation report.

9.2.1 Field Data Verification

Verification of field data, as well as ongoing review of environmental sample collection records is performed in the field. All field data is reviewed during the time of collection and is later subjected to on-site peer review. Care will be taken to ensure that correct sample identifications, units, sample locations, and other pertinent information are included and correct. Any inconsistencies discovered will be resolved immediately, when possible. Corrections to field data sheets or field logbooks will be made by placing a single line through the entry, and initialing and dating the correction. If information is added without a correction being necessary, that entry will be initialed and dated to indicate that it was not entered at the original time of data entry.

The Project Geologist is responsible for ensuring that accurate and correct data and representative samples are obtained by following field investigation procedures as they are described in the work plan to achieve work objectives. It is important that the Project Geologist ensures that the field team adheres to the approved work plan and follows QA/QC measures as outlined in this document, sampling according to standard operating procedures, and collection of sufficient sample volume.

9.2.2 Laboratory Peer Review

Each laboratory group will provide for peer review of the data prior to reporting according to the procedures in the laboratory's quality assurance manual.

9.2.3 Independent Third-Party Data Validation

The Project Quality Assurance Manager will validate the data using method specifications and the following guidelines:

- "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," EPA 540/R-99/008, October 1999, and
- "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," EPA 540-R-04-004, October 2004.

To accomplish this review, laboratory data packages will include the elements specified in Section 9.3.1 as they apply to the objectives defined in the work plan.

9.3 Data Reporting

Data reporting includes laboratory reports submitted to the Project Manager. The Project Manager will direct the distribution of the data for validation, further interpretation, and subsequent reporting. Section 9.3.1 specifies the laboratory data deliverables that will satisfy the requirements for third-party data validation. Section 9.3.2 specifies the reporting format that the Laboratory Managers will follow for report submission. Section 9.3.3 specifies the formatting requirements for electronic data deliverables.

9.3.1 Laboratory Reports

The complete laboratory reports will be submitted to the Project Manager. The laboratory reports will include the following at a minimum, unless otherwise specified in the work plan:

For all data (organic and inorganic):

- A descriptive case narrative identifying any problems encountered during sample receipt, log-in, preparation, and analysis of the samples;
- Completed and legible COC records for all samples contained within each data package;
- A sample chronicle indicating which analyses were requested and performed for the samples contained in the data package;
- A summary of the laboratory sample identifications and the correlating field sample identifications; and
- Analytical summary reports for each sample with results, complete sample identifications, the sample dilutions (if necessary), and the individual sample preparation and analysis dates.

The following specific information will be reported for organic constituents:

- Analytical results;
- Surrogate recoveries (Soxhlet);
- Replicate calibration response (SPME); and
- Method blank summaries.

The following specific data will be reported for inorganic constituents:

- Analytical results;
- ICVs and CCVs;
- Reporting Limit Standards;
- ICBs and CCBs;
- Method Preparation Blanks;
- Matrix Spike Recoveries;
- Laboratory Duplicates;
- Laboratory Control Sample;
- Preparation logs; and
- Run logs.

In addition, the following data deliverables will be provided for the toxicity testing:

- A statistical determination as to whether there was reduced survival compared to the field reference sample control treatments; and reduced growth.

- The standard package it will consist of a report containing a description of the work conducted and results (including statistics) in tabular format. The statistics reported will be the mean comparison results.

9.3.2 Data Reports and Document Retention

Upon receipt of the laboratory data reports, the Project Manager will direct the distribution of the data for validation, further interpretation, and subsequent reporting. The Project Manager shall maintain project data files for a period of seven years that include the following:

- All laboratory data and QA/QC information;

- All field survey data, sample collection data and geospatial data; and

- All data interpretation, field notes, electronic data files, interim project reports and final project reports.

9.3.3 Electronic Data Deliverables

Laboratories performing analytical work must supply the data in an Access-compatible format.

10 Quality Control Procedures

Quality control (QC) procedures and checks are used to assess the precision and accuracy of analytical data. Field QC checks are used to identify potential problems associated with sample handling and sampling procedures. Laboratory QC checks are used to identify potential problems associated with sample preparation and analysis.

10.1 Field QC Checks

Given the rigorous sample homogenization and sieving procedure that will be used following collection of each sediment sample, field duplicate and field blank samples will not be collected for this project.

10.2 Internal Laboratory Organic and Inorganic QC Checks

Internal laboratory QC checks will be performed as described in the laboratory's quality assurance manual or peer-reviewed published literature to ensure that measurement systems are in-control. The QC checks may include some or all of the following:

- Initial and continuing calibration verifications;
- Preparation blanks, instrument blanks, and calibration blanks;
- Matrix spikes and matrix spike duplicates, and laboratory duplicates;
- Surrogate spikes;
- Laboratory control standards;
- Performance evaluation samples; and
- Calibration check standards.

10.3 Sediment Toxicity Test Quality Control

Toxicity laboratory internal laboratory QC checks will be performed as described in the laboratory's standard operating procedures to ensure that measurement systems are in-control and that daily activities that affect the quality of the final data are carried out in accordance with the overall quality assurance program. The QC checks will include the following:

- Water quality for culturing and testing will be tested for chemical and physical characteristics to ensure quality and uniformity;
- Cultures will be tested using reference toxicant(s) to assess organism health, demonstrate laboratory performance and reproducibility in performing the test, and

comparability of the laboratory's organism strain sensitivity to that of other laboratories;

- Test replicates will be prepared and tested to ensure an adequate level of sensitivity to meet the objectives of the test and the statistical method used for analysis of the data. Six or more replicates will be prepared for the 28-d amphipod tests; and

- Control treatments will be analyzed to provide confidence that the toxicity is due to the PAH toxicant and that there is no other cause.

  One or more field reference samples will be collected and submitted for the preparation of toxicity test controls, a fine-grained sediment, and a coarse-grained sediment. The negative control will contain no PAH toxicant in the fine-grained treatment. The PAH positive control treatments will be prepared by adding a PAH toxicant to both the fine and coarse-grained treatments.

11 Performance and Systems Audits

Two types of audit procedures are conducted during any environmental investigation: performance and systems audits. Performance and systems audits are required to monitor the capability and performance of the internal and external measurement systems.

11.1 Performance Audits

11.1.1 Laboratory Performance Audits

The Laboratory Quality Assurance Manager conducts internal laboratory performance audits on a periodic basis (e.g., quarterly). Each laboratory analyst is given a single or double-blind performance evaluation sample containing analytes of known concentration for the method(s) that he/she was trained to perform. The audit sample results are used to monitor accuracy and identify and resolve problems in sample preparation and analysis techniques, which lead to the generation of nonconforming data.

These criteria apply to commercial laboratories. Research laboratories are not required to perform internal or external performance audits.

11.1.2 Field Performance Audits

The Project Geologist and Lead Field Scientist shall provide direct supervision of field sampling activities. Field performance audits will not be required.

11.2 Systems Audits

11.2.1 Laboratory Systems Audits

Systems audits are used to ensure that all aspects of the laboratory's quality control program are being implemented and are effective. This involves a review of laboratory practices and documentation to confirm that work can be performed according to regulatory and project specifications. The Laboratory Quality Assurance Managers shall conduct periodic (e.g., annual) systems audits against their internal analytical and administrative SOPs and quality manual.

This criteria applies to commercial laboratories. Research laboratories are not required to perform internal or external systems audits.

11.2.2 Field Systems Audits

The Project Geologist and Lead Field Scientist shall provide direct supervision of field sampling activities. Field systems audits will not be required.

12 Assessment Procedures for Data Usability

The following discussion describes the data quality indicators that will be assessed to determine data quality. Data validation will be performed on the laboratory data to assess precision, accuracy, representativeness, completeness, and comparability (method compliance) of the sample data to determine if it is adequate for its intended use. The reporting limits and quality control limits for this project are summarized in Appendix 3 and Section 10.3.

12.1 Precision

Precision is the measure of variability of individual sample measurements. Evaluation of laboratory duplicates for precision is done using the Relative Percent Difference (RPD) or Percent Difference (%D). The RPD is defined as the difference between two duplicate samples divided by the mean and expressed as a percent. RPD and %D limits reference specification limits, or laboratory control quality control (QC) limits.

12.2 Accuracy

Laboratory accuracy, a measure of the system bias, is measured by evaluating laboratory blanks, laboratory control sample/laboratory control sample duplicate (LCS/LCSD), matrix spike/matrix spike duplicate (MS/MSD), reporting limit standards, and organic system monitoring compounds (surrogate) percent recoveries (%Rs). The analysis of laboratory blanks determines the existence and magnitude of any contamination resulting from laboratory activities. LCS/LCSD %Rs, which demonstrate the overall performance of the analytical procedure, are compared to published or recommended QC limits. MS/MSD %Rs, which provide information on the effects of sample matrix interferences, are compared to advisory limits. The reporting limit standard %R verifies the calibration (sensitivity) near the detection limit. System monitoring compounds or surrogate recoveries, which measure system performance and extraction efficiency during organic analysis, are compared to specification limits or laboratory control limits.

12.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of population, parameter variations at a sampling point, a process condition, or an environmental condition within a defined spatial and/or temporal boundary.

In the project-planning stage, an understanding was developed of the scale over which statistical comparisons would occur, what uncertainties were to be resolved, and a data generation plan was established. Sampling will be performed according to standard operating procedures and analyses will be performed using U. S. EPA-approved and research laboratory methodology. However, matrix interferences in the sample analyses may be encountered that must be taken into account when making statistical comparisons.

12.4 Completeness

Completeness is the overall ratio of the number of sample analyses planned versus the number of samples with valid analyses. The completeness goal is set at greater than or equal to 95%. Determination of completeness includes a review of COC records, laboratory analytical methods and detection limits, laboratory case narratives, and project requirements. Completeness also includes a 100% review of the laboratory sample data results, QC summary reports, and electronic data deliverables (EDDs).

12.5 Comparability (Method Compliance)

Comparability is an expression of the confidence with which one data set can be compared to another. U. S. EPA-approved methodology or methods developed in a research laboratory setting will be used throughout the project. Method compliance will be determined by evaluating sample integrity, holding times, system performance checks, initial and continuing instrument calibrations, internal standards, and target analyte identification, and quantitation against method specifications. Sample integrity and holding time are evaluated to ensure that the sample condition is acceptable and that the analyses are completed within a timeframe conducive to the analytes being measured. Organic system performance checks (tunes) are performed to ensure mass resolution and proper identification of target analytes. Instrument calibration requirements are established to measure system performance throughout the analytical procedure and to establish the quantitation criteria for the target analytes. GC/MS internal standard performance criteria ensure that instrument sensitivity and response are stable during every analytical run.

12.6 Criteria for Determining Data Usability

The data quality objectives (DQOs) specified in the work plan will be used as criteria for determining the usability of the data. All data from the sampling events will be assessed using the validation criteria set forth in the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," EPA/R-99/008, October 1999 and, "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," EPA 540-R-04-004, October 2004 as they apply to the methods used.

13 Preventive Maintenance

Preventive maintenance is required for equipment whose performance can affect analytical results and critical time schedules. A supply and list of critical spare parts and consumable materials, and a schedule of important preventive maintenance tasks shall be maintained to minimize downtime. The contract laboratory shall document the specific protocols for the operation and maintenance of laboratory instrumentation. Maintenance shall be documented for major equipment.

For research laboratories, maintenance will be performed on a routine basis so that the GC/MS tuning criteria are met based on the manufacturer's instructions.

Associated field sampling instrumentation shall be maintained in accordance with manufacturers' recommendations or field sampling SOPs.

14 Corrective Action

Corrective action is imperative and implemented whenever a systems deficiency or performance failure is detected. Corrective measures are deemed adequate when they prevent recurrence of the problem.

14.1 Laboratory Corrective Action

A standard operating procedure shall be established to provide for the initiation, documentation and ultimate closure of corrective action issues within the laboratory. The procedure shall include:

- Predetermined limits for data acceptability, beyond which corrective action is initiated;
- A procedure for implementation and documentation of corrective action; and
- Identification of the staff members responsible for initiating corrective action and also the individual responsible for approving the corrective action.

For research laboratories, corrective action is initiated and conducted by the individual performing the analysis as needed.

14.2 Field Corrective Action

Field corrective action is initiated and conducted by any individual performing or supervising field activities. Ultimately, the Lead Field Scientist or Project Geologist must inform the Project Manager (verbally or in a written report) of the problem(s) encountered and the measures taken, if any, to solve them. The Project Manager documents the details and files the report(s) in the project files. The Project Manager or designee is responsible for periodic follow-ups to ensure that corrective measures are complete and effective.

15 QA Reports to Management

15.1 Laboratory Quality Assurance Reports to Management

The laboratory QA Manual must require periodic reporting to management on the effectiveness of quality systems, performance of measurement systems, and data quality.

15.2 Project Quality Assurance Reports to Management

The Project Quality Assurance Manager shall provide validation reports on data quality to the Project Manager. In addition, the Project Geologist and the Lead Field Scientist shall report on field activities to the Project Manager. The reports shall include all aspects that have potential liability issues or adversely affect the achievement of project objectives.

The report shall include but not be limited to:

- A discussion of laboratory and field data quality;
- A summary of major problems encountered at the Site and the corrective measures taken to prevent recurrence;
- Significant recurring problems or trends that may require global corrective measures; and
- Recommended or on-going solutions to issues uncovered during management of the PAH toxicity and Bioavailability Evaluation Program.

Appendix 1:
Laboratory Methods

PARAMETER	METHOD	METHOD REFERENCE(S)	LABORATORY[1]
Total PAH extraction and analysis – screening level	Sonication for 4 hrs. in 1:1 methylene chloride:acetone mixture followed by GC/MS analysis.	[Hawthorne, 2005b], [U.S. EPA EPA/600/R-02/013, 2003] , [NOAA, 1998]	EERC
Total PAHs parent & alkylated (34 compounds)	Sohxlet extraction in methylene chloride: followed by GC/MS using Selected Ion Monitoring (SIM) for measuring parent and alkylated PAHs	[Hawthorne, 2005b], [U.S. EPA EPA/600/R-02/013, 2003] , [[NOAA, 1998]	EERC
PAH extraction – pore water	Centrifugation and flocculation followed by solid phase microextraction and GC/MS analysis for measuring parent and estimating alkylated PAHs	[Hawthorne et al., 2005a], [Hawthorne, 2005b], [NOAA, 1998]	EERC
Total Organic Carbon (TOC)	Sample acidified to remove carbonates, followed by analysis using a Leeman CE44 Elemental Analyzer modified for sediment analysis. (triplicate analyses on sediments)	[Hawthorne, 2001]	EERC
Soot Organic Carbon (SOC)	Heat stable TOC following pretreatment at 375º C for 24 hrs. modified for sediment	[Gustafsson et al., 1997]	EERC
Percent Solids	STL SOP IN623	[U.S. EPA, 1983], [U.S. EPA, CLP SOW OLM04.2/4.3], [U.S. EPA, CLP SOW ILM05.2]	STL
Grain Size	ASTM Methods D422-63 and D421-85	[ASTM, 1990]	STL
pH in Sediment	U.S. EPA SW-846 Method 9045C	[U.S. EPA, 1996]	STL
Ammonia	U.S. EPA Method 350.2	[U.S. EPA, 1983]	STL
Sediment toxicity test	Amphipod (*H. azteca*) toxicity 28-day chronic test	[U.S. EPA, 2000]	ERDC

Note 1: Laboratories

EERC – Energy & Environmental Research Center, ND
STL – Severn Trent Laboratories
ERDC – Engineering Research and Development Center

Appendix 2:
Sample Handling Requirements

Sample Type	Matrix	Parameter	Qty	Container Type[1]	Minimum Volume	Preservation [3]	Holding Time from Sample Date	Laboratory [4]
Toxicity Tests	Sediment	Amphipod Toxicity (*H. azteca*) 28-day chronic test	15	glass	2 L	Cool to 4° C	28 days[2]	ERDC
Chemical/ Physical Characterization	Pore Water	Dissolved pore water PAHs (parent cmpds and estimate of alkylated)	15	(5)	(5)	Cool to 4° C	28 days[2]	EERC
	Sediment	Total Organic Carbon	15	Glass	8 oz.		28 days[2]	
		Soot Carbon	15				28 days[2]	
		Total sediment PAHs parent & alkylated (34 compounds)	15				28 days[2]	
		Percent Solids	15	Glass	2 oz.	Cool to 4° C	28 days[2]	STL
		Grain Size	15	Glass	16 oz.		NA	
		pH	15	glass	8 oz.	Cool to 4° C	ASAP	
		Ammonia	15				28 days[2]	
Surface Water	Surface Water	pH, Temperature, Conductivity, Salinity, DO, Turbidity	15	field	field	field	15 min.	Field

Notes: (1) All glass jars must have Teflon-lined lids
(2) Test to be initiated within 28 days of sample collection.
(3) Samples requiring thermal preservation must be maintained at 2º - 6º C.
(4) EERC-Energy & Environmental Research Center
ERDC – Engineering Research & Development Center
STL Severn Trent Laboratories
(5) Pore water samples are generated in the EERC laboratory from the sediment samples.

Appendix 3:
Quality Control and Detection Limits

Table A3-1: Pore Water and Sediment Detection Limits – Organic Parameters

PAHs	Method	Pore Water Target Detection Limits[1] (ug/L)	Pore Water Actual Detection Limits[3] (ug/L)	Sediment Target Detection Limits[1,2] (mg/Kg dry wt)	Sediment Actual Detection Limits[3] (mg/Kg dry wt)
Naphthalene	GC/MS	5.7	0.500	0.11	0.001
2-Methylnaphthalene	GC/MS	2.4	0.200	0.13	0.001
C1 naphthalenes	GC/MS	2.4	0.200	0.13	0.001
C2 naphthalenes	GC/MS	0.89	0.600	0.15	0.005
C3 naphthalenes	GC/MS	0.33	0.300	0.17	0.010
C4 naphthalenes	GC/MS	0.12	0.050	0.19	0.010
Acenaphthylene	GC/MS	9.0	0.100	0.13	0.001
Acenaphthene	GC/MS	1.6	0.050	0.14	0.001
Fluorene	GC/MS	1.2	0.050	0.16	0.001
C1 fluorenes	GC/MS	0.41	0.100	0.18	0.005
C2 fluorenes	GC/MS	0.16	0.100	0.20	0.01
C3 fluorenes	GC/MS	0.06	0.050	0.23	0.030
Phenanthrene	GC/MS	0.56	0.200	0.18	0.001
Anthracene	GC/MS	0.61	0.050	0.17	0.001
C1 phenanthrenes/anthracenes	GC/MS	0.22	0.200	0.20	0.005
C2 phenanthrenes/anthracenes	GC/MS	0.09	0.050	0.22	0.010
C3 phenanthrenes/anthracenes	GC/MS	0.04	0.020	0.24	0.020
C4 phenanthrenes/anthracenes	GC/MS	0.02	0.020	0.27	0.030
Fluoranthene	GC/MS	0.21	0.040	0.21	0.001
Pyrene	GC/MS	0.30	0.040	0.21	0.001
C1 pyrene/fluoranthenes	GC/MS	0.14	0.050	0.23	0.005
Benz(a)anthracene	GC/MS	0.066	0.010	0.25	0.002
Chrysene	GC/MS	0.060	0.010	0.25	0.002
C1 benz(a)anthracene/chrysenes	GC/MS	0.025	0.020	0.27	0.010
C2 benz(a)anthracene/chrysenes	GC/MS	0.014	0.008	0.30	0.030
C3 benz(a)anthracene/chrysenes	GC/MS	0.005	0.008	0.33	0.050
C4 benz(a)anthracene/chrysenes	GC/MS	0.002	0.008	0.36	0.080
Benzo(b & k)fluoranthene[4]	GC/MS	0.019	0.010	0.29	0.002
Benzo(a)pyrene	GC/MS	0.028	0.005	0.28	0.002
Benzo(e)pyrene	GC/MS	0.026	0.005	0.28	0.002

Perylene	GC/MS	0.026	0.005	0.28	0.002
Indeno(1,2,3-cd)pyrene	GC/MS	0.008	0.002	0.33	0.002
Dibenz(a,h)anthracene	GC/MS	0.008	0.002	0.33	0.002
Benzo(g,h,i)perylene	GC/MS	0.013	0.002	0.32	0.002

Notes:

(1) Target detection limits for both sediment and pore water are 1/34th of the concentration of each individual PAH that corresponds to one toxic unit as described in U. S. EPA (2003) *Equilibrium Partitioning Sediment Guidelines (ESGs) for the Protection of Benthic Organisms*

(2) Sediment target detection limits were estimated assuming 1.0 % total organic carbon.

(3) Actual laboratory detection limit for sediments and pore water are based on previous research studies. Final detection limits will vary based analyte levels and matrix interferences.

(4) Benzo[b]fluoranthene and benzo[k]fluoranthene are reported as their sum because of insufficient chromatographic resolution.

Table A3-2: Sediment Method Detection Limits – General Chemistry

Analyte	Method	Reporting Limit[1]
Ammonia-Nitrogen	350.2	2.4 mg/Kg
pH of Sediment	9045C	0.1 S U.
Acid-Soluble Sulfide	9030B/9034	8.0 mg/Kg
Total Organic Carbon	See Table A1-1	NA
Soot Organic Carbon	See Table A1-1	NA

Notes:

(1) Final Reporting Limits are adjusted for aliquot weight, dilution, and percent solids.

Table A3-3: Quality Control Criteria – Organic Parameters

Quality Control Check	Total PAHs	Pore Water PAHs
GC/MS Tuning Criteria	As per manufacturer's instructions using PFTBA. Must pass criteria prior to analysis of each sample set or every 24 hrs.	As per manufacturer's instructions using PFTBA. Must pass criteria prior to analysis of each sample set or every 24 hrs.
Calibrations	Daily three-point initial calibration established with each sample set. The RSD must be less than 30% for high molecular weight PAHs and less than 25% for low molecular weight PAHs, and the $r^2 > 0.99$. The calibration curve must not be forced through the origin. (1)	Replicate calibration standards are analyzed at a low level with each sample set. The RSDs must be less than 25% for high molecular weight PAHs and less than 20% for low molecular weight PAHs. (2)
Multiple Analyses	Quadruplicate samples are prepared and analyzed. Statistical outliers are rejected and additional analyses may be necessary. Final results are an average.	Quadruplicate samples are prepared and analyzed. Statistical outliers are rejected and additional analyses may be necessary. Final results are an average.
Method Preparation Blanks	A solvent blank is prepared with each sediment sample (one blank for each set of four	Analyzed between every sample to monitor the baseline. Should high background levels be

	replicates of every sediment sample). Should the PAH concentrations in the blank be greater than 20% of the sample concentrations, the sample set is rejected except in cases where the sample results are < PQL.	detected, analysis does not proceed.
Internal Standards	Deuterated parent PAHs, representing each molecular ring size, are spiked into every sample as an analytical calibration aid.	Deuterated parent PAHs, representing each molecular ring size, are spiked into every sample as an analytical calibration aid.
Surrogates and System Monitoring Compounds	Benzo(b)fluoranthene-d12 and biphenyl-d10 are spiked into the sediment samples prior to Soxhlet extraction to monitor extraction efficiency. Surrogates must recover 70-120%.	Replicate calibration standards are analyzed at a low level with each sample set. The RSDs must be less than 25% for high molecular weight PAHs and less than 20% for low molecular weight PAHs. (2)

Note: The PAH screening will determine the relative levels of PAHs and the data will be used to choose the samples for further detailed analysis only.

(1) The calibration will not be reestablished in response to a nonconforming RSD if the sample results are less than the PQL.

(2) A single point calibration is established. The response factor from the daily calibration standards is used to quantitate the results. The quality of the low-level peak responses will be assessed to ensure a signal to noise ratio greater than 3:1.

Table A3-4: Quality Control Criteria – Organic Parameters

Analyte	Method	Precision RPD (%)	Accuracy (%Recovery)
Ammonia-Nitrogen	350.2	20	90-110
pH of Sediment	9045C	20	90-110
Acid-Soluble Sulfide	9030B/9034	20	75-125
Total Organic Carbon	See Table A1-1	Triplicates[1]	NA
Soot Organic Carbon	See Table A1-1	Triplicates[1]	NA

Notes:
(1) Triplicate samples are analyzed. Statistical outliers are rejected. Final results are an average.

Appendix H: General Sediment Physical and Chemical Properties

TestAmerica
South Burlington, VT

Sample Data Summary Package

SDG: 123121

Case Narrative **1**

Chain of Custody **3**

Sample Data Summary Wet Chemistry **7**

Sample Data Summary Geotechnical **27**

Sample Handling **44**

Last Page of this Document **49**



Case Narrative

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

December 14, 2007

Mr. Nick Azzolina
ENSR International
1001 W. Seneca Street, Suite 204
Ithaca, NY 14850-3342

Re: Laboratory Project No. 27000
Case: ANACOST; SDG: 123121

Dear Mr. Azzolina:

Enclosed are analytical results for samples that were received by TestAmerica Burlington on November 7th, November 8th, and November 9th 2007. Laboratory identification numbers were assigned, and designated as follows:

Lab ID	Client Sample ID	Date Released For Analysis	Sample Date	Sample Matrix
		ETR No: 123121		
733049	AR11		11/06/07	SEDIMENT
733050	AR13	11/13/07	11/06/07	SEDIMENT
733051	AR14	11/13/07	11/06/07	SEDIMENT
733051DP	AR14REP	11/13/07	11/06/07	SEDIMENT
733051MD	AR14MSD	11/13/07	11/06/07	SEDIMENT
733052	AR15		11/06/07	SEDIMENT
733053	AR16	11/13/07	11/06/07	SEDIMENT
733054	AR17	11/13/07	11/06/07	SEDIMENT
733055	AR18		11/06/07	SEDIMENT
733056	AR19		11/06/07	SEDIMENT
733057	AR26	11/13/07	11/06/07	SEDIMENT
733058	AR24		11/07/07	SEDIMENT
733059	AR25	11/13/07	11/07/07	SEDIMENT
733060	AR20		11/07/07	SEDIMENT
733061	AR27	11/13/07	11/07/07	SEDIMENT
733062	AR21	11/13/07	11/07/07	SEDIMENT
733063	AR28		11/07/07	SEDIMENT
733064	AR29		11/07/07	SEDIMENT
733065	AR01		11/07/07	SEDIMENT

30 Community Drive, Suite 11 South Burlington, VT 05403 **tel** 802.660.1990 **fax** 802.660.1919 **www.testamericainc.com**

Lab ID	Client Sample ID	Date Released For Analysis	Sample Date	Sample Matrix
		ETR No: 123121 (Continued)		
733066	AR02	11/13/07	11/07/07	SEDIMENT
733067	AR03	11/13/07	11/07/07	SEDIMENT
733068	AR30		11/07/07	SEDIMENT
733069	AR31		11/07/07	SEDIMENT
733070	AR22		11/07/07	SEDIMENT
733071	AR23		11/07/07	SEDIMENT
733072	AR04		11/07/07	SEDIMENT
733073	AR05	11/13/07	11/07/07	SEDIMENT
733074	AR12		11/08/07	SEDIMENT
733075	AR32		11/08/07	SEDIMENT
733076	AR08	11/13/07	11/08/07	SEDIMENT
733077	AR10	11/13/07	11/08/07	SEDIMENT
733078	AR09		11/08/07	SEDIMENT
733079	AR06		11/08/07	SEDIMENT
733080	AR07	11/13/07	11/08/07	SEDIMENT
733081	AR33		11/08/07	SEDIMENT
733082	AR34		11/08/07	SEDIMENT
733083	AR35		11/08/07	SEDIMENT
733084	AR36	11/13/07	11/08/07	SEDIMENT

Documentation of the condition of the samples at the time of their receipt and any exception to the laboratory's Sample Acceptance Policy is documented in the Sample Handling section of this submittal.

Particle size determinations were performed on each sample by ASTM D422-63. Additionally, each sample was analyzed for ammonia nitrogen by USEPA Method 350.2, and for pH by SW846 Method 9045C. Matrix spike and replicate analyses were performed on sample AR14 specific to ammonia. The matrix spike analysis did yield a relatively high recovery of ammonia (117 percent). The laboratory does use an upper control limit of 115 percent in the assessment of recovery performance. There was an acceptable correlation of the results of the replicate analyses in the interanalysis comparison. Replicate analyses were also performed on sample AR14 specific to the particle size determination and pH, and, for each set of replicate analyses, there was an acceptable correspondence in the results. Laboratory control samples were analyzed in association with the samples, and there was an acceptable demonstrated performance in each those analyses. The method blanks associated with the analytical work were free of analyte contamination.

The analytical results associated with the samples presented in this test report were generated under a quality system that adheres to requirements specified in the NELAC standard. Release of the data in this test report and any associated electronic deliverables is authorized by the Laboratory Director's designee as verified by the following signature.

If there are any questions regarding this submittal, please contact me at 802 660-1990.

Sincerely,

Kirk F. Young
Project Manager

KFY/hsf
Enclosure

TestAmerica Burlington Data Qualifier Definitions

Organic

U: Compound analyzed but not detected at a concentration above the reporting limit.

J: Estimated value.

N: Indicates presumptive evidence of a compound. This flag is used only for tentatively identified compounds (TICs) where the identification of a compound is based on a mass spectral library search.

P: SW-846: Greater than 40% difference for detected concentrations between two GC columns. Unless otherwise specified the higher of the two values is reported on the Form I.

CLP SOW: Greater than 25% difference for detected concentrations between two GC columns. Unless otherwise specified the lower of the two values is reported on the Form I.

C: Pesticide result whose identification has been confirmed by GC/MS.

B: Analyte is found in the sample and the associated method blank. The flag is used for tentatively identified compounds as well as positively identified compounds.

E: Compounds whose concentrations exceed the upper limit of the calibration range of the instrument for that specific analysis.

D: Concentrations identified from analysis of the sample at a secondary dilution.

A: Tentatively identified compound is a suspected aldol condensation product.

X,Y,Z: Laboratory defined flags that may be used alone or combined, as needed. If used, the description of the flag is defined in the project narrative.

Inorganic/Metals

E: Reported value is estimated due to the presence of interference.

N: Matrix spike sample recovery is not within control limits.

*: Duplicate sample analysis is not within control limits.

B: The result reported is less than the reporting limit but greater than the instrument detection limit.

U: Analyte was analyzed for but not detected above the reporting limit.

Method Codes:

P	ICP-AES
MS	ICP-MS
CV	Cold Vapor AA
AS	Semi-Automated Spectrophotometric

FQA009:04.24.06:3
TestAmerica Burlington



Chain of Custody

Chain of Custody Record

No 0367

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: FedEx
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: Test America
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #: _____

Analysis Requested: Grain Size; Ammonia; Soil pH

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR11	11/6/07	1021	Sed	2		
AR13		1047				
AR14		1101				
AR15		1116				
AR16		1153				
AR17		1505				
AR18		1446				
AR19		1424				
AR26		1520				

Relinquished by: (Signature) Helen A. Jones	Received by: (Signature)	Date: 11/7/07	Time: 0930
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround	
Level I	☐	Routine	☑
Level II	☐	24 Hour	☐
Level III	☐	1 Week	☐
Other	☐	Other _____	

Sample Receipt	
Total # Containers Received?	3 JEC 11/7/07 3 9 18
COC Seals Present?	Yes
COC Seals Intact?	Yes
Received Containers Intact? AR13	NO
Temperature?	5.5 °C

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

4

Chain of Custody Record

No 0327

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St Suite 204 Ithaca NY 14850	Sampler (Print Name): H. Albert
	Shipment Method: FedEx
	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: Test America
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Analysis Requested: grain size, ammonia, pH, solids	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR24	11/7/07	0802	Sed	2		please hold until further contact	
AR25		0816					
AR20		0902					
AR27		0913					
AR21		0920					
AR28		1002					
AR29		1010					
AR01		1015					
AR02		1025					
AR03		1032					
AR30		1110					
AR31		1125					
AR22		1134					
AR23		1300					
AR04		1315					
AR05		1330					

Relinquished by: (Signature) Helen A. Jones	Received by: (Signature)	Date: 11/8/07	Time: 0930
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround	
Level I	☐	Routine	☑
Level II	☐	24 Hour	☐
Level III	☐	1 Week	☐
Other	☐	Other ____	

Sample Receipt	
Total # Containers Received?	32
COC Seals Present?	Yes
COC Seals Intact?	Yes
Received Containers Intact?	Yes
Temperature?	5.8°C

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

No 0332

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St Suite 204 Ithaca NY 14850	Sampler (Print Name): H. Albert
	Shipment Method: FedEx
	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: Test America
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #:

Analysis Requested: Trace Size, Cyanide, PM, Solids

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR12	11/8/07	0847	Sed	2	please hold until further notice	
AR32		0855				
AR08		0900				
AR10		0910				
AR09		0916				
AR06		0955				
AR07		1002				
AR33		1009				
AR34		1013				
AR35		1022				
AR36		1028				

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen A. Jones		11/9/07	0930
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level		Turnaround	
Level I	☐	Routine	☐
Level II	☐	24 Hour	☐
Level III	☐	1 Week	☐
Other	☐	Other ____	

Sample Receipt	
Total # Containers Received?	22
COC Seals Present?	Yes
COC Seals Intact?	Yes
Received Containers Intact?	Yes
Temperature?	4.4°C

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Sample Data Summary – Wet Chemistry

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR13

Lab Name: TAL BURLINGTON | Contract: 2055227 | SDG No.: 123121

Lab Code: TALVT | Case No.: ANACOST | Lab Sample ID: 733050

Matrix: SEDIMENT | Client: ENSCON | Date Received: 11/07/07

% Solids: 33.8

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	2	55.8	595	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	6.5	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		33.8	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR14

Lab Name: TAL BURLINGTON
Contract: 2055227
SDG No.: 123121

Lab Code: TALVT
Case No.: ANACOST
Lab Sample ID: 733051

Matrix: SEDIMENT
Client: ENSCON
Date Received: 11/07/07

% Solids: 35.4

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	26.7	271	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	6.8	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		35.4	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR16

Lab Name: TAL BURLINGTON | Contract: 2055227 | SDG No.: 123121

Lab Code: TALVT | Case No.: ANACOST | Lab Sample ID: 733053

Matrix: SEDIMENT | Client: ENSCON | Date Received: 11/07/07

% Solids: 43.1

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	21.9	136	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	6.9	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		43.1	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR17

Lab Name: TAL BURLINGTON | **Contract:** 2055227 | **SDG No.:** 123121

Lab Code: TALVT | **Case No.:** ANACOST | **Lab Sample ID:** 733054

Matrix: SEDIMENT | **Client:** ENSCON | **Date Received:** 11/07/07

% Solids: 34.4

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	29.1	161	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	7.1	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		34.4	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR26

Lab Name: TAL BURLINGTON | **Contract:** 2055227 | **SDG No.:** 123121

Lab Code: TALVT | **Case No.:** ANACOST | **Lab Sample ID:** 733057

Matrix: SEDIMENT | **Client:** ENSCON | **Date Received:** 11/07/07

% Solids: 48.7

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	19.7	123	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	7.1	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		48.7	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR25

Lab Name: TAL BURLINGTON | Contract: 2055227 | SDG No.: 123121

Lab Code: TALVT | Case No.: ANACOST | Lab Sample ID: 733059

Matrix: SEDIMENT | Client: ENSCON | Date Received: 11/07/07

% Solids: 38.4

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	25.0	408	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	6.9	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		38.4	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR27

Lab Name: TAL BURLINGTON **Contract:** 2055227 **SDG No.:** 123121

Lab Code: TALVT **Case No.:** ANACOST **Lab Sample ID:** 733061

Matrix: SEDIMENT **Client:** ENSCON **Date Received:** 11/07/07

% Solids: 48.1

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	20.4	182	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	6.9	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		48.1	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR21

Lab Name: TAL BURLINGTON **Contract:** 2055227 **SDG No.:** 123121

Lab Code: TALVT **Case No.:** ANACOST **Lab Sample ID:** 733062

Matrix: SEDIMENT **Client:** ENSCON **Date Received:** 11/07/07

% Solids: 27.2

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	35.4	431	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	6.9	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		27.2	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR02

Lab Name: TAL BURLINGTON | Contract: 2055227 | SDG No.: 123121

Lab Code: TALVT | Case No.: ANACOST | Lab Sample ID: 733066

Matrix: SEDIMENT | Client: ENSCON | Date Received: 11/07/07

% Solids: 48.5

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	19.8	225	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	7.1	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		48.5	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR03

Lab Name: TAL BURLINGTON **Contract:** 2055227 **SDG No.:** 123121

Lab Code: TALVT **Case No.:** ANACOST **Lab Sample ID:** 733067

Matrix: SEDIMENT **Client:** ENSCON **Date Received:** 11/07/07

% Solids: 80.8

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	12.4	37.1	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	7.2	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		80.8	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR05

Lab Name: TAL BURLINGTON | Contract: 2055227 | SDG No.: 123121

Lab Code: TALVT | Case No.: ANACOST | Lab Sample ID: 733073

Matrix: SEDIMENT | Client: ENSCON | Date Received: 11/07/07

% Solids: 47.6

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	19.8	303	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	6.9	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		47.6	

Printed on: 12/05/07 11:10 AM

WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR08

Lab Name: TAL BURLINGTON **Contract:** 2055227 **SDG No.:** 123121

Lab Code: TALVT **Case No.:** ANACOST **Lab Sample ID:** 733076

Matrix: SEDIMENT **Client:** ENSCON **Date Received:** 11/07/07

% Solids: 37.6

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	25.6	315	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	6.9	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		37.6	

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WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR10

Lab Name: TAL BURLINGTON **Contract:** 2055227 **SDG No.:** 123121

Lab Code: TALVT **Case No.:** ANACOST **Lab Sample ID:** 733077

Matrix: SEDIMENT **Client:** ENSCON **Date Received:** 11/07/07

% Solids: 38.7

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	24.4	335	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	6.9	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		38.7	

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WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR07

Lab Name: TAL BURLINGTON **Contract:** 2055227 **SDG No.:** 123121

Lab Code: TALVT **Case No.:** ANACOST **Lab Sample ID:** 733080

Matrix: SEDIMENT **Client:** ENSCON **Date Received:** 11/07/07

% Solids: 39.6

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	23.0	309	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	6.9	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		39.6	

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WET CHEMISTRY

Sample Report Summary

Client Sample No.
AR36

Lab Name: TAL BURLINGTON **Contract:** 2055227 **SDG No.:** 123121

Lab Code: TALVT **Case No.:** ANACOST **Lab Sample ID:** 733084

Matrix: SEDIMENT **Client:** ENSCON **Date Received:** 11/07/07

% Solids: 36.9

Method	Parameter	Analytical Run Date	Analytical Batch	Units	DF	RL	Conc.	Qual.
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	1	24.6	325	
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	1	0.0	7.0	
IN623	Solids, Percent	11/28/07	N/A	%	1.0		36.9	

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WET CHEMISTRY

Method Blank Report Summary

Lab Name: TAL BURLINGTON **Contract:** 2055227 **SDG No.:** 123121

Lab Code: TALVT **Case No.:** ANACOST

Matrix: SOIL **Client:** ENSCON

% Solids:

Lab Sample ID	Method	Parameter	Conc.	Units	Qual.	DF	RL	Analytical Run Date	Analytical Batch
BLKNH120107A	350.2	Ammonia-Nitrogen	10.0	mg/Kg	U	1	10.0	12/01/07	BLKNH120107A
BLKPH112807A	9045	Soil pH (std. units)	5.8	pH Units		1	0.0	11/28/07	BLKPH112807A

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WET CHEMISTRY

Matrix Spike Sample Report Summary

Client Sample No.: AR14MS

Lab Name: TAL BURLINGTON | Contract: 2055227 | SDG No.: 123121

Lab Code: TALVT | Case No.: ANACOST | Lab Sample ID: 733051MS

Matrix: SEDIMENT | Client: ENSCON | Date Received: 11/07/07

% Solids: 35.4

Method	Parameter	Analytical Run Date	Analytical Batch	Units	Matrix Spike Result Conc.	Qual.	Sample Result Conc.	Qual.	Spike Added	% Rec.	Control Limit
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	520		271		213	117	85-115

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WET CHEMISTRY

Duplicate Sample Report Summary

Client Sample No.

AR14REP

Lab Name: TAL BURLINGTON | **Contract: 2055227** | **SDG No.: 123121**

Lab Code: TALVT | **Case No.: ANACOST** | **Lab Sample ID: 733051DP**

Matrix: SEDIMENT | **Client: ENSCON** | **Date Received: 11/07/07**

% Solids: 34.6

Method	Parameter	Analytical Run Date	Analytical Batch	Units	Sample Result Conc.	Sample Result Qual.	Duplicate Sample Result Conc.	Duplicate Sample Result Qual.	RPD*
350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	271		280		3
9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	6.8		6.9		1
IN623	Solids, Percent	11/28/07	N/A	%	35.4		34.6		2

** Control Limit for RPD is +/- 20%, unless otherwise specified.*

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WET CHEMISTRY

Laboratory Control Sample Report Summary

Lab Name: TAL BURLINGTON | Contract: 2055227 | SDG No.: 123121

Lab Code: TALVT | Case No.: ANACOST

Matrix: SOIL | Client: ENSCON

% Solids:

Lab Sample ID	Method	Parameter	Analytical Run Date	Analytical Batch	Units	LCS Conc.	True Value	% Rec.	Control Limit
LCSNH120107A	350.2	Ammonia-Nitrogen	12/01/07	BLKNH120107A	mg/Kg	78.0	80	98	85-115
LCSPH112807A	9045	Soil pH (std. units)	11/28/07	BLKPH112807A	pH Units	6.0	6.0	100	99-101

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TestAmerica

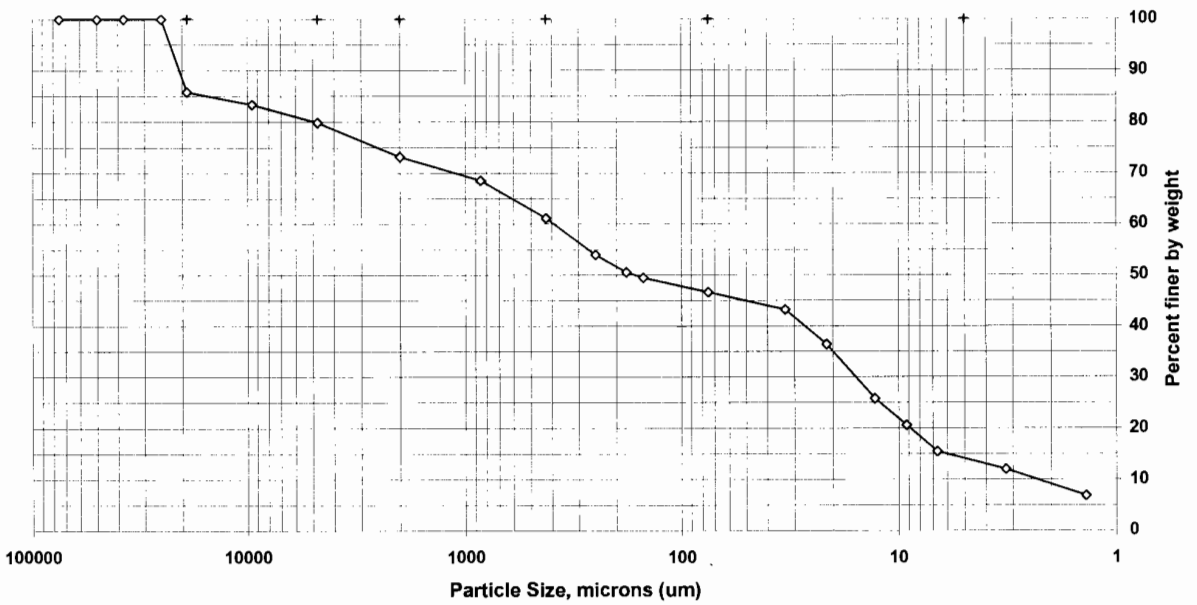
THE LEADER IN ENVIRONMENTAL TESTING

Sample Data Summary – Geotechnical

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR13	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733050			**End Date:**	12/10/2007

Percent Solids:	38.1%		**Non-soil material:**	plant, plastic
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	subrounded
Maximum Particle Size:	25 mm		**Hardness (> #10):**	hard



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	85.8	14.2
3/8 inch	9500	83.3	2.5
#4	4750	79.8	3.5
#10	2000	73.2	6.7
#20	850	68.5	4.6
#40	425	61.2	7.4
#60	250	53.9	7.3
#80	180	50.6	3.4
#100	150	49.4	1.1
#200	75	46.7	2.8
Hydrometer	33.1	43.2	3.4
|	21.4	36.4	6.9
|	12.8	25.8	10.6
|	9.2	20.6	5.2
|	6.7	15.5	5.2
|	3.2	12.0	3.4
V	1.4	6.9	5.2

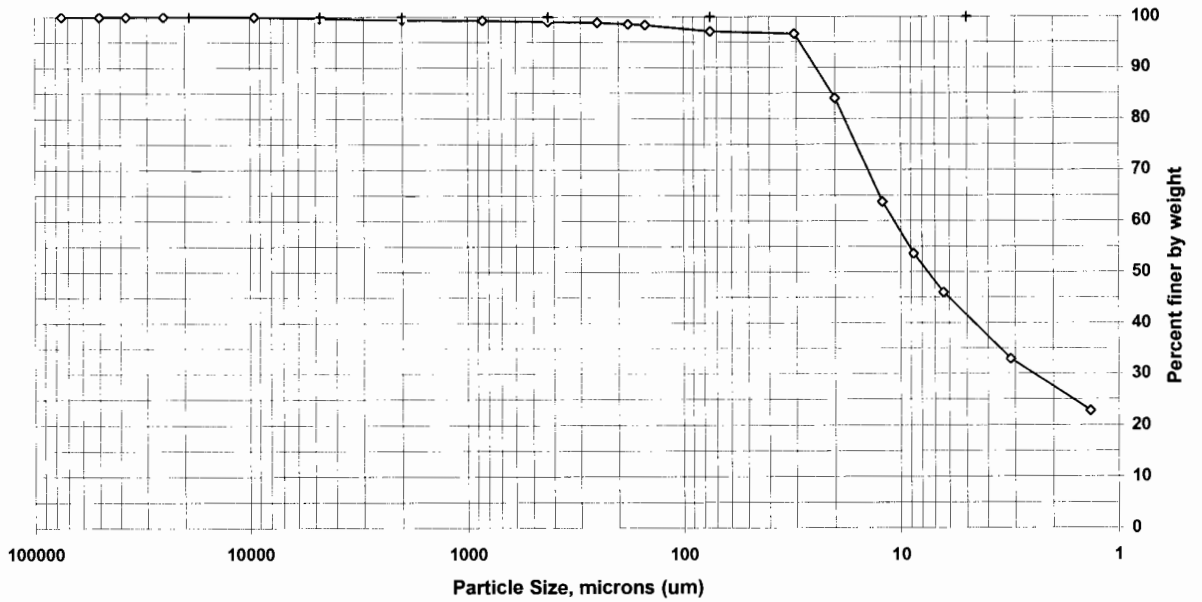
Soil Classification	Percent of Total Sample
Gravel	20.2
Sand	33.2
Coarse Sand	6.7
Medium Sand	12.0
Fine Sand	14.5
Silt	31.2
Clay	15.5

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR14	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733051			**End Date:**	12/10/2007

Percent Solids:	38.4%		**Non-soil material:**	plant, plastic
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	angular
Maximum Particle Size:	9.5 mm		**Hardness (> #10):**	hard



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	100.0	0.0
#4	4750	99.7	0.3
#10	2000	99.4	0.3
#20	850	99.3	0.1
#40	425	99.1	0.3
#60	250	98.8	0.2
#80	180	98.5	0.3
#100	150	98.4	0.2
#200	75	97.1	1.2
Hydrometer	30.7	96.6	0.5
|	20.0	84.0	12.7
|	12.1	63.7	20.3
|	8.7	53.6	10.1
|	6.4	46.0	7.6
|	3.1	32.9	13.1
V	1.3	22.8	10.1

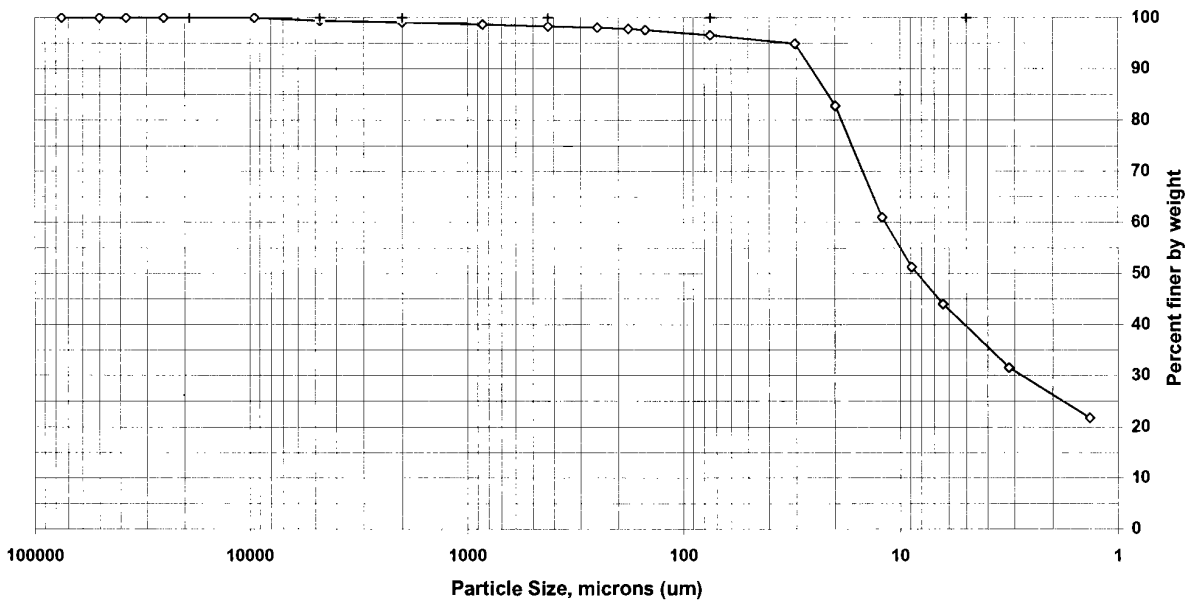
Soil Classification	Percent of Total Sample
Gravel	0.3
Sand	2.6
Coarse Sand	0.3
Medium Sand	0.3
Fine Sand	1.9
Silt	51.1
Clay	46.0

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR14REP	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733051DP			**End Date:**	12/10/2007

Percent Solids:	38.2%		**Non-soil material:**	plant, plastic
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	subangular
Maximum Particle Size:	9.5 mm		**Hardness (> #10):**	hard



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	100.0	0.0
#4	4750	99.4	0.6
#10	2000	99.1	0.3
#20	850	98.7	0.4
#40	425	98.3	0.4
#60	250	98.1	0.2
#80	180	97.9	0.2
#100	150	97.6	0.2
#200	75	96.6	1.1
Hydrometer	30.5	94.9	1.7
|	19.9	82.8	12.1
|	12.1	61.0	21.8
|	8.9	51.3	9.7
|	6.4	44.0	7.3
|	3.2	31.5	12.5
V	1.4	21.8	9.7

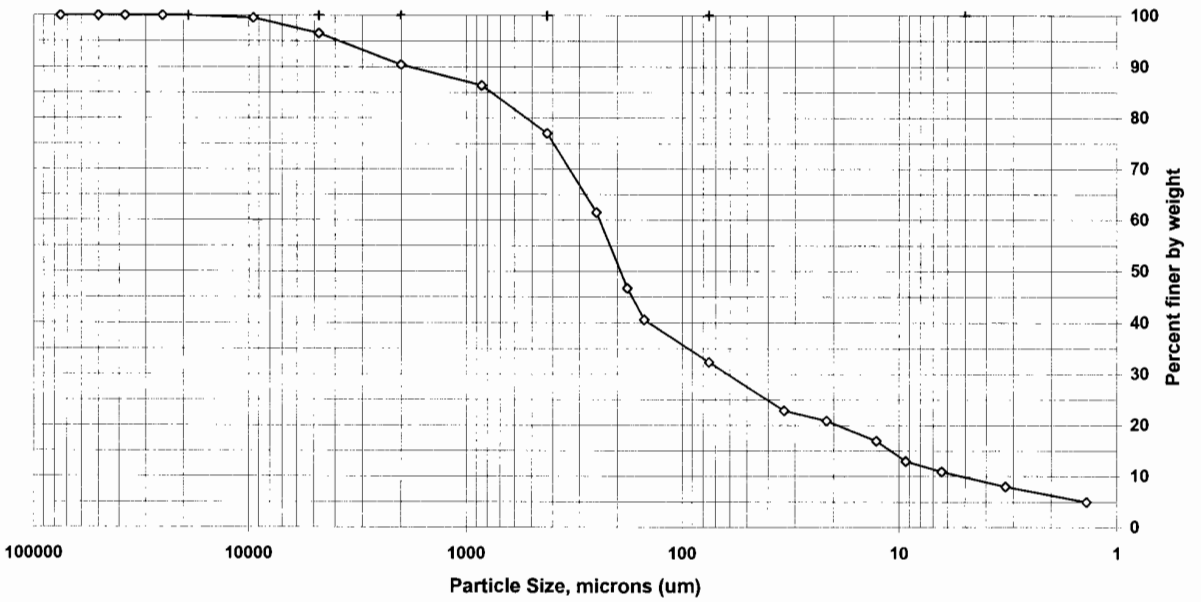
Soil Classification	Percent of Total Sample
Gravel	0.6
Sand	2.9
Coarse Sand	0.3
Medium Sand	0.8
Fine Sand	1.8
Silt	52.5
Clay	44.0

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR16	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733053			**End Date:**	12/10/2007

Percent Solids:	49.7%		**Non-soil material:**	plant, shell	
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	subangular	
Maximum Particle Size:	19 mm		**Hardness (> #10):**	hard	



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	99.5	0.5
#4	4750	96.5	3.0
#10	2000	90.4	6.1
#20	850	86.4	4.0
#40	425	77.0	9.4
#60	250	61.4	15.6
#80	180	46.7	14.7
#100	150	40.6	6.1
#200	75	32.3	8.3
Hydrometer	33.7	22.9	9.4
|	21.6	20.9	2.0
|	12.7	16.9	4.0
|	9.3	12.9	4.0
|	6.4	10.9	2.0
|	3.3	8.0	3.0
V	1.4	5.0	3.0

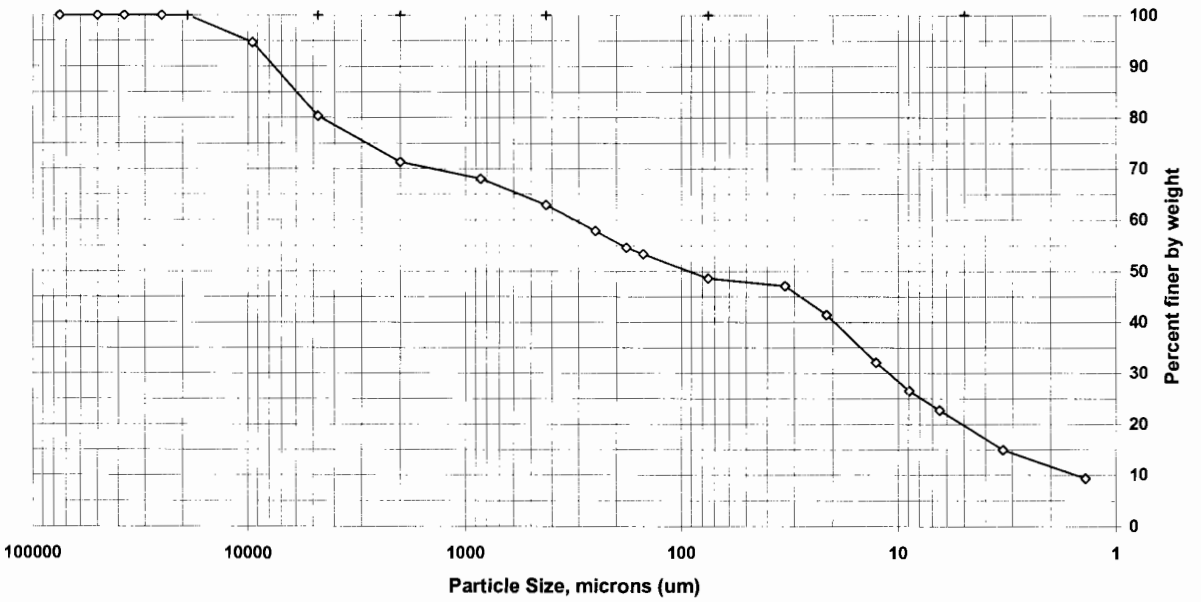
Soil Classification	Percent of Total Sample
Gravel	3.5
Sand	64.2
Coarse Sand	6.1
Medium Sand	13.4
Fine Sand	44.7
Silt	21.4
Clay	10.9

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR17	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733054			**End Date:**	12/10/2007

Percent Solids:	42.7%		**Non-soil material:**	plant	
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	subrounded	
Maximum Particle Size:	19 mm		**Hardness (> #10):**	hard	



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	94.7	5.3
#4	4750	80.3	14.4
#10	2000	71.3	9.0
#20	850	68.0	3.3
#40	425	62.9	5.1
#60	250	57.9	5.0
#80	180	54.6	3.3
#100	150	53.3	1.3
#200	75	48.6	4.7
Hydrometer	33.1	47.1	1.5
|	21.3	41.5	5.6
|	12.6	32.1	9.4
|	8.9	26.5	5.6
|	6.5	22.8	3.7
|	3.3	15.0	7.8
V	1.4	9.4	5.6

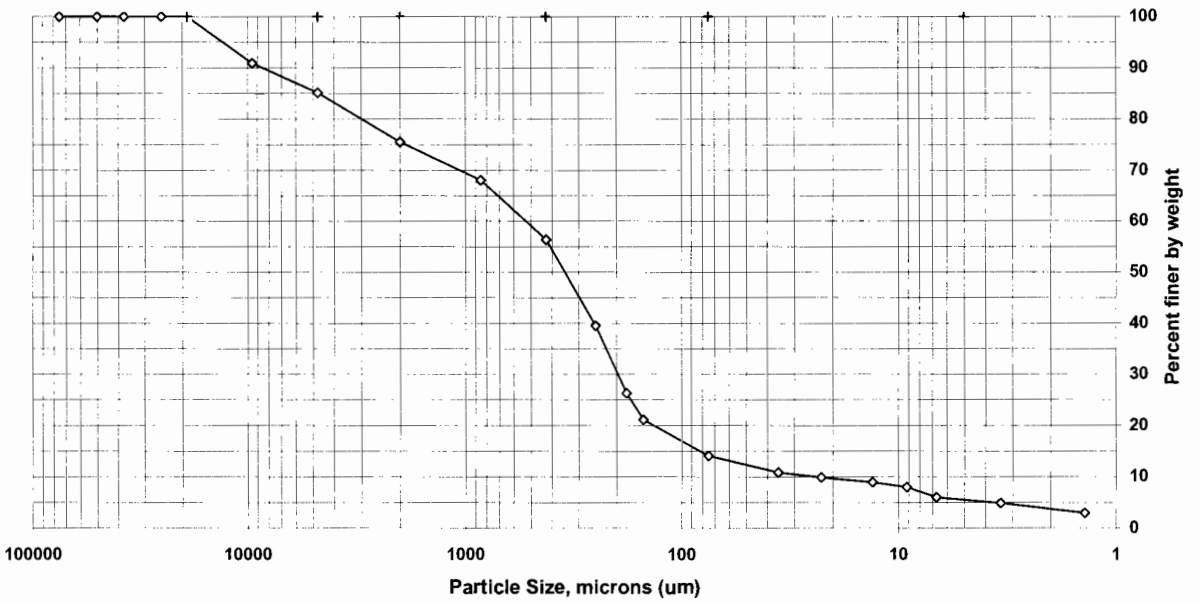
Soil Classification	Percent of Total Sample
Gravel	19.7
Sand	31.7
Coarse Sand	9.0
Medium Sand	8.4
Fine Sand	14.3
Silt	25.8
Clay	22.8

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR26	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733057			**End Date:**	12/10/2007

Percent Solids:	53.2%		**Non-soil material:**	plant, shell
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	angular
Maximum Particle Size:	19 mm		**Hardness (> #10):**	hard



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	90.9	9.1
#4	4750	85.1	5.7
#10	2000	75.5	9.6
#20	850	68.1	7.4
#40	425	56.4	11.7
#60	250	39.5	16.9
#80	180	26.3	13.2
#100	150	21.2	5.2
#200	75	14.1	7.1
Hydrometer	35.6	10.9	3.2
|	22.6	9.9	1.0
|	13.1	8.9	1.0
|	9.2	8.0	1.0
|	6.7	6.0	1.9
|	3.4	4.9	1.1
V	1.4	2.9	1.9

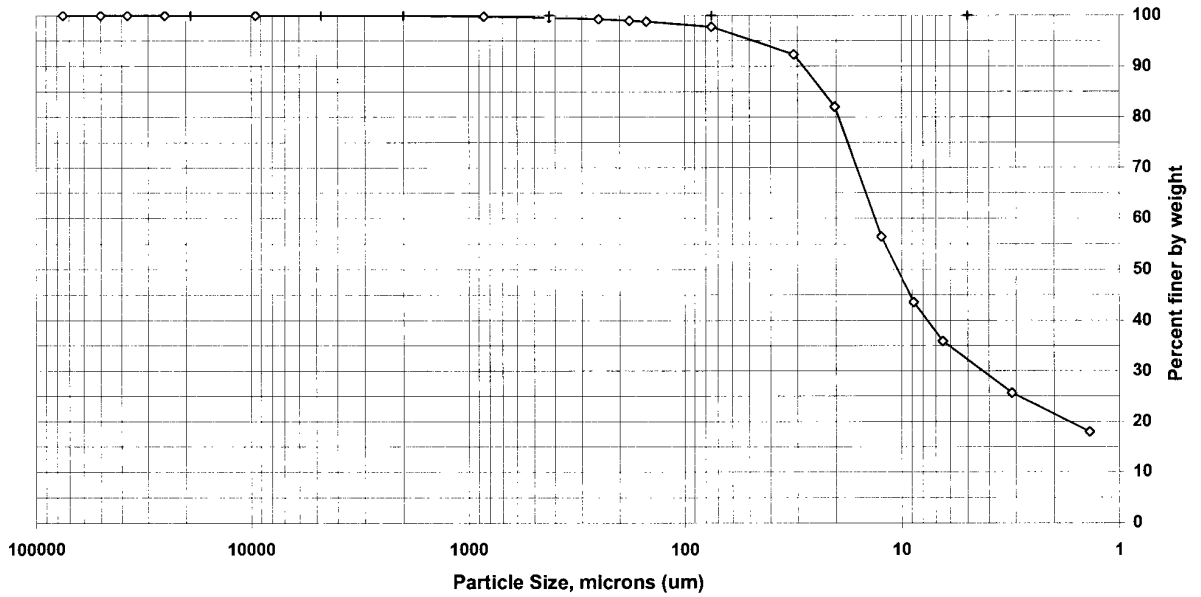
Soil Classification	Percent of Total Sample
Gravel	14.9
Sand	71.1
Coarse Sand	9.6
Medium Sand	19.2
Fine Sand	42.3
Silt	8.1
Clay	6.0

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR25	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733059			**End Date:**	12/10/2007

Percent Solids:	38.8%		**Non-soil material:**	n/a
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	n/a
Maximum Particle Size:	Med sand		**Hardness (> #10):**	n/a



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	100.0	0.0
#4	4750	100.0	0.0
#10	2000	100.0	0.0
#20	850	99.8	0.2
#40	425	99.6	0.3
#60	250	99.3	0.3
#80	180	99.0	0.3
#100	150	98.8	0.2
#200	75	97.8	1.0
Hydrometer	31.2	92.3	5.5
|	20.3	82.0	10.3
|	12.4	56.4	25.6
|	8.8	43.6	12.8
|	6.5	35.9	7.7
|	3.1	25.6	10.3
V	1.4	17.9	7.7

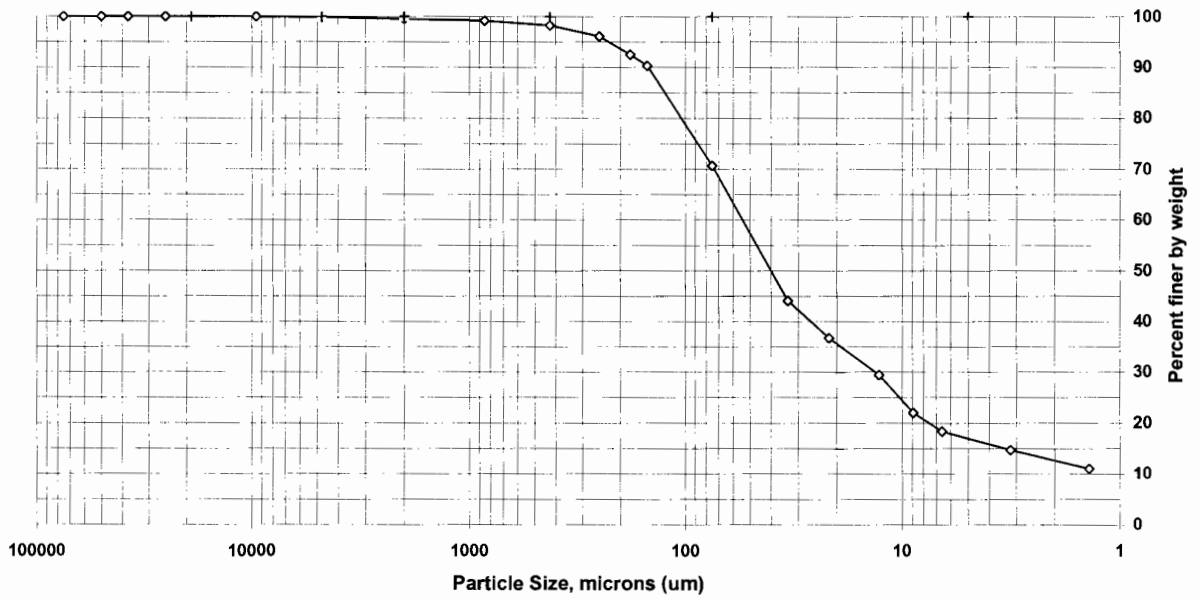
Soil Classification	Percent of Total Sample
Gravel	0.0
Sand	2.2
Coarse Sand	0.0
Medium Sand	0.4
Fine Sand	1.8
Silt	61.9
Clay	35.9

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR27	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733061			**End Date:**	12/10/2007

Percent Solids:	48.1%		**Non-soil material:**	plant
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	n/a
Maximum Particle Size:	9.5 mm		**Hardness (> #10):**	n/a



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	100.0	0.0
#4	4750	99.9	0.1
#10	2000	99.5	0.4
#20	850	99.2	0.4
#40	425	98.3	0.9
#60	250	96.1	2.2
#80	180	92.5	3.6
#100	150	90.3	2.2
#200	75	70.6	19.7
Hydrometer	33.5	44.1	26.5
|	21.7	36.8	7.4
|	12.8	29.4	7.4
|	8.9	22.1	7.4
|	6.6	18.4	3.7
|	3.2	14.7	3.7
V	1.4	11.0	3.7

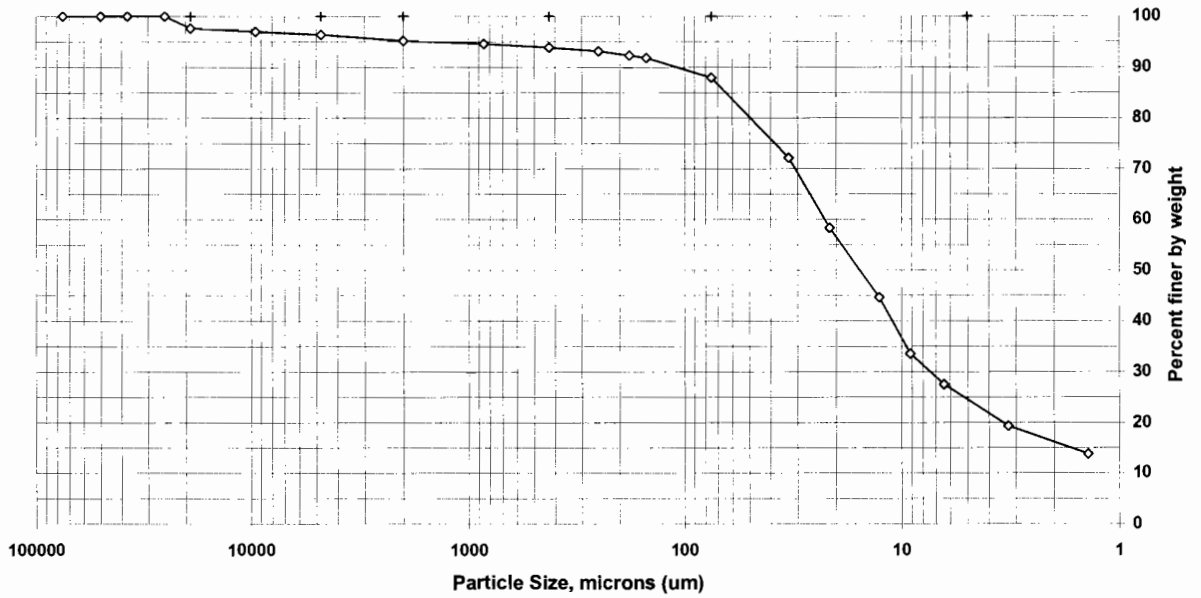
Soil Classification	Percent of Total Sample
Gravel	0.1
Sand	29.2
Coarse Sand	0.4
Medium Sand	1.2
Fine Sand	27.6
Silt	52.3
Clay	18.4

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR21	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733062			**End Date:**	12/10/2007

Percent Solids:	31.3%		**Non-soil material:**	plant, plastic
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	n/a
Maximum Particle Size:	25 mm		**Hardness (> #10):**	n/a



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	97.6	2.4
3/8 inch	9500	97.0	0.7
#4	4750	96.4	0.6
#10	2000	95.2	1.2
#20	850	94.6	0.6
#40	425	93.9	0.7
#60	250	93.1	0.8
#80	180	92.3	0.8
#100	150	91.9	0.4
#200	75	87.9	3.9
Hydrometer	33.0	72.2	15.8
|	21.4	58.4	13.8
|	12.7	44.6	13.8
|	9.2	33.6	11.0
|	6.4	27.6	6.0
|	3.2	19.3	8.3
V	1.4	13.8	5.5

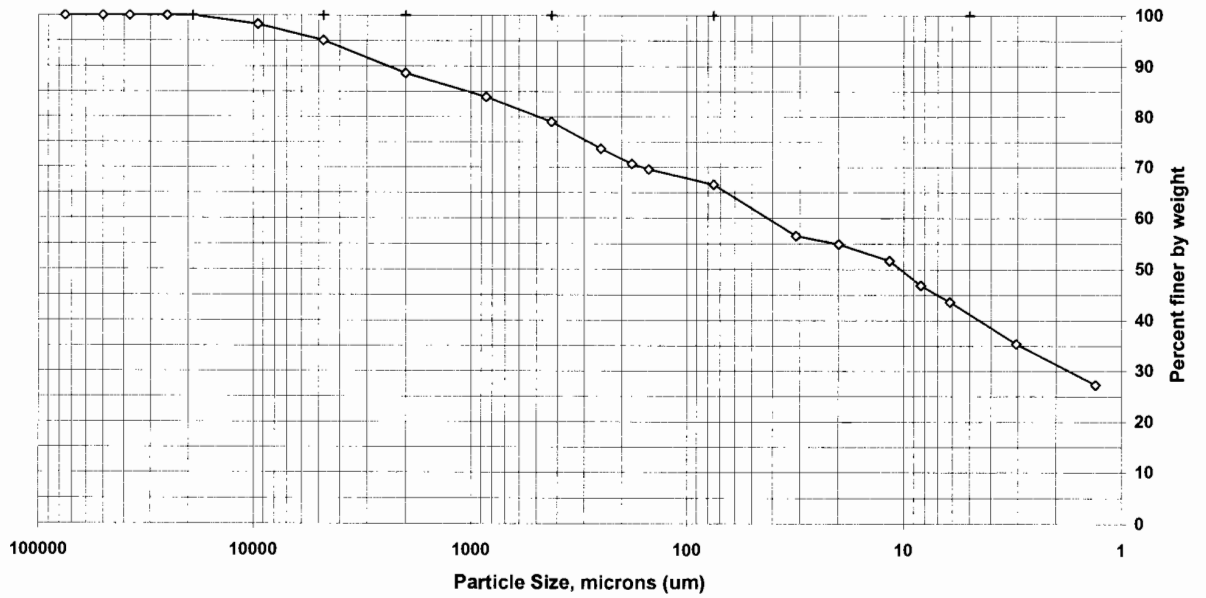
Soil Classification	Percent of Total Sample
Gravel	3.6
Sand	8.5
Coarse Sand	1.2
Medium Sand	1.3
Fine Sand	6.0
Silt	60.3
Clay	27.6

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR02	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733066			**End Date:**	12/10/2007

Percent Solids:	53.3%		**Non-soil material:**	plant	
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	angular	
Maximum Particle Size:	19 mm		**Hardness (> #10):**	hard	



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	98.3	1.7
#4	4750	95.1	3.2
#10	2000	88.6	6.5
#20	850	83.9	4.6
#40	425	79.0	4.9
#60	250	73.6	5.4
#80	180	70.6	3.0
#100	150	69.6	1.0
#200	75	66.5	3.0
Hydrometer	31.3	56.5	10.0
|	19.9	54.9	1.6
|	11.6	51.7	3.2
|	8.4	46.9	4.8
|	6.1	43.7	3.2
|	3.1	35.4	8.3
V	1.3	27.3	8.0

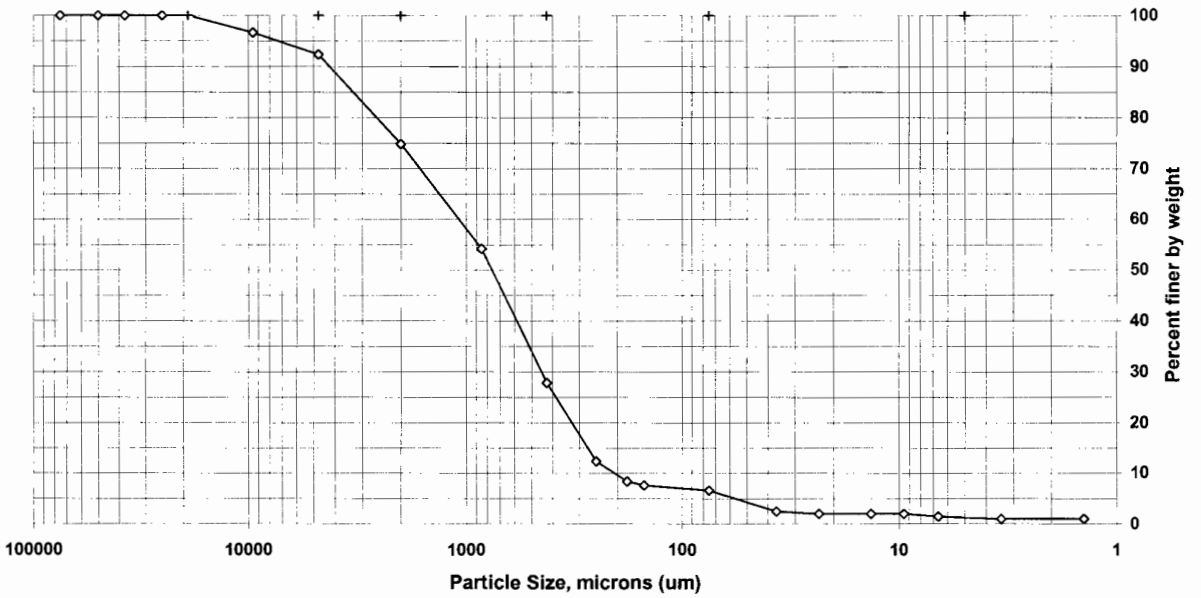
Soil Classification	Percent of Total Sample
Gravel	4.9
Sand	28.5
Coarse Sand	6.5
Medium Sand	9.6
Fine Sand	12.5
Silt	22.9
Clay	43.7

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR03	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733067			**End Date:**	12/10/2007

Percent Solids:	82.5%		**Non-soil material:**	glass, plastic	
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	rounded	
Maximum Particle Size:	19 mm		**Hardness (> #10):**	hard	



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	96.6	3.4
#4	4750	92.3	4.3
#10	2000	74.9	17.4
#20	850	54.2	20.7
#40	425	27.8	26.4
#60	250	12.4	15.5
#80	180	8.4	3.9
#100	150	7.6	0.8
#200	75	6.6	1.0
Hydrometer	36.6	2.5	4.1
|	23.3	2.0	0.5
|	13.4	2.0	0.0
|	9.5	2.0	0.0
|	6.6	1.4	0.6
|	3.4	1.0	0.5
V	1.4	1.0	0.0

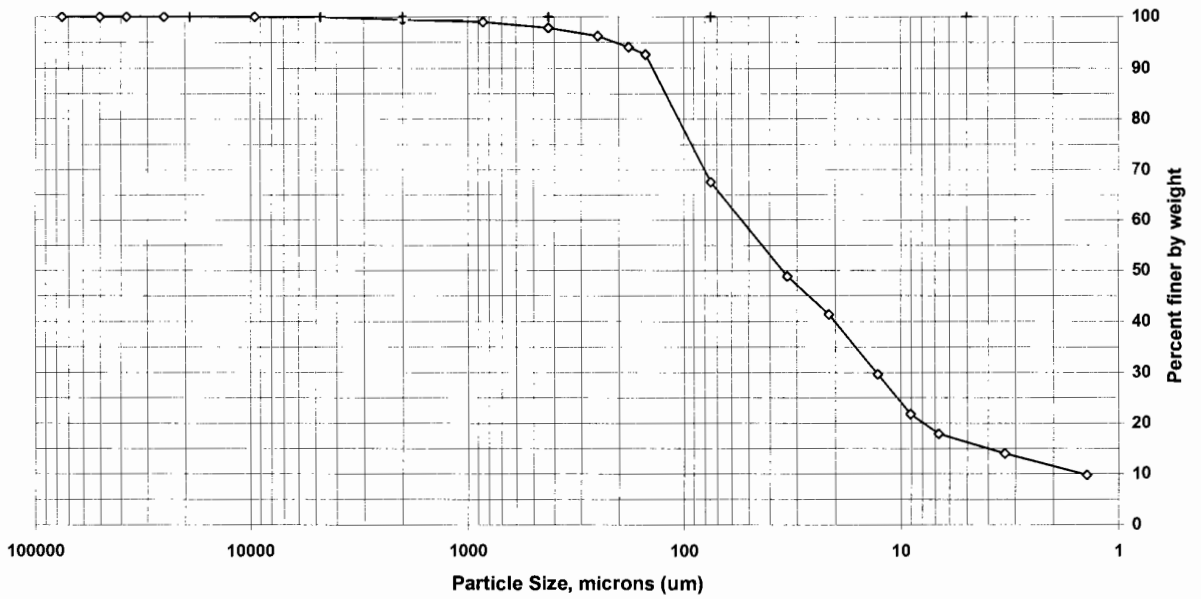
Soil Classification	Percent of Total Sample
Gravel	7.7
Sand	85.7
Coarse Sand	17.4
Medium Sand	47.0
Fine Sand	21.2
Silt	5.2
Clay	1.4

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR05	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733073			**End Date:**	12/10/2007

Percent Solids:	47.1%		**Non-soil material:**	plant
Specific Gravity:	2.650	(assumed)	**Shape (> #10):**	n/a
Maximum Particle Size:	9.5 mm		**Hardness (> #10):**	n/a



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	100.0	0.0
#4	4750	99.9	0.1
#10	2000	99.5	0.5
#20	850	99.0	0.5
#40	425	97.8	1.2
#60	250	96.3	1.6
#80	180	94.1	2.1
#100	150	92.7	1.4
#200	75	67.5	25.2
Hydrometer	33.2	48.8	18.7
|	21.4	41.4	7.4
|	12.8	29.6	11.7
|	9.0	21.8	7.8
|	6.7	17.9	3.9
|	3.3	14.0	3.9
V	1.4	9.8	4.2

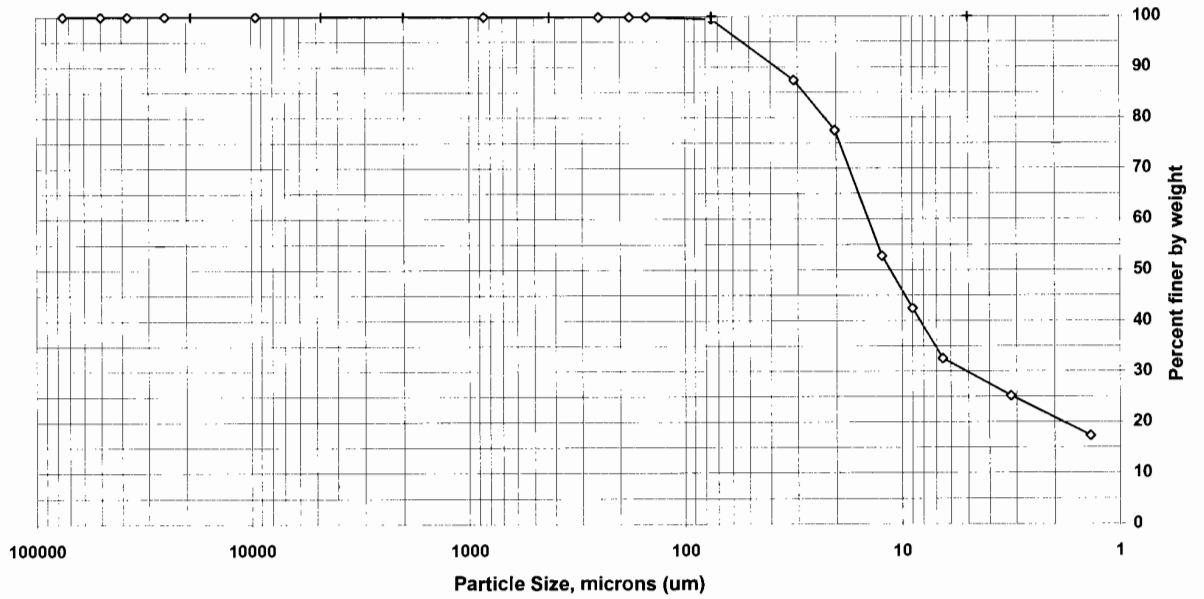
Soil Classification	Percent of Total Sample
Gravel	0.1
Sand	32.4
Coarse Sand	0.5
Medium Sand	1.7
Fine Sand	30.3
Silt	49.6
Clay	17.9

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR08	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733076			**End Date:**	12/10/2007

Percent Solids:	38.7%	**Non-soil material:**	n/a
Specific Gravity:	2.650	**Shape (> #10):**	n/a
Maximum Particle Size:	Med sand	**Hardness (> #10):**	n/a



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	100.0	0.0
#4	4750	100.0	0.0
#10	2000	100.0	0.0
#20	850	100.0	0.0
#40	425	99.9	0.1
#60	250	99.9	0.0
#80	180	99.9	0.0
#100	150	99.9	0.0
#200	75	99.4	0.4
Hydrometer	31.3	87.4	12.1
|	20.2	77.5	9.9
|	12.4	52.8	24.7
|	9.0	42.4	10.3
|	6.5	32.6	9.9
|	3.2	25.1	7.4
V	1.4	17.3	7.8

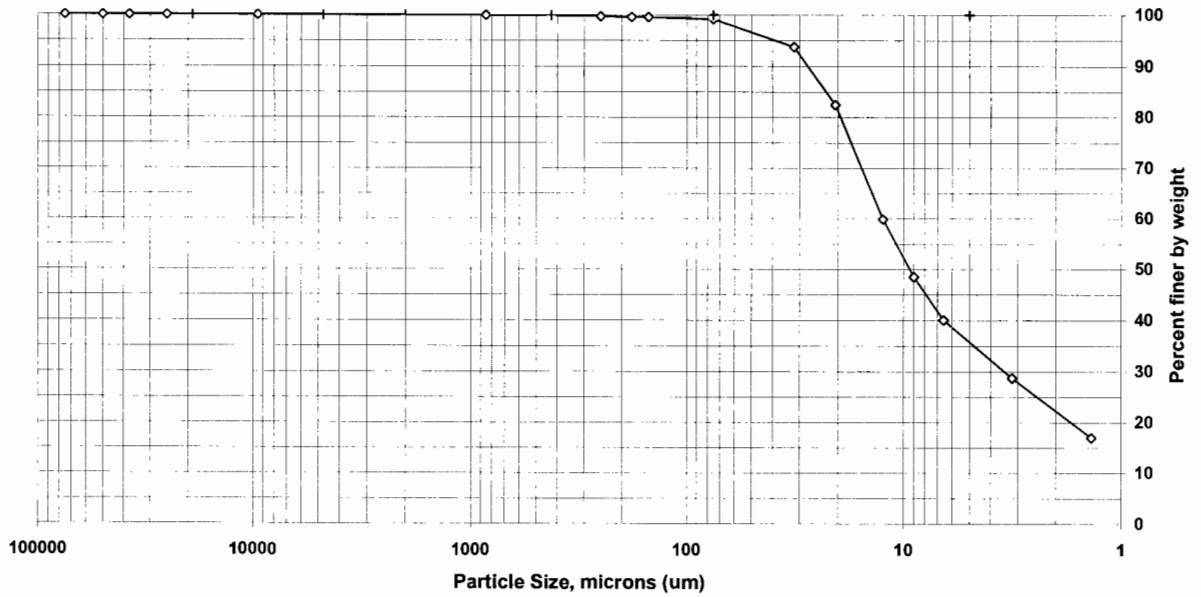
Soil Classification	Percent of Total Sample
Gravel	0.0
Sand	0.6
Coarse Sand	0.0
Medium Sand	0.1
Fine Sand	0.4
Silt	66.9
Clay	32.6

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR10	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733077			**End Date:**	12/10/2007

Percent Solids:	39.7%	**Non-soil material:**	plant
Specific Gravity:	2.650	**Shape (> #10):**	n/a
Maximum Particle Size:	Crs sand	**Hardness (> #10):**	n/a



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	100.0	0.0
#4	4750	100.0	0.0
#10	2000	99.9	0.1
#20	850	99.9	0.0
#40	425	99.9	0.1
#60	250	99.7	0.1
#80	180	99.6	0.1
#100	150	99.6	0.0
#200	75	99.2	0.4
Hydrometer	31.8	93.8	5.4
|	20.6	82.4	11.3
|	12.4	59.8	22.6
|	9.0	48.5	11.3
|	6.6	40.0	8.5
|	3.2	28.7	11.3
V	1.4	17.0	11.8

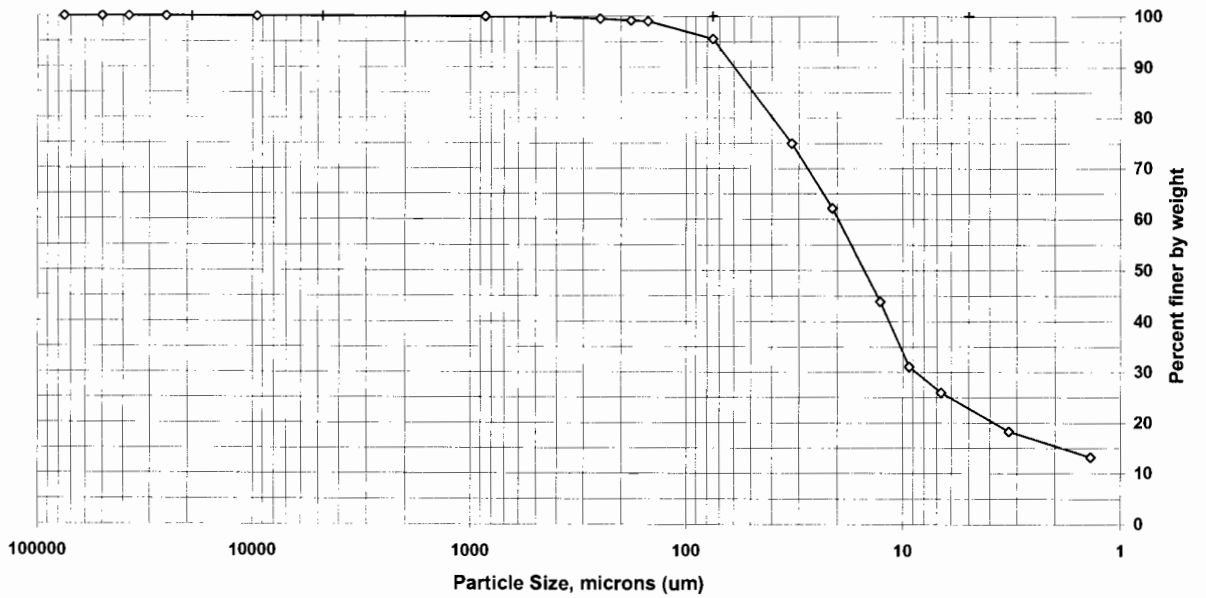
Soil Classification	Percent of Total Sample
Gravel	0.0
Sand	0.8
Coarse Sand	0.1
Medium Sand	0.1
Fine Sand	0.7
Silt	59.1
Clay	40.0

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR07	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733080			**End Date:**	12/10/2007

Percent Solids:	41.2%	**Non-soil material:**	n/a
Specific Gravity:	2.650	**Shape (> #10):**	n/a
Maximum Particle Size:	Med sand	**Hardness (> #10):**	n/a



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	100.0	0.0
#4	4750	100.0	0.0
#10	2000	100.0	0.0
#20	850	100.0	0.0
#40	425	99.8	0.2
#60	250	99.5	0.3
#80	180	99.2	0.4
#100	150	99.0	0.1
#200	75	95.6	3.5
Hydrometer	32.4	75.0	20.6
I	21.1	62.2	12.8
I	12.7	43.9	18.3
I	9.4	31.1	12.8
I	6.7	26.0	5.1
I	3.3	18.3	7.7
V	1.4	13.2	5.1

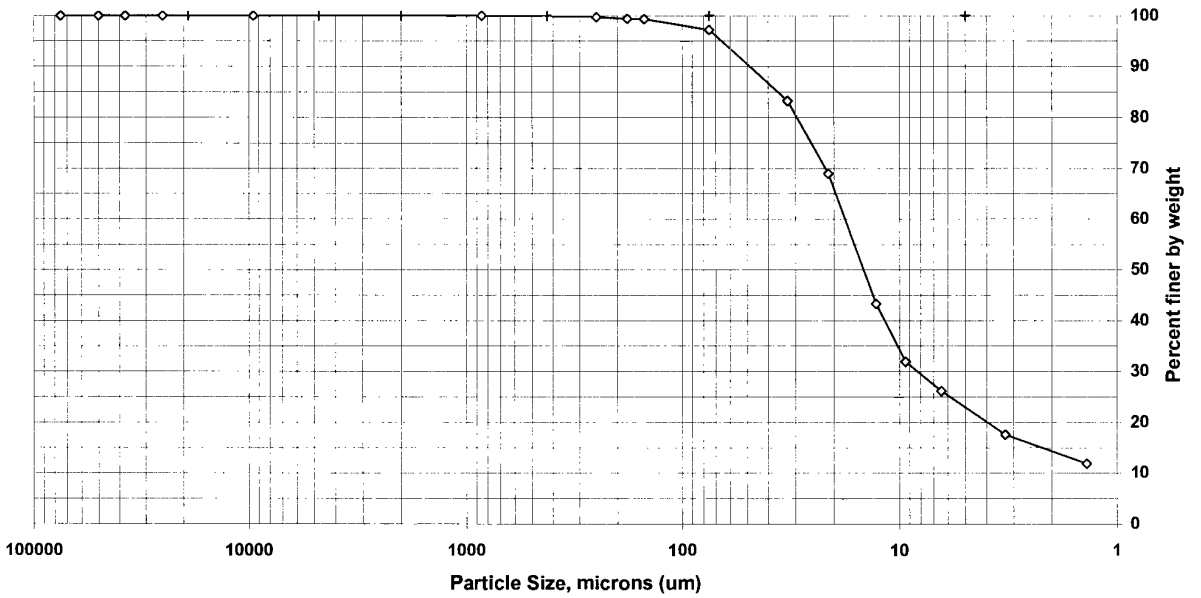
Soil Classification	Percent of Total Sample
Gravel	0.0
Sand	4.4
Coarse Sand	0.0
Medium Sand	0.2
Fine Sand	4.2
Silt	69.6
Clay	26.0

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute

Particle Size of Soils by ASTM D422

Client Code:	ENSCON	**SDG:**	123121	**Date Received:**	11/7/2007
Sample ID:	AR36	**ETR(s):**	123121	**Start Date:**	11/28/2007
Lab ID:	733084			**End Date:**	12/10/2007

Percent Solids:	39.1%	**Non-soil material:**	n/a
Specific Gravity:	2.650	**Shape (> #10):**	n/a
Maximum Particle Size:	Med sand	**Hardness (> #10):**	n/a



Sieve size	Particle size, um	Percent finer	Incremental percent
3 inch	75000	100.0	0.0
2 inch	50000	100.0	0.0
1.5 inch	37500	100.0	0.0
1 inch	25000	100.0	0.0
3/4 inch	19000	100.0	0.0
3/8 inch	9500	100.0	0.0
#4	4750	100.0	0.0
#10	2000	100.0	0.0
#20	850	100.0	0.0
#40	425	99.9	0.1
#60	250	99.7	0.2
#80	180	99.4	0.3
#100	150	99.4	0.0
#200	75	97.2	2.2
Hydrometer	32.6	83.2	14.0
|	21.2	69.0	14.3
|	12.8	43.3	25.7
|	9.4	31.9	11.4
|	6.4	26.2	5.7
|	3.3	17.6	8.6
V	1.4	11.9	5.7

Soil Classification	Percent of Total Sample
Gravel	0.0
Sand	2.8
Coarse Sand	0.0
Medium Sand	0.1
Fine Sand	2.7
Silt	71.0
Clay	26.2

Preparation Method: **D2217**
Dispersion Device: Mechanical mixer with a metal paddle.
Dispersion Period: 1 minute



Sample Handling

11/27/07 1412

This portion can be removed for Recipient's records.

Date 11/6/07 FedEx Tracking Number 861422672336

Sender's Name [redacted] Helen Jones Phone [redacted]

Company [redacted]

Address [redacted] Dept./Floor/Suite/Room

City [redacted] State NY ZIP [redacted]

Your Internal Billing Reference 0930 - 422 - 100

RECEIVED
11/07/07
0930

This portion can be removed for Recipient's records.

Date 11/7/07 FedEx Tracking Number 861422672299

Sender's Name [redacted] Helen Jones Phone [redacted]

Company [redacted]

Address [redacted] Dept./Floor/Suite/Room

City [redacted] State NY ZIP [redacted]

Your Internal Billing Reference 0930 - 422 - 100

RECEIVED
11/06/07
0930

This portion can be removed for Recipient's records.

Date 11/5/07 FedEx Tracking Number 861422672174

Sender's Name [redacted] Helen Jones Phone [redacted]

Company [redacted]

Address [redacted] Dept./Floor/Suite/Room

City [redacted] State NY ZIP [redacted]

Your Internal Billing Reference 0930 - 422 - 100

RECEIVED
11/9/07 093

STL BURLINGTON
SAMPLE RECEIPT & LOG IN CHECKLIST

Client: ENSCON	Date Received: 11/07/07	Log In Date: 11/27/07	
ETR: 123121	Time Received: 0930	By: JEG	
SDG: 123121	Received By: JEG	Signature:	
Project: 27000	# Coolers Received: 1	PM Signature:	
Samples Delivered By: X Shipping Service ☐ Courier ☐ Hand ☐ Other (specify)		Date: 11/27/07	
List Air bill Number(s) or Attach a photocopy of the Air Bill:			

COOLER SCREEN	YES	NO	NA	COMMENTS
There is ***no*** evidence to indicate tampering	X			
Custody seals are present and intact		X		
Custody seal numbers are present			X	
If yes, list custody seal numbers:				

Thermal Preservation Type: X Wet Ice ☐ Blue Ice ☐ None ☐ Other (specify)

IR Gun ID: 62 Correction Factor (CF) = 0 °C

Cooler 1:	5.5 °C	Cooler 6	°C	Cooler 11	°C	Cooler 16	°C
Cooler 2:	°C	Cooler 7	°C	Cooler 12	°C	Cooler 17	°C
Cooler 3:	°C	Cooler 8	°C	Cooler 13	°C	Cooler 18	°C
Cooler 4:	°C	Cooler 9	°C	Cooler 14	°C	Cooler 19	°C
Cooler 5	°C	Cooler 10	°C	Cooler 15	°C	Cooler 20	°C

Unless otherwise documented, the recorded temperature readings are adjusted readings to account for the CF of the IR Gun

EPA Criteria: 0-6°C, except for air and geo samples which should be at ambient temperature and tissue samples, which may be frozen.

Some clients require thermal preservation criteria of 2-4°C or other such criteria. The PM must notify SM when alternate criteria is specified.

SAMPLE CONDITION	YES	NO	NA	COMMENTS
Sample containers were received intact		X		See below
Legible sample labels are affixed to each container	X			

CHAIN OF CUSTODY (COC)	YES	NO	NA	COMMENTS
COC is present and includes the following information for each container:				
• Sample ID / Sample Description	X			
• Date of Sample Collection	X			
• Time of Sample Collection	X			
• Identification of the Sampler	X			
• Preservation Type			X	
• Requested Tests Method(s)	X			
• Necessary Signatures	X			
Internal Chain of Custody (ICOC) Required		X		
If yes to above, ICOC Record initiated for every Worksheet			X	

SAMPLE INTEGRITY / USABILITY	YES	NO	NA	COMMENTS
The sample container matches the COC	X			
Appropriate sample containers were received for the tests requested	X			
Samples were received within holding time	X			
Sufficient amount of sample is provided for requested analyses	X			
VOA vials do not have headspace or a bubble >6mm (1/4" diameter)			X	
Appropriate preservatives were used for the tests requested			X	
pH of inorganic samples checked and is within method specification			X	
If no, attach Inorganic Sample pH Adjustment Form			X	

ANOMALY / NCR SUMMARY:

1 of 2 250ml sample containers was received broken, sample ID AR13. The sample was placed into a new container and logged in with other samples.

STL BURLINGTON
SAMPLE RECEIPT & LOG IN CHECKLIST

Client: ENSCON	Date Received: 11.08.07	Log In Date: 11/27/07	
ETR: 123121	Time Received: 0930	By: JEG	
SDG: 123121	Received By: JEG	Signature:	
Project: 27000	# Coolers Received: 1	PM Signature:	
Samples Delivered By: X Shipping Service ☐ Courier ☐ Hand ☐ Other (specify)		Date: 11/27/07	
List Air bill Number(s) or Attach a photocopy of the Air Bill:			

COOLER SCREEN	YES	NO	NA	COMMENTS
There is ***no*** evidence to indicate tampering	X			
Custody seals are present and intact		X		
Custody seal numbers are present			X	
If yes, list custody seal numbers:				

Thermal Preservation Type: X Wet Ice ☐ Blue Ice ☐ None ☐ Other (specify)

IR Gun ID: 62 Correction Factor (CF) = 0 °C

Cooler 1:	5.8 °C	Cooler 6	°C	Cooler 11	°C	Cooler 16	°C
Cooler 2:	°C	Cooler 7	°C	Cooler 12	°C	Cooler 17	°C
Cooler 3:	°C	Cooler 8	°C	Cooler 13	°C	Cooler 18	°C
Cooler 4:	°C	Cooler 9	°C	Cooler 14	°C	Cooler 19	°C
Cooler 5	°C	Cooler 10	°C	Cooler 15	°C	Cooler 20	°C

Unless otherwise documented, the recorded temperature readings are adjusted readings to account for the CF of the IR Gun

EPA Criteria: 0-6°C, except for air and geo samples which should be at ambient temperature and tissue samples, which may be frozen.

Some clients require thermal preservation criteria of 2-4°C or other such criteria. The PM must notify SM when alternate criteria is specified.

SAMPLE CONDITION	YES	NO	NA	COMMENTS
Sample containers were received intact	X			
Legible sample labels are affixed to each container	X			

CHAIN OF CUSTODY (COC)	YES	NO	NA	COMMENTS
COC is present and includes the following information for each container:				
• Sample ID / Sample Description	X			
• Date of Sample Collection	X			
• Time of Sample Collection	X			
• Identification of the Sampler	X			
• Preservation Type			X	
• Requested Tests Method(s)	X			
• Necessary Signatures	X			
Internal Chain of Custody (ICOC) Required		X		
If yes to above, ICOC Record initiated for every Worksheet			X	

SAMPLE INTEGRITY / USABILITY	YES	NO	NA	COMMENTS
The sample container matches the COC	X			
Appropriate sample containers were received for the tests requested	X			
Samples were received within holding time	X			
Sufficient amount of sample is provided for requested analyses	X			
VOA vials do not have headspace or a bubble >6mm (1/4" diameter)			X	
Appropriate preservatives were used for the tests requested			X	
pH of inorganic samples checked and is within method specification			X	
If no, attach Inorganic Sample pH Adjustment Form			X	

ANOMALY / NCR SUMMARY

STL BURLINGTON
SAMPLE RECEIPT & LOG IN CHECKLIST

Client: ENSCON	Date Received: 11/09/07	Log In Date: 11/27/07	
ETR: 123121	Time Received: 0930	By: JEC	
SDG: 123121	Received By: JEC	Signature:	
Project: 27000	# Coolers Received: 1	PM Signature:	
Samples Delivered By: X Shipping Service ☐ Courier ☐ Hand ☐ Other (specify)		Date: 12/12/07	
List Air bill Number(s) or Attach a photocopy of the Air Bill:			

COOLER SCREEN	YES	NO	NA	COMMENTS
There is ***no*** evidence to indicate tampering	X			
Custody seals are present and intact		X		
Custody seal numbers are present			X	
If yes, list custody seal numbers:				

Thermal Preservation Type: X Wet Ice ☐ Blue Ice ☐ None ☐ Other (specify)

IR Gun ID: 62 Correction Factor (CF) = 0 °C

Cooler 1:	4.4 °C	Cooler 6	°C	Cooler 11	°C	Cooler 16	°C
Cooler 2:	°C	Cooler 7	°C	Cooler 12	°C	Cooler 17	°C
Cooler 3:	°C	Cooler 8	°C	Cooler 13	°C	Cooler 18	°C
Cooler 4:	°C	Cooler 9	°C	Cooler 14	°C	Cooler 19	°C
Cooler 5	°C	Cooler 10	°C	Cooler 15	°C	Cooler 20	°C

Unless otherwise documented, the recorded temperature readings are adjusted readings to account for the CF of the IR Gun

EPA Criteria: 0-6°C, except for air and geo samples which should be at ambient temperature and tissue samples, which may be frozen.

Some clients require thermal preservation criteria of 2-4°C or other such criteria. The PM must notify SM when alternate criteria is specified.

SAMPLE CONDITION	YES	NO	NA	COMMENTS
Sample containers were received intact	X			
Legible sample labels are affixed to each container	X			

CHAIN OF CUSTODY (COC)	YES	NO	NA	COMMENTS
COC is present and includes the following information for each container:				
• Sample ID / Sample Description	X			
• Date of Sample Collection	X			
• Time of Sample Collection	X			
• Identification of the Sampler	X			
• Preservation Type			X	
• Requested Tests Method(s)	X			
• Necessary Signatures	X			
Internal Chain of Custody (ICOC) Required		X		
If yes to above, ICOC Record initiated for every Worksheet			X	

SAMPLE INTEGRITY / USABILITY	YES	NO	NA	COMMENTS
The sample container matches the COC	X			
Appropriate sample containers were received for the tests requested	X			
Samples were received within holding time	X			
Sufficient amount of sample is provided for requested analyses	X			
VOA vials do not have headspace or a bubble >6mm (1/4" diameter)			X	
Appropriate preservatives were used for the tests requested			X	
pH of inorganic samples checked and is within method specification			X	
If no, attach Inorganic Sample pH Adjustment Form			X	

ANOMALY / NCR SUMMARY

Young, Kirk

From: Azzolina, Nick [NAzzolina@ensr.aecom.com]
Sent: Tuesday, November 13, 2007 5:12 PM
To: Anderson, Chris
Cc: Young, Kirk; Geiger, Steve
Subject: Anacostia River sediment samples

Chris,

TestAmerica should have received 36 samples last week from the Anacostia River. Please analyze the following 15 samples for grain size, total solids, pH, and ammonia, per the work order.

AR02
AR03
AR05
AR07
AR08
AR10
AR13
AR14
AR16
AR17
AR21
AR25
AR26
AR27
AR36

Thanks very much,

Nick

12/13/2007

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

Last Page of this Document

Appendix I: EERC Laboratory Report: Total and SPME PAHs, TOC and SOC

Report of Laboratory Analysis

January 11, 2008

Dr. Steven B. Hawthorne
Energy and Environmental Research Center, Campus Box 9018
University of North Dakota, Grand Forks, North Dakota 58201
701-777-5256

Narrative

Thirty-six sediment samples variously labeled AR1 to AR36 were received on November 7, 8, and 9, 2007. All samples were in good condition, and had measured temperatures of 3 to 10 ^{o}C, as noted on the attached chain of custody forms. Each sample was subjected to an initial analysis to estimate the "34" PAH concentrations. These data were provided to ENSR on an informal basis, and were used to select 15 sediments for the determination of pore water PAH concentrations, total PAH concentrations, total organic carbon, soot organic carbon, and dissolved organic carbon as described in the methods document.

All analyses for reported data met all QA/QC criteria listed in the *Quality Assurance Project Plan (QAPP) for Evaluating PAH Toxicity and Bioavailability in Sediments* and the method specifications listed in the *Generic Sediment Sample Collection and Analysis Work Plan for Evaluating PAH Bioavailability*. All pore water and total PAH analyses were performed in quadruplicate. All extracts met the 70 to 120% recovery criteria and met all QA/QC criteria.

Data qualifiers are listed with each determination, and include "J" (for values estimated below the lowest calibration concentrations), "E" (for values exceeding the highest calibration concentration, and "R" (for values rejected on the basis of the reasons described in the footnotes listed at the bottom of the table).

Chain of Custody Record

No. 0368

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: Fed Ex
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: EERC
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Analysis Requested: Soluble PAH	Pore water PAH	TOC	SOC	DOC	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR11 7C 8C	11/6/07	1021	sed	2							
AR13 9C 9C		1047									
AR14 9C 9C		1101									
AR15 10C 10C		1116									
AR16 9C 9C		1153									
AR17 7C 7C		1505									
AR18 8C 8C		1446									
AR19 8C 9C		1424									
AR26 5C 7C		1520									

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen Jones	[signature]	11/7/07	9:30a
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level	Turnaround	Sample Receipt	
Level I ___	Routine ___	Total # Containers Received?	
Level II ___	24 Hour ___	COC Seals Present?	
Level III ___	1 Week ___	COC Seals Intact?	
Other ___	Other ___	Received Containers Intact?	
		Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

No 0329

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St Suite 204	Sampler (Print Name): H. Albert
Ithaca NY 14850	Shipment Method: FedEx
Phone: 607-277-5716	Airbill Number:
Fax: 607-277-9057	Laboratory Receiving: EERC

Page 1 of 1

Purchase Order #:

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Analysis Requested: Soluble PAHs / Porewater PAHs / TOC / SOC / DOC	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
✓AR24. 7C 6C	11/7/07	0802	Sed	2			
✓AR25. 6C 6C		0816					
✓AR20. 7C 7C		0902					
✓AR27. 8C 8C		0913					
✓AR21. 7C 6C		0920					
✓AR28. 7C 7C		1002					
✓AR29. 8C 8C		1010					
✓AR01. 8C 9C		1015					
✓AR02. 8C 7C		1025					
✓AR03. 7C 7C		1032				No H_2O	
✓AR30. 5C 4C		1110					
✓AR31. 7C 4C		1125					
✓AR22. 7C 8C		1134					
✓AR23. 9C 9C		1300					
✓AR04. 5C 6C		1315					
✓AR05. 6C 6C		1330					

Relinquished by: (Signature) Helen d. J...	Received by: (Signature) [signature]	Date: 11/8/07	Time: 9:35a
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level	Turnaround	Sample Receipt	
		Total # Containers Received?	
Level I ☐	Routine ☐	COC Seals Present?	
Level II ☐	24 Hour ☐	COC Seals Intact?	
Level III ☐	1 Week ☐	Received Containers Intact?	
Other ☐	Other ____	Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

Chain of Custody Record

No 0330

The RETEC Group, Inc.
1001 W. Seneca Street, Suite 204 • Ithaca, NY 14850-3342
(607) 277-5716 Phone • (607) 277-9057 Fax
www.retec.com

RETEC

Project Name: Anacostia River	Project Number: 09000-422-100
Send Report To: Helen Jones	Sampler (Print Name): H. Jones
Address: 1001 W. Seneca St.	Sampler (Print Name): H. Albert
Suite 204	Shipment Method: FedEx
Ithaca NY 14850	Airbill Number:
Phone: 607-277-5716	Laboratory Receiving: EERC
Fax: 607-277-9057	

Page 1 of 1

Purchase Order #: _____

Field Sample ID	Sample Date	Sample Time	Sample Matrix	Number of Containers	Comments, Special Instructions, etc.	Lab Sample ID (to be completed by lab)
AR12 6C 5C	11/8/07	0847	Sed	2		
AR32 4C 4C		0855				
AR08 5C 7C		0900				
AR10 5C 6C		0910				
AR09 5C 6C		0916				
AR06 5C 6C		0955				
AR07 4C 5C		1002				
AR33 5C 5C		1009				
AR34 4C 7C		1013				
AR35 3C 7C		1022				
AR36 5C 4C		1028				

Analysis Requested: Sulfur, PAH, Pore water PAH, SOC, TOC, DOC

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Helen d. Jones			
Relinquished by: (Signature)	Received by: (Signature)	11/9/07	9:30a
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
		Date:	Time:

Sample Custodian Remarks (Completed By Laboratory):

QA/QC Level	Turnaround
Level I ☐	Routine ☐
Level II ☐	24 Hour ☐
Level III ☐	1 Week ☐
Other ☐	Other _____

Sample Receipt	
Total # Containers Received?	
COC Seals Present?	
COC Seals Intact?	
Received Containers Intact?	
Temperature?	

White: Lab Copy Yellow: PM Copy Pink: Field Copy Gold: PM/QA/QC Copy

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University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

Anacostia River TOC and SOC values

sample	analysis date	mean TOC wt. % (dry)	SD	mean SOC wt. % (dry)	SD
AR02	12/4/2007	9.42	0.12	3.70	0.13
AR03	12/4/2007	0.88	0.14	0.18	0.03
AR05	12/4/2007	3.30	0.17	0.30	0.04
AR07	12/4/2007	3.93	0.06	0.36	0.08
AR08	12/4/2007	3.69	0.03	0.42	0.07
AR10	12/4/2007	3.68	0.04	0.35	0.03
AR13	12/4/2007	4.89	0.26	0.67	0.30
AR14	12/4/2007	3.91	0.07	0.45	0.03
AR16	12/4/2007	6.64	0.32	1.64	0.28
AR17	12/4/2007	3.50	0.19	0.57	0.09
AR21	12/4/2007	5.01	0.21	0.31	0.01
AR25	12/4/2007	3.86	0.23	0.47	0.03
AR26	12/4/2007	11.11	0.59	6.38	0.23
AR27	12/4/2007	2.97	0.10	0.29	0.03
AR36	12/4/2007	4.45	0.12	0.54	0.06

SD based on 3 replicate CHN determinations

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Anacostia dissolved organic carbon (DOC) values

sample	analysis date	DOC mg/L
AR02	12/14/2008	15.5
AR03	12/14/2008	4.8
AR05	12/14/2008	4.1
AR07	12/14/2008	6.9
AR08	12/14/2008	4.5
AR10	12/14/2008	3.4
AR13	12/14/2008	3.3
AR14	12/14/2008	4.1
AR16	12/14/2008	2.8
AR17	12/14/2008	4.0
AR21	12/14/2008	12.8
AR25	12/14/2008	4.2
AR26	12/14/2008	3.5
AR27	12/14/2008	7.9
AR36	12/14/2008	5.0

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EERC ID	71A-1		71A-2		71A-3		71A-4	
RETEC Sample Collection Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Sample Receipt Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Run Date	11/14/2007		11/14/2007		11/14/2007		11/14/2007	
EERC Run Number	2361A15.D		2361A16.D		2361A17.D		2361A18.D	
AQUATOX Sample ID								
Sample Name	AR02		AR02		AR02		AR02	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.500		1.500		1.510		1.500	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	1.404	J	1.356	J	1.423	J	1.401	J
2-methylnaphthalene	0.500	J	0.465	J	0.505	J	0.546	J
1-methylnaphthalene	1.770		1.797		1.857		1.915	
C2 naphthalenes	95.081		86.353		102.332		100.395	
C3 naphthalenes	75.255		64.974		84.872		79.076	
C4 naphthalenes	28.060		22.906		34.475		27.436	
acenaphthylene	8.649		8.604		9.088		10.409	
acenaphthene	45.691		46.929		49.095		48.587	
fluorene	15.910		16.128		17.111		16.627	
C1 fluorenes	12.968		12.346		14.212		14.104	
C2 fluorenes	7.036		6.508		8.106		7.432	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	31.058		31.469		35.272		34.429	
anthracene	3.150		3.197		3.756		3.665	
C1 phenanthrenes/anthracenes	24.209		23.520		28.755		26.669	
C2 phenanthrenes/anthracenes	11.540		10.986		13.570		12.299	
C3 phenanthrenes/anthracenes	2.457	J	2.803	J	2.945	J	2.694	J
C4 phenanthrenes/anthracenes	1.902	J	2.625	J	2.232	J	2.129	J
fluoranthene	5.772		5.699		7.810		6.652	
pyrene	9.767		9.408		13.564		11.189	
C1 fluoranthenes/pyrenes	5.035		5.675		7.850		6.172	
benz[a]anthracene	0.468		0.497		0.571		0.506	
chrysene	0.410		0.478		0.480		0.468	
C1 chrysenes	0.139		0.135		0.184		0.163	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	0.209		0.191		0.305		0.241	
benzo[e]pyrene	0.249		0.348		0.235		0.217	
benzo[a]pyrene	0.360		0.464		0.583		0.392	
perylene	0.030		0.021	J	0.021	J	0.025	J
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

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EERC ID	84A-1		84A-2		84A-3		84A-4	
RETEC Sample Collection Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Sample Receipt Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Run Date	11/20/2007		11/20/2007		11/20/2007		11/20/2007	
EERC Run Number	2365A21.D		2365A22.D		2365A23.D		2365A24.D	
AQUATOX Sample ID								
Sample Name	AR02		AR02		AR02		AR02	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.510		1.500		1.500		1.500	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	1.008	J	1.052	J	1.198	J	1.077	J
2-methylnaphthalene	0.399	J	0.332	J	0.395	J	0.372	J
1-methylnaphthalene	1.720		1.574		1.721		1.603	
C2 naphthalenes	63.645		65.445		72.544		71.361	
C3 naphthalenes	43.096		42.936		51.060		49.193	
C4 naphthalenes	18.058		22.379		25.692		23.279	
acenaphthylene	7.433		6.555		7.508		7.693	
acenaphthene	41.657		41.846		43.873		43.099	
fluorene	12.163		12.608		13.469		13.146	
C1 fluorenes	9.484		10.391		11.847		10.875	
C2 fluorenes	4.595		5.364		6.782		6.248	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	20.988		21.417		23.366		23.521	
anthracene	2.402		2.549		2.907		2.796	
C1 phenanthrenes/anthracenes	13.061		14.764		16.755		16.359	
C2 phenanthrenes/anthracenes	3.014	J	3.935	J	4.536	J	4.642	J
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	2.644		2.823		3.202		3.241	
pyrene	4.168		4.508		5.221		5.366	
C1 fluoranthenes/pyrenes	1.741		2.090		2.373		2.560	
benz[a]anthracene	0.180		0.175		0.283		0.255	
chrysene	0.161		0.208		0.258		0.279	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

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EERC ID	83A-1		83A-2		83A-3		83A-4	
RETEC Sample Collection Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Sample Receipt Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Run Date	11/20/2007		11/20/2007		11/20/2007		11/20/2007	
EERC Run Number	2365A15.D		2365A16.D		2365A17.D		2365A18.D	
AQUATOX Sample ID								
Sample Name	AR03		AR03		AR03		AR03	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.510		1.500		1.500		1.510	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	0.105	J	0.083	J	0.079	J	0.082	J
2-methylnaphthalene	ND		ND		ND		0.055	J
1-methylnaphthalene	0.299	J	0.274	J	0.323	J	0.308	J
C2 naphthalenes	1.299	J	1.380	J	1.218	J	1.358	J
C3 naphthalenes	1.576	J	1.392	J	1.359	J	1.176	J
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	0.119	J	0.157	J	0.169	J	0.147	J
acenaphthene	0.600	J	0.617	J	0.671	J	0.587	J
fluorene	0.198	J	0.185	J	0.238	J	0.241	J
C1 fluorenes	0.744	J	0.633	J	0.590	J	0.586	J
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.181	J	0.191	J	0.206	J	0.197	J
anthracene	0.064	J	0.090	J	0.066	J	0.083	J
C1 phenanthrenes/anthracenes	0.478	J	0.446	J	0.466	J	0.367	J
C2 phenanthrenes/anthracenes	0.353	J	0.318	J	0.281	J	0.267	J
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.087	J	0.086	J	0.086	J	0.087	J
pyrene	0.120	J	0.118	J	0.126	J	0.133	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

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University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	74A-1		74A-2		74A-3		74A-4	
RETEC Sample Collection Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Sample Receipt Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Run Date	11/15/2007		11/15/2007		11/15/2007		11/15/2007	
EERC Run Number	2362A15.D		2362A16.D		2362A17.D		2362A18.D	
AQUATOX Sample ID								
Sample Name	AR05		AR05		AR05		AR05	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.510		1.500		1.510		1.510	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	ND		ND		ND		ND	
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	ND		ND		ND		ND	
fluorene	ND		ND		ND		ND	
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.100	J	0.101	J	0.135	J	0.095	J
anthracene	ND		ND		ND		ND	
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.026	J	0.033	J	0.025	J	0.039	J
pyrene	ND		ND		ND		ND	
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

Energy and Environmental Research Center, GC/MS Lab
University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	73A-1		73A-2		73A-3		73A-4	
RETEC Sample Collection Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Sample Receipt Date	11/9/2007		11/9/2007		11/9/2007		11/9/2007	
EERC Run Date	11/15/2007		11/15/2007		11/15/2007		11/15/2007	
EERC Run Number	2362A09.D		2362A10.D		2362A11.D		2362A12.D	
AQUATOX Sample ID								
Sample Name	AR07		AR07		AR07		AR07	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.500		1.510		1.510		1.500	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	ND		ND		ND		ND	
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	0.024	J	0.070	J	0.060	J	0.020	J
acenaphthene	0.080	J	0.092	J	0.062	J	0.053	J
fluorene	0.061	J	0.105	J	0.073	J	0.064	J
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.148	J	0.145	J	0.138	J	0.104	J
anthracene	ND		ND		ND		ND	
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.052	J	0.070	J	0.057	J	0.061	J
pyrene	ND		0.053	J	ND		0.046	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

Energy and Environmental Research Center, GC/MS Lab
University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	72A-1		72A-2		72A-3		72A-4	
RETEC Sample Collection Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Sample Receipt Date	11/9/2007		11/9/2007		11/9/2007		11/9/2007	
EERC Run Date	11/15/2007		11/15/2007		11/15/2007		11/15/2007	
EERC Run Number	2362A03.D		2362A04.D		2362A05.D		2362A06.D	
AQUATOX Sample ID								
Sample Name	AR08		AR08		AR08		AR08	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.500		1.500		1.510		1.500	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	ND		ND		ND		ND	
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	0.140	J	0.086	J	0.028	J	0.067	J
fluorene	0.057	J	0.120	J	0.052	J	0.028	J
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.123	J	0.072	J	0.037	J	0.039	J
anthracene	ND		ND		ND		ND	
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	ND		ND		ND		ND	
pyrene	ND		ND		ND		ND	
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

Energy and Environmental Research Center, GC/MS Lab
University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	75A-1		75A-2		75A-3		75A-4	
RETEC Sample Collection Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Sample Receipt Date	11/9/2007		11/9/2007		11/9/2007		11/9/2007	
EERC Run Date	11/16/2007		11/16/2007		11/16/2007		11/16/2007	
EERC Run Number	2363A09.D		2363A10.D		2363A11.D		2363A12.D	
AQUATOX Sample ID								
Sample Name	AR10		AR10		AR10		AR10	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.500		1.510		1.510		1.510	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	ND		ND		ND		ND	
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	ND		ND		ND		ND	
fluorene	ND		ND		ND		ND	
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.071	J	0.067	J	0.053	J	0.044	J
anthracene	0.033	J	0.012	J	0.010	J	ND	
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.046	J	0.042	J	0.040	J	0.041	J
pyrene	0.045	J	0.039	J	0.034	J	0.028	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

Energy and Environmental Research Center, GC/MS Lab
University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	76A-1		76A-2		76A-3		76A-4	
RETEC Sample Collection Date	11/6/2007		11/6/2007		11/6/2007		11/6/2007	
EERC Sample Receipt Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Run Date	11/16/2007		11/16/2007		11/16/2007		11/16/2007	
EERC Run Number	2363A15.D		2363A16.D		2363A17.D		2363A18.D	
AQUATOX Sample ID								
Sample Name	AR13		AR13		AR13		AR13	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.510		1.500		1.500		1.510	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	0.028	J	0.047	J	0.046	J	0.051	J
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	ND		ND		ND		ND	
fluorene	0.050	J	0.085	J	0.075	J	0.053	J
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.115	J	0.097	J	0.103	J	0.098	J
anthracene	0.029	J	0.014	J	0.021	J	0.021	J
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.048	J	0.048	J	0.055	J	0.045	J
pyrene	0.032	J	0.038	J	0.048	J	0.047	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

Energy and Environmental Research Center, GC/MS Lab
University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	77A-1		77A-2		77A-3		77A-4	
RETEC Sample Collection Date	11/6/2007		11/6/2007		11/6/2007		11/6/2007	
EERC Sample Receipt Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Run Date	11/16/2007		11/16/2007		11/16/2007		11/16/2007	
EERC Run Number	2363A21.D		2363A22.D		2363A23.D		2363A24.D	
AQUATOX Sample ID								
Sample Name	AR14		AR14		AR14		AR14	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.510		1.510		1.510		1.500	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	0.028	J	0.026	J	0.020	J	0.022	J
1-methylnaphthalene	0.025	J	0.031	J	0.022	J	0.033	J
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	ND		ND		ND		ND	
fluorene	ND		ND		ND		ND	
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.069	J	0.075	J	0.088	J	0.092	J
anthracene	0.013	J	0.019	J	0.021	J	0.016	J
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.057	J	0.046	J	0.048	J	0.047	J
pyrene	0.039	J	0.036	J	0.048	J	0.042	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

Energy and Environmental Research Center, GC/MS Lab
University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	78A-1		78A-2		78A-3		78A-4	
RETEC Sample Collection Date	11/6/2007		11/6/2007		11/6/2007		11/6/2007	
EERC Sample Receipt Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Run Date	11/19/2007		11/19/2007		11/19/2007		11/19/2007	
EERC Run Number	2364A03.D		2364A04.D		2364A05.D		2364A06.D	
AQUATOX Sample ID								
Sample Name	AR16		AR16		AR16		AR16	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.510		1.500		1.500		1.510	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	ND		ND		ND		ND	
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	ND		ND		ND		ND	
fluorene	ND		ND		ND		ND	
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.032	J	0.032	J	0.021	J	0.030	J
anthracene	0.008	J	0.012	J	0.013	J	0.010	J
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.025	J	0.032	J	0.016	J	0.020	J
pyrene	0.017	J	0.016	J	0.015	J	0.012	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

Energy and Environmental Research Center, GC/MS Lab
University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	68A-1		68A-2		68A-3		68A-4	
RETEC Sample Collection Date	11/6/2007		11/6/2007		11/6/2007		11/6/2007	
EERC Sample Receipt Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Run Date	11/14/2007		11/14/2007		11/14/2007		11/14/2007	
EERC Run Number	2361A03.D		2361A04.D		2361A05.D		2361A06.D	
AQUATOX Sample ID								
Sample Name	AR17		AR17		AR17		AR17	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.500		1.500		1.500		1.500	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	ND		ND		ND		ND	
C2 naphthalenes	0.397	J	0.577	J	0.529	J	0.395	J
C3 naphthalenes	0.387	J	0.489	J	0.353	J	0.326	J
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	ND		ND		ND		ND	
fluorene	ND		ND		ND		ND	
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.207	J	0.168	J	0.113	J	0.096	J
anthracene	ND		ND		ND		ND	
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.065	J	0.054	J	0.044	J	0.043	J
pyrene	ND		ND		ND		ND	
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

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University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	79A-1		79A-2		79A-3		79A-4	
RETEC Sample Collection Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Sample Receipt Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Run Date	11/19/2007		11/19/2007		11/19/2007		11/19/2007	
EERC Run Number	2364A09.D		2364A10.D		2364A11.D		2364A12.D	
AQUATOX Sample ID								
Sample Name	AR21		AR21		AR21		AR21	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.510		1.500		1.500		1.510	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	ND		ND		ND		ND	
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	ND		ND		ND		ND	
fluorene	ND		ND		ND		ND	
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.064	J	0.069	J	0.046	J	0.062	J
anthracene	ND		ND		ND		ND	
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.038	J	0.029	J	0.027	J	0.027	J
pyrene	0.023	J	0.031	J	0.020	J	0.020	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

Energy and Environmental Research Center, GC/MS Lab
University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	80A-1		80A-2		80A-3		80A-4	
RETEC Sample Collection Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Sample Receipt Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Run Date	11/19/2007		11/19/2007		11/19/2007		11/19/2007	
EERC Run Number	2364A15.D		2364A16.D		2364A17.D		2364A18.D	
AQUATOX Sample ID								
Sample Name	AR25		AR25		AR25		AR25	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.510		1.510		1.500		1.510	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	ND		ND		ND		ND	
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	0.026	J	0.043	J	0.027	J	0.029	J
acenaphthene	0.098	J	0.013	J	0.024	J	0.039	J
fluorene	ND		ND		ND		ND	
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.031	J	0.046	J	0.063	J	0.040	J
anthracene	ND		ND		ND		ND	
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.024	J	0.029	J	0.040	J	0.029	J
pyrene	0.022	J	0.031	J	0.028	J	0.022	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

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University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	69A-1		69A-2		69A-3		69A-4	
RETEC Sample Collection Date	11/6/2007		11/6/2007		11/6/2007		11/6/2007	
EERC Sample Receipt Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Run Date	11/14/2007		11/14/2007		11/14/2007		11/14/2007	
EERC Run Number	2361A09.D		2361A10.D		2361A11.D		2361A12.D	
AQUATOX Sample ID								
Sample Name	AR26		AR26		AR26		AR26	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.510		1.510		1.510		1.500	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	0.089	J	0.067	J	0.091	J	0.069	J
2-methylnaphthalene	0.014	J	0.014	J	0.017	J	0.016	J
1-methylnaphthalene	0.035	J	0.024	J	0.034	J	0.031	J
C2 naphthalenes	0.453	J	0.379	J	0.447	J	0.369	J
C3 naphthalenes	0.302	J	0.255	J	0.342	J	0.273	J
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	ND		ND		ND		ND	
fluorene	0.068	J	0.060	J	0.056	J	0.046	J
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.079	J	0.084	J	0.076	J	0.078	J
anthracene	ND		ND		ND		ND	
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.075	J	0.079	J	0.071	J	0.070	J
pyrene	0.051	J	0.052	J	0.042	J	0.052	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

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University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	81A-1		81A-2		81A-3		81A-4	
RETEC Sample Collection Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Sample Receipt Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Run Date	11/20/2007		11/20/2007		11/20/2007		11/20/2007	
EERC Run Number	2365A03.D		2365A04.D		2365A05.D		2365A06.D	
AQUATOX Sample ID								
Sample Name	AR27		AR27		AR27		AR27	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.510		1.500		1.500		1.500	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	ND		ND		ND		ND	
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	ND		ND		ND		ND	
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	0.061	J	0.029	J	0.065	J	0.050	J
fluorene	ND		ND		ND		ND	
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	ND		ND		ND		ND	
anthracene	ND		ND		ND		ND	
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.040	J	0.029	J	0.010	J	0.023	J
pyrene	0.019	J	0.036	J	0.010	J	0.026	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

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15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

EERC ID	82A-1		82A-2		82A-3		82A-4	
RETEC Sample Collection Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Sample Receipt Date	11/9/2007		11/9/2007		11/9/2007		11/9/2007	
EERC Run Date	11/20/2007		11/20/2007		11/20/2007		11/20/2007	
EERC Run Number	2365A09.D		2365A10.D		2365A11.D		2365A12.D	
AQUATOX Sample ID								
Sample Name	AR36		AR36		AR36		AR36	
Treatment	Pore Water		Pore Water		Pore Water		Pore Water	
Sample Weight, g	1.500		1.510		1.520		1.500	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ng/g		ng/g		ng/g		ng/g	
naphthalene	0.096	J	0.104	J	0.054	J	0.072	J
2-methylnaphthalene	ND		ND		ND		ND	
1-methylnaphthalene	ND		ND		ND		ND	
C2 naphthalenes	ND		ND		ND		ND	
C3 naphthalenes	ND		ND		ND		ND	
C4 naphthalenes	ND		ND		ND		ND	
acenaphthylene	ND		ND		ND		ND	
acenaphthene	ND		ND		ND		ND	
fluorene	0.046	J	0.027	J	0.036	J	0.018	J
C1 fluorenes	ND		ND		ND		ND	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	0.054	J	0.053	J	0.045	J	0.043	J
anthracene	0.013	J	0.007	J	0.014	J	0.008	J
C1 phenanthrenes/anthracenes	ND		ND		ND		ND	
C2 phenanthrenes/anthracenes	ND		ND		ND		ND	
C3 phenanthrenes/anthracenes	ND		ND		ND		ND	
C4 phenanthrenes/anthracenes	ND		ND		ND		ND	
fluoranthene	0.039	J	0.036	J	0.038	J	0.038	J
pyrene	0.024	J	0.031	J	0.026	J	0.022	J
C1 fluoranthenes/pyrenes	ND		ND		ND		ND	
benz[a]anthracene	ND		ND		ND		ND	
chrysene	ND		ND		ND		ND	
C1 chrysenes	ND		ND		ND		ND	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	ND		ND		ND		ND	
benzo[e]pyrene	ND		ND		ND		ND	
benzo[a]pyrene	ND		ND		ND		ND	
perylene	ND		ND		ND		ND	
indeno[1,2,3-cd]pyrene	ND		ND		ND		ND	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	ND		ND		ND		ND	

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701-777-5000

EERC ID	**71C-1**		**71C-2**		**71C-3**		**71C-4**	
RETEC Sample Collection Date	**11/7/2007**		**11/7/2007**		**11/7/2007**		**11/7/2007**	
EERC Sample Receipt Date	**11/8/2007**		**11/8/2007**		**11/8/2007**		**11/8/2007**	
EERC Run Date	**11/16/2007**		**11/16/2007**		**11/16/2007**		**11/16/2007**	
EERC Run Number	**2679A08.D**		**2679A09.D**		**2679A10.D**		**2679A11.D**	
AQUATOX Sample ID								
Sample Name	**AR02**		**AR02**		**AR02**		**AR02**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**1.04**		**1.04**		**1.04**		**1.04**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	13.129		11.476		12.358		12.478	
2-methylnaphthalene	12.374		10.608		11.354		12.047	
1-methylnaphthalene	5.812		5.491		5.471		5.566	
C2 naphthalenes	135.731	E	137.585	E	138.545	E	134.448	E
C3 naphthalenes	97.987	E	96.127	E	101.574	E	97.233	E
C4 naphthalenes	57.139		55.542		59.419		56.073	
acenaphthylene	14.975		18.504		18.401		16.650	
acenaphthene	47.840		51.529		48.181		46.279	
fluorene	23.503		24.389		23.568		22.706	
C1 fluorenes	47.608	E	47.932	E	49.894	E	49.241	E
C2 fluorenes	67.426		71.687		77.687		72.123	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	98.078		99.714		101.279		99.657	
anthracene	55.342		59.139		60.522		57.275	
C1 phenanthrenes/anthracenes	143.282	E	146.282	E	156.808	E	146.180	E
C2 phenanthrenes/anthracenes	277.893	E	300.395	E	320.124	E	296.851	E
C3 phenanthrenes/anthracenes	139.771		157.261		159.258		150.725	
C4 phenanthrenes/anthracenes	35.719	E	27.207		26.298		25.931	
fluoranthene	57.646		57.324		57.312		55.807	
pyrene	93.833		92.822		94.576	E	90.505	
C1 fluoranthenes/pyrenes	116.370	E	120.366	E	127.688	E	119.794	E
benz[a]anthracene	29.831		29.401		30.463		29.386	
chrysene	33.322		34.208		35.809		33.536	
C1 chrysenes	95.667		97.607		102.493		96.861	
C2 chrysenes	76.459		84.676		84.155		77.093	
C3 chrysenes	29.535		36.723		31.392		28.073	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	28.985		27.261		27.986		26.885	
benzo[e]pyrene	14.043		12.399		12.939		12.901	
benzo[a]pyrene	40.066		35.365		37.680		37.596	
perylene	8.533		7.382		7.575		7.142	
indeno[1,2,3-cd]pyrene	30.311	E	30.122	E	30.491	E	28.583	E
dibenz[ah]anthracene	6.745		6.896		7.685		7.313	
benzo[ghi]perylene	24.962	E	24.197	E	24.285	E	22.920	E

Total NOAA PAHs	1960		2018		2083		1976	
Total EPA PAHs (16)	599		602		611		588	

Surrogate recoveries								
biphenyl-d10	105.9%		104.5%		105.3%		103.5%	
benzo[b]fluoranthene-d12	99.7%		95.2%		93.2%		95.7%	

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701-777-5000

EERC ID	**83C-1**		**83C-2**		**83C-3**		**83C-4**	
RETEC Sample Collection Date	**11/7/2007**		**11/7/2007**		**11/7/2007**		**11/7/2007**	
EERC Sample Receipt Date	**11/8/2007**		**11/8/2007**		**11/8/2007**		**11/8/2007**	
EERC Run Date	**11/27/2007**		**11/27/2007**		**11/27/2007**		**11/27/2007**	
EERC Run Number	**2682A01.D**		**2682A02.D**		**2682A03.D**		**2682A04.D**	
AQUATOX Sample ID								
Sample Name	**AR03**		**AR03**		**AR03**		**AR03**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**1.5**		**1.5**		**1.5**		**1.5**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.476		0.391		1.525		0.510	
2-methylnaphthalene	0.589		0.382		2.024		0.745	
1-methylnaphthalene	1.344		0.970		2.825		1.506	
C2 naphthalenes	2.821		2.071		4.788		3.494	
C3 naphthalenes	1.252		1.149		2.206		1.775	
C4 naphthalenes	0.499		0.537		1.114		0.821	
acenaphthylene	0.125		0.120		0.589		0.267	
acenaphthene	0.925		0.871		1.594		1.025	
fluorene	0.480		0.576		0.650		0.493	
C1 fluorenes	0.558		0.604		1.308		0.820	
C2 fluorenes	0.858		1.144		2.493		1.495	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	1.514		3.597		2.126		1.660	
anthracene	0.989		2.587		1.805		1.256	
C1 phenanthrenes/anthracenes	1.586		2.208		4.093		2.199	
C2 phenanthrenes/anthracenes	1.735		2.198		5.429		2.701	
C3 phenanthrenes/anthracenes	1.680		1.900		4.847		2.585	
C4 phenanthrenes/anthracenes	0.302		0.358		0.975		0.744	
fluoranthene	0.851		4.027		1.118		0.714	
pyrene	0.973		3.517		1.645		0.951	
C1 fluoranthenes/pyrenes	0.770		1.993		2.681		1.177	
benz[a]anthracene	0.308		1.410		0.632		0.302	
chrysene	ND		2.031		0.996		0.539	
C1 chrysenes	1.458		3.362		3.861		1.748	
C2 chrysenes	ND		ND		ND		ND	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	1.248		2.481		0.703		0.569	
benzo[e]pyrene	0.672		1.069		0.406		0.320	
benzo[a]pyrene	0.643		1.703		0.720		0.424	
perylene	0.210		0.399		0.156		0.066	
indeno[1,2,3-cd]pyrene	0.173		0.664		0.197		0.147	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	0.316		0.820		0.311		0.250	

Total NOAA PAHs	25		45		54		31	
Total EPA PAHs (16)	9		25		15		9	

Surrogate recoveries								
biphenyl-d10	113.1%		108.4%		106.1%		104.8%	
benzo[b]fluoranthene-d12	94.8%		112.4%		104.5%		118.0%	

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EERC ID	**74C-1**		**74C-2**		**74C-3**		**74C-4**	
RETEC Sample Collection Date	**11/7/2007**		**11/7/2007**		**11/7/2007**		**11/7/2007**	
EERC Sample Receipt Date	**11/8/2007**		**11/8/2007**		**11/8/2007**		**11/8/2007**	
EERC Run Date	**11/27/2007**		**11/27/2007**		**11/27/2007**		**11/27/2007**	
EERC Run Number	**2682A05.D**		**2682A06.D**		**2682A07.D**		**2682A08.D**	
AQUATOX Sample ID								
Sample Name	**AR05**		**AR05**		**AR05**		**AR05**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**0.75**		**0.75**		**0.75**		**0.75**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.222		0.223		0.240		0.200	
2-methylnaphthalene	0.207		0.207		0.196		0.195	
1-methylnaphthalene	0.093		0.142		0.107		0.095	
C2 naphthalenes	1.694		1.514		1.175		1.140	
C3 naphthalenes	1.184		1.252		1.149		1.169	
C4 naphthalenes	1.281		1.642		1.309		1.318	
acenaphthylene	0.288		0.405		0.298		0.276	
acenaphthene	0.225		0.309		0.351		0.180	
fluorene	0.299		0.370		0.384		0.235	
C1 fluorenes	0.545		0.742		0.786		0.624	
C2 fluorenes	3.085		2.970		2.957		2.457	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	1.968		3.666		3.299		1.895	
anthracene	1.240		2.192		1.932		1.070	
C1 phenanthrenes/anthracenes	1.940		2.858		2.692		1.933	
C2 phenanthrenes/anthracenes	4.574		5.624		5.296		4.570	
C3 phenanthrenes/anthracenes	8.121		8.345		7.480		6.482	
C4 phenanthrenes/anthracenes	2.890		2.238		2.381		2.175	
fluoranthene	3.358		7.463		5.717		4.059	
pyrene	2.924		6.076		4.695		3.387	
C1 fluoranthenes/pyrenes	2.969		4.815		4.358		3.120	
benz[a]anthracene	1.226		2.603		2.271		1.540	
chrysene	2.825		4.897		4.078		3.173	
C1 chrysenes	5.725		8.366		7.464		5.799	
C2 chrysenes	6.580		7.194		7.444		5.904	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	4.936		8.153		6.967		5.733	
benzo[e]pyrene	1.831		2.770		2.257		1.863	
benzo[a]pyrene	2.778		5.204		4.483		3.428	
perylene	0.713		1.223		1.065		0.816	
indeno[1,2,3-cd]pyrene	1.744		3.086		3.039		2.266	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	1.749		2.991		2.590		2.092	

Total NOAA PAHs	69		100		88		69	
Total EPA PAHs (16)	26		48		40		30	

Surrogate recoveries								
biphenyl-d10	103.5%		103.8%		104.4%		104.2%	
benzo[b]fluoranthene-d12	116.7%		108.3%		115.9%		111.3%	

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EERC ID	73C-1		73C-2		73C-3		73C-4	
RETEC Sample Collection Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Sample Receipt Date	11/9/2007		11/9/2007		11/9/2007		11/9/2007	
EERC Run Date	11/16/2007		11/16/2007		11/16/2007		11/16/2007	
EERC Run Number	2679A15.D		2679A16.D		2679A17.D		2679A18.D	
AQUATOX Sample ID								
Sample Name	AR07		AR07		AR07		AR07	
Treatment	Soxhlet		Soxhlet		Soxhlet		Soxhlet	
Sample Weight, g	0.89		0.89		0.89		0.89	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ug/g		ug/g		ug/g		ug/g	
naphthalene	0.128		0.105	J	0.137		0.130	
2-methylnaphthalene	0.137		0.130		0.140		0.131	
1-methylnaphthalene	0.074		0.059	J	0.074		0.077	
C2 naphthalenes	1.113		0.970		1.081		1.108	
C3 naphthalenes	1.444		1.414		1.556		1.438	
C4 naphthalenes	1.747		1.773		1.931		1.921	
acenaphthylene	0.316		0.268		0.250		0.238	
acenaphthene	0.156		0.163		0.175		0.180	
fluorene	0.224		0.208		0.219		0.191	
C1 fluorenes	0.549		0.543		0.568		0.560	
C2 fluorenes	2.647		2.718		2.845		2.408	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	1.935		1.721		2.010		1.707	
anthracene	0.961		0.887		0.958		0.811	
C1 phenanthrenes/anthracenes	1.807		1.675		1.828		1.659	
C2 phenanthrenes/anthracenes	6.891		6.852		7.592		6.700	
C3 phenanthrenes/anthracenes	7.475		7.124		7.656		6.950	
C4 phenanthrenes/anthracenes	2.069		1.951		1.993		1.984	
fluoranthene	3.675		3.348		3.866		3.543	
pyrene	3.220		2.884		3.313		3.102	
C1 fluoranthenes/pyrenes	2.575		2.397		2.666		2.586	
benz[a]anthracene	1.363		1.191		1.378		1.304	
chrysene	2.569		2.351		2.561		2.520	
C1 chrysenes	4.408		3.983		4.318		4.229	
C2 chrysenes	4.420		4.265		4.191		4.167	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	3.071		2.879		3.036		3.012	
benzo[e]pyrene	1.186		1.100		1.148		1.135	
benzo[a]pyrene	2.009		1.792		1.953		1.889	
perylene	0.906		0.807		0.818		0.841	
indeno[1,2,3-cd]pyrene	2.573		2.509		2.682		2.642	
dibenz[ah]anthracene	ND		ND		ND		0.508	
benzo[ghi]perylene	1.761		1.666		1.762		1.772	

Total NOAA PAHs	63		60		65		61	
Total EPA PAHs (16)	24		22		24		24	

Surrogate recoveries								
biphenyl-d10	109.2%		101.3%		101.8%		103.1%	
benzo[b]fluoranthene-d12	101.7%		99.1%		99.5%		96.6%	

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EERC ID	**72C-1**		**72C-2**		**72C-3**		**72C-4**	
RETEC Sample Collection Date	**11/8/2007**		**11/8/2007**		**11/8/2007**		**11/8/2007**	
EERC Sample Receipt Date	**11/9/2007**		**11/9/2007**		**11/9/2007**		**11/9/2007**	
EERC Run Date	**11/16/2007**		**11/16/2007**		**11/16/2007**		**11/16/2007**	
EERC Run Number	**2679A12.D**		**2679A13.D**		**2679A14.D**		**2679A16.D**	
AQUATOX Sample ID								
Sample Name	**AR08**		**AR08**		**AR08**		**AR08**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**0.77**		**0.77**		**0.77**		**0.77**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.126		0.148		0.133		0.160	
2-methylnaphthalene	0.139		0.129		0.140		0.119	
1-methylnaphthalene	0.067		0.060	J	0.067		0.071	
C2 naphthalenes	1.168		1.032		1.072		1.140	
C3 naphthalenes	0.902		0.857		0.946		1.028	
C4 naphthalenes	0.928		1.058		1.145		1.218	
acenaphthylene	0.380		0.315		0.300		0.322	
acenaphthene	0.131		0.143		0.152		0.160	
fluorene	0.173		0.161		0.166		0.176	
C1 fluorenes	0.502		0.532		0.511		0.555	
C2 fluorenes	2.223		2.485		2.480		2.637	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	1.529		1.422		1.546		1.542	
anthracene	0.892		0.819		0.837		0.902	
C1 phenanthrenes/anthracenes	1.336		1.326		1.335		1.425	
C2 phenanthrenes/anthracenes	5.165		5.485		5.226		5.272	
C3 phenanthrenes/anthracenes	5.939		6.030		5.912		5.999	
C4 phenanthrenes/anthracenes	1.973		2.275		1.738		1.749	
fluoranthene	2.745		2.821		3.067		3.186	
pyrene	2.445		2.538		2.756		2.741	
C1 fluoranthenes/pyrenes	2.256		2.306		2.370		2.353	
benz[a]anthracene	1.072		1.148		1.182		1.256	
chrysene	2.203		2.299		2.455		2.943	
C1 chrysenes	3.755		3.861		3.956		4.466	
C2 chrysenes	4.221		4.348		4.428		4.828	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	2.707		2.899		3.020		3.509	
benzo[e]pyrene	1.048		1.124		1.158		1.408	
benzo[a]pyrene	1.681		1.758		1.848		2.006	
perylene	0.775		0.838		0.849		0.966	
indeno[1,2,3-cd]pyrene	2.634		2.822		2.886		2.709	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	1.683		1.742		1.822		1.972	

Total NOAA PAHs	53		55		56		59	
Total EPA PAHs (16)	20		21		22		24	

Surrogate recoveries								
biphenyl-d10	105.3%		102.8%		105.7%		104.6%	
benzo[b]fluoranthene-d12	93.9%		97.1%		110.9%		104.4%	

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EERC ID	**75C-1**		**75C-2**		**75C-3**		**75C-4**	
RETEC Sample Collection Date	**11/8/2007**		**11/8/2007**		**11/8/2007**		**11/8/2007**	
EERC Sample Receipt Date	**11/9/2007**		**11/9/2007**		**11/9/2007**		**11/9/2007**	
EERC Run Date	**11/20/2007**		**11/20/2007**		**11/20/2007**		**11/20/2007**	
EERC Run Number	**2680A01.D**		**2680A02.D**		**2680A03.D**		**2680A04.D**	
AQUATOX Sample ID								
Sample Name	**AR10**		**AR10**		**AR10**		**AR10**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**0.68**		**0.68**		**0.68**		**0.68**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.181		0.179		0.166		0.177	
2-methylnaphthalene	0.145		0.162		0.152		0.157	
1-methylnaphthalene	0.075		0.090		0.088		0.079	
C2 naphthalenes	1.578		1.431		1.457		1.749	
C3 naphthalenes	0.654		0.766		0.715		0.869	
C4 naphthalenes	1.196		1.110		1.304		1.375	
acenaphthylene	0.369		0.436		0.413		0.383	
acenaphthene	0.157		0.164		0.156		0.175	
fluorene	0.234		0.241		0.235		0.249	
C1 fluorenes	0.395		0.474		0.468		0.469	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	1.750		1.838		1.807		1.842	
anthracene	1.157		1.315		1.102		1.235	
C1 phenanthrenes/anthracenes	1.663		1.814		1.705		1.807	
C2 phenanthrenes/anthracenes	3.816		4.689		4.073		4.286	
C3 phenanthrenes/anthracenes	6.518		6.679		6.090		6.400	
C4 phenanthrenes/anthracenes	2.099		2.279		2.874		2.143	
fluoranthene	3.480		3.498		3.672		3.554	
pyrene	3.119		3.196		3.274		3.204	
C1 fluoranthenes/pyrenes	2.565		2.924		2.685		2.644	
benz[a]anthracene	1.436		1.581		1.412		1.418	
chrysene	2.879		3.158		2.827		2.858	
C1 chrysenes	4.878		4.847		4.603		4.629	
C2 chrysenes	4.619		5.061		5.023		4.842	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	3.865		3.910		3.839		3.828	
benzo[e]pyrene	1.659		1.608		1.593		1.578	
benzo[a]pyrene	2.444		2.490		2.479		2.389	
perylene	1.174		1.232		1.211		1.165	
indeno[1,2,3-cd]pyrene	2.613		2.957		3.066		2.900	
dibenz[ah]anthracene	0.667		0.560		0.477		0.503	
benzo[ghi]perylene	2.049		2.098		2.127		1.979	

Total NOAA PAHs	59		63		61		61	
Total EPA PAHs (16)	26		28		27		27	

Surrogate recoveries								
biphenyl-d10	109.6%		106.2%		107.2%		108.0%	
benzo[b]fluoranthene-d12	110.4%		109.0%		106.1%		106.6%	

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EERC ID	76C-1		76C-2		76C-3		76C-4	
RETEC Sample Collection Date	11/6/2007		11/6/2007		11/6/2007		11/6/2007	
EERC Sample Receipt Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Run Date	11/27/2007		11/27/2007		11/27/2007		11/27/2007	
EERC Run Number	2682A09.D		2682A10.D		2682A11.D		2682A12.D	
AQUATOX Sample ID								
Sample Name	AR13		AR13		AR13		AR13	
Treatment	Soxhlet		Soxhlet		Soxhlet		Soxhlet	
Sample Weight, g	0.97		0.97		0.97		0.97	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ug/g		ug/g		ug/g		ug/g	
naphthalene	0.117		0.133		0.152		21.470	
2-methylnaphthalene	0.090		0.125		0.119		9.447	
1-methylnaphthalene	0.049	J	0.070		0.074		5.499	
C2 naphthalenes	1.039		0.967		1.234		16.354	
C3 naphthalenes	0.744		0.735		0.743		8.609	
C4 naphthalenes	1.241		1.083		1.160		4.719	
acenaphthylene	0.327		0.367		0.368		1.568	
acenaphthene	0.219		0.232		0.236		59.577	
fluorene	0.323		0.350		0.414		33.423	
C1 fluorenes	0.551		0.658		0.625		24.609	E
C2 fluorenes	2.732		2.255		2.481		27.886	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	3.152		3.794		3.724		190.205	E
anthracene	1.709		2.011		2.119		122.462	E
C1 phenanthrenes/anthracenes	1.894		2.226		2.318		94.841	E
C2 phenanthrenes/anthracenes	4.108		4.070		4.552		84.355	E
C3 phenanthrenes/anthracenes	9.092		7.374		7.611		54.940	
C4 phenanthrenes/anthracenes	2.219		1.648		1.877		9.671	
fluoranthene	6.851		8.369		7.296		182.485	E
pyrene	5.500		6.820		5.812		152.841	E
C1 fluoranthenes/pyrenes	3.931		4.927		4.844		137.253	E
benz[a]anthracene	2.319		2.905		2.704		86.741	E
chrysene	4.682		5.732		5.026		102.336	E
C1 chrysenes	7.729		9.977		8.845		167.198	E
C2 chrysenes	7.731		9.467		7.501		108.607	
C3 chrysenes	ND		ND		ND		38.713	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	8.279		10.195		8.508		142.927	E
benzo[e]pyrene	2.872		3.410		2.694		36.635	E
benzo[a]pyrene	5.171		6.532		5.535		114.949	E
perylene	1.117		1.400		1.168		23.105	
indeno[1,2,3-cd]pyrene	3.485		4.791		4.301		99.977	E
dibenz[ah]anthracene	0.556		0.764		0.680		20.714	E
benzo[ghi]perylene	3.223		4.065		3.382		56.456	E

Total NOAA PAHs	93		107		98		2241	
Total EPA PAHs (16)	46		57		50		1388	

Surrogate recoveries								
biphenyl-d10	105.5%		109.1%		103.4%		106.2%	
benzo[b]fluoranthene-d12	119.3%		117.5%		118.7%		94.7%	

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EERC ID	**77C-1**		**77C-2**		**77C-3**		**77C-4**	
RETEC Sample Collection Date	**11/6/2007**		**11/6/2007**		**11/6/2007**		**11/6/2007**	
EERC Sample Receipt Date	**11/7/2007**		**11/7/2007**		**11/7/2007**		**11/7/2007**	
EERC Run Date	**11/20/2007**		**11/20/2007**		**11/20/2007**		**11/20/2007**	
EERC Run Number	**2680A05.D**		**2680A06.D**		**2680A07.D**		**2680A08.D**	
AQUATOX Sample ID								
Sample Name	**AR14**		**AR14**		**AR14**		**AR14**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**0.72**		**0.72**		**0.72**		**0.72**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.170		0.208		0.174		0.156	
2-methylnaphthalene	0.146		0.159		0.150		0.146	
1-methylnaphthalene	0.070		0.089		0.067		0.068	
C2 naphthalenes	1.539		1.521		1.449		1.549	
C3 naphthalenes	0.651		0.606		0.679		0.719	
C4 naphthalenes	1.002		0.925		0.989		1.114	
acenaphthylene	0.430		0.421		0.478		0.826	
acenaphthene	0.152		0.172		0.158		0.165	
fluorene	0.224		0.238		0.242		0.227	
C1 fluorenes	0.438		0.514		0.451		0.499	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	1.607		2.168		1.828		1.745	
anthracene	1.277		1.418		1.667		2.294	
C1 phenanthrenes/anthracenes	1.649		1.950		2.081		2.000	
C2 phenanthrenes/anthracenes	4.039		4.450		4.508		5.416	
C3 phenanthrenes/anthracenes	5.889		6.472		6.989		7.835	
C4 phenanthrenes/anthracenes	1.612		1.869		2.189		2.065	
fluoranthene	3.655		4.301		3.897		4.138	
pyrene	3.354		3.785		3.622		7.483	
C1 fluoranthenes/pyrenes	3.179		3.334		4.556		4.948	
benz[a]anthracene	1.704		1.930		2.517		2.813	
chrysene	3.392		3.726		4.924		4.844	
C1 chrysenes	5.217		5.904		8.201		8.495	
C2 chrysenes	5.555		5.869		7.365		6.968	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	4.290		4.603		6.030		6.130	
benzo[e]pyrene	1.775		1.872		2.376		2.304	
benzo[a]pyrene	2.809		3.047		4.194		3.957	
perylene	1.252		1.308		1.602		1.561	
indeno[1,2,3-cd]pyrene	3.360		3.756		4.296		4.022	
dibenz[ah]anthracene	0.563		0.744		0.952		0.783	
benzo[ghi]perylene	2.241		2.524		2.800		2.578	

Total NOAA PAHs	63		70		81		88	
Total EPA PAHs (16)	29		33		38		42	

Surrogate recoveries								
biphenyl-d10	108.1%		105.8%		104.6%		103.9%	
benzo[b]fluoranthene-d12	106.0%		102.0%		101.0%		105.1%	

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EERC ID	**78C-1**		**78C-2**		**78C-3**		**78C-4**	
RETEC Sample Collection Date	**11/6/2007**		**11/6/2007**		**11/6/2007**		**11/6/2007**	
EERC Sample Receipt Date	**11/7/2007**		**11/7/2007**		**11/7/2007**		**11/7/2007**	
EERC Run Date	**11/20/2007**		**11/20/2007**		**11/20/2007**		**11/20/2007**	
EERC Run Number	**2680A09.D**		**2680A10.D**		**2680A11.D**		**2680A12.D**	
AQUATOX Sample ID								
Sample Name	**AR16**		**AR16**		**AR16**		**AR16**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**0.76**		**0.76**		**0.76**		**0.76**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.162		0.332		0.265		0.400	
2-methylnaphthalene	0.151		0.314		0.198		0.281	
1-methylnaphthalene	0.082		0.253		0.115		0.228	
C2 naphthalenes	1.350		1.946		1.478		2.112	
C3 naphthalenes	0.671		1.283		0.891		1.352	
C4 naphthalenes	0.908		1.899		1.301		1.873	
acenaphthylene	0.295		0.333		0.376		0.388	
acenaphthene	0.159		0.269		0.226		0.353	
fluorene	0.249		0.473		0.363		0.516	
C1 fluorenes	0.424		0.552		0.513		0.649	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	1.726		2.572		2.124		3.374	
anthracene	1.078		1.736		1.638		2.072	
C1 phenanthrenes/anthracenes	1.523		2.752		2.124		3.143	
C2 phenanthrenes/anthracenes	3.740		6.606		5.510		6.857	
C3 phenanthrenes/anthracenes	5.913		9.267		7.650		9.940	
C4 phenanthrenes/anthracenes	1.989		2.543		2.633		3.417	
fluoranthene	3.074		5.306		4.862		6.873	
pyrene	2.752		4.772		4.412		5.900	
C1 fluoranthenes/pyrenes	2.416		4.285		3.693		4.318	
benz[a]anthracene	1.275		2.387		2.192		2.299	
chrysene	2.513		3.931		3.800		4.213	
C1 chrysenes	4.546		6.914		6.459		6.967	
C2 chrysenes	4.437		7.271		6.241		7.552	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	3.371		4.954		4.928		5.143	
benzo[e]pyrene	1.360		2.043		1.965		2.072	
benzo[a]pyrene	2.075		3.333		3.285		3.403	
perylene	0.949		1.253		1.328		1.354	
indeno[1,2,3-cd]pyrene	2.030		3.410		3.731		3.695	
dibenz[ah]anthracene	0.452		0.613		0.690		0.601	
benzo[ghi]perylene	1.616		2.463		2.576		2.495	

Total NOAA PAHs	53		86		78		94	
Total EPA PAHs (16)	23		37		35		42	

Surrogate recoveries								
biphenyl-d10	105.4%		108.1%		104.7%		104.4%	
benzo[b]fluoranthene-d12	104.7%		107.2%		103.6%		99.4%	

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EERC ID	**68C-1**		**68C-2**		**68C-3**		**68C-4**	
RETEC Sample Collection Date	**11/6/2007**		**11/6/2007**		**11/6/2007**		**11/6/2007**	
EERC Sample Receipt Date	**11/7/2007**		**11/7/2007**		**11/7/2007**		**11/7/2007**	
EERC Run Date	**11/16/2007**		**11/16/2007**		**11/16/2007**		**11/16/2007**	
EERC Run Number	**2679A01.D**		**2679A02.D**		**2679A03.D**		**2679A04.D**	
AQUATOX Sample ID								
Sample Name	**AR17**		**AR17**		**AR17**		**AR17**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**0.75**		**0.75**		**0.75**		**0.75**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.173		0.190		0.228		0.192	
2-methylnaphthalene	0.161		0.169		0.183		0.173	
1-methylnaphthalene	0.097		0.101		0.104		0.100	
C2 naphthalenes	1.538		1.392		1.731		1.563	
C3 naphthalenes	1.635		0.866		1.217		1.251	
C4 naphthalenes	2.390		0.958		1.236		1.367	
acenaphthylene	0.250		0.292		0.281		0.302	
acenaphthene	0.255		0.219		0.436		0.245	
fluorene	0.312		0.267		0.515		0.286	
C1 fluorenes	0.639		0.454		0.681		0.610	
C2 fluorenes	3.056		2.009		2.286		2.632	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	2.491		2.342		5.020		2.875	
anthracene	1.357		1.309		2.787		1.549	
C1 phenanthrenes/anthracenes	2.016		1.631		2.766		2.128	
C2 phenanthrenes/anthracenes	7.493		5.502		7.568		6.748	
C3 phenanthrenes/anthracenes	9.371		5.850		7.370		7.107	
C4 phenanthrenes/anthracenes	3.431		2.003		2.282		2.419	
fluoranthene	4.753		4.027		9.216		5.401	
pyrene	4.607		3.601		7.944		4.662	
C1 fluoranthenes/pyrenes	3.812		2.912		6.612		3.550	
benz[a]anthracene	2.553		1.986		4.629		2.344	
chrysene	4.591		3.104		6.554		3.644	
C1 chrysenes	9.772		5.831		10.585		6.204	
C2 chrysenes	10.745		5.582		9.670		6.486	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	6.753		4.186		6.926		4.454	
benzo[e]pyrene	2.871		1.651		2.635		1.737	
benzo[a]pyrene	4.280		2.822		4.865		3.104	
perylene	1.605		1.121		1.723		1.217	
indeno[1,2,3-cd]pyrene	4.212		2.577		4.455		3.214	
dibenz[ah]anthracene	ND		ND		ND		ND	
benzo[ghi]perylene	3.201		1.909		2.991		2.117	

Total NOAA PAHs	100		67		115		80	
Total EPA PAHs (16)	40		29		57		34	

Surrogate recoveries								
biphenyl-d10	106.6%		104.3%		105.1%		106.3%	
benzo[b]fluoranthene-d12	116.4%		110.5%		109.9%		109.7%	

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EERC ID	**79C-1**		**79C-2**		**79C-3**		**79C-4**	
RETEC Sample Collection Date	**11/7/2007**		**11/7/2007**		**11/7/2007**		**11/7/2007**	
EERC Sample Receipt Date	**11/8/2007**		**11/8/2007**		**11/8/2007**		**11/8/2007**	
EERC Run Date	**11/27/2007**		**11/27/2007**		**11/27/2007**		**11/27/2007**	
EERC Run Number	**2682A13.D**		**2682A14.D**		**2682A15.D**		**2682A16.D**	
AQUATOX Sample ID								
Sample Name	**AR21**		**AR21**		**AR21**		**AR21**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**0.68**		**0.68**		**0.68**		**0.68**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.192		0.165		0.195		0.191	
2-methylnaphthalene	0.177		0.164		0.182		0.190	
1-methylnaphthalene	0.112		0.094		0.109		0.094	
C2 naphthalenes	1.462		1.422		1.413		1.777	
C3 naphthalenes	1.563		1.685		1.882		1.748	
C4 naphthalenes	1.836		1.904		2.067		1.951	
acenaphthylene	0.328		0.303		0.415		0.356	
acenaphthene	0.194		0.201		0.216		0.204	
fluorene	0.314		0.309		0.319		0.316	
C1 fluorenes	0.725		0.737		0.786		0.767	
C2 fluorenes	2.765		3.351		3.667		3.704	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	2.249		2.349		2.536		2.574	
anthracene	1.394		1.379		1.573		1.484	
C1 phenanthrenes/anthracenes	2.240		2.431		2.702		2.634	
C2 phenanthrenes/anthracenes	5.663		6.411		6.560		6.399	
C3 phenanthrenes/anthracenes	8.513		9.114		9.345		9.378	
C4 phenanthrenes/anthracenes	2.382		2.551		3.195		2.620	
fluoranthene	4.697		5.041		5.246		5.510	
pyrene	3.995		4.330		4.604		4.720	
C1 fluoranthenes/pyrenes	4.074		4.300		4.667		4.683	
benz[a]anthracene	1.912		1.849		2.013		1.940	
chrysene	3.902		3.782		4.019		4.065	
C1 chrysenes	7.489		7.126		7.536		7.394	
C2 chrysenes	7.315		7.258		7.316		7.737	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	6.534		6.467		7.115		7.373	
benzo[e]pyrene	2.008		1.987		2.153		2.261	
benzo[a]pyrene	3.920		3.923		4.433		4.579	
perylene	1.003		0.991		1.110		1.050	
indeno[1,2,3-cd]pyrene	3.492		3.560		3.948		3.929	
dibenz[ah]anthracene	0.613		0.598		0.753		0.532	
benzo[ghi]perylene	2.656		2.693		2.780		2.913	

Total NOAA PAHs	86		88		95		95	
Total EPA PAHs (16)	36		37		40		41	

Surrogate recoveries								
biphenyl-d10	105.9%		106.0%		109.3%		103.3%	
benzo[b]fluoranthene-d12	110.8%		109.3%		112.7%		111.7%	

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EERC ID	**80C-1**		**80C-2**		**80C-3**		**80C-4**	
RETEC Sample Collection Date	**11/7/2007**		**11/7/2007**		**11/7/2007**		**11/7/2007**	
EERC Sample Receipt Date	**11/8/2007**		**11/8/2007**		**11/8/2007**		**11/8/2007**	
EERC Run Date	**11/20/2007**		**11/20/2007**		**11/20/2007**		**11/20/2007**	
EERC Run Number	**2680A13.D**		**2680A14.D**		**2680A15.D**		**2680A16.D**	
AQUATOX Sample ID								
Sample Name	**AR25**		**AR25**		**AR25**		**AR25**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**0.72**		**0.72**		**0.72**		**0.72**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.154		0.142		0.150		0.165	
2-methylnaphthalene	0.134		0.136		0.136		0.139	
1-methylnaphthalene	0.090		0.081		0.084		0.093	
C2 naphthalenes	1.284		1.275		1.226		1.570	
C3 naphthalenes	0.674		0.673		0.691		0.816	
C4 naphthalenes	0.859		0.938		0.928		1.194	
acenaphthylene	0.287		0.253		0.299		0.332	
acenaphthene	0.130		0.121		0.126		0.144	
fluorene	0.175		0.167		0.177		0.185	
C1 fluorenes	0.362		0.331		0.356		0.442	
C2 fluorenes	ND		ND		ND		ND	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	1.445		1.414		1.504		1.595	
anthracene	0.811		0.848		0.886		0.948	
C1 phenanthrenes/anthracenes	1.442		1.344		1.409		1.651	
C2 phenanthrenes/anthracenes	3.517		3.439		3.697		4.018	
C3 phenanthrenes/anthracenes	5.481		5.143		5.630		5.876	
C4 phenanthrenes/anthracenes	1.435		2.216		2.467		1.821	
fluoranthene	2.919		2.853		3.066		3.132	
pyrene	2.602		2.598		2.773		2.783	
C1 fluoranthenes/pyrenes	2.030		2.115		2.156		2.205	
benz[a]anthracene	1.156		1.168		1.213		1.245	
chrysene	2.210		2.251		2.275		2.293	
C1 chrysenes	3.669		3.777		3.950		4.009	
C2 chrysenes	4.033		4.121		4.623		4.474	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	3.125		2.992		3.273		3.208	
benzo[e]pyrene	1.265		1.226		1.339		1.309	
benzo[a]pyrene	2.030		1.946		2.159		2.081	
perylene	1.136		1.085		1.213		1.170	
indeno[1,2,3-cd]pyrene	2.327		2.381		2.418		2.484	
dibenz[ah]anthracene	0.394		0.442		0.392		0.403	
benzo[ghi]perylene	1.571		1.657		1.731		1.771	

Total NOAA PAHs	49		49		52		54	
Total EPA PAHs (16)	21		21		22		23	

Surrogate recoveries								
biphenyl-d10	107.9%		102.6%		109.2%		108.0%	
benzo[b]fluoranthene-d12	108.7%		100.8%		111.1%		104.2%	

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EERC ID	**69C-1**		**69C-2**		**69C-3**		**69C-4**	
RETEC Sample Collection Date	**11/6/2007**		**11/6/2007**		**11/6/2007**		**11/6/2007**	
EERC Sample Receipt Date	**11/7/2007**		**11/7/2007**		**11/7/2007**		**11/7/2007**	
EERC Run Date	**11/16/2007**		**11/16/2007**		**11/16/2007**		**11/16/2007**	
EERC Run Number	**2679A05.D**		**2679A06.D**		**2679A07.D**		**2679A08.D**	
AQUATOX Sample ID								
Sample Name	**AR26**		**AR26**		**AR26**		**AR26**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**0.93**		**0.93**		**0.93**		**0.93**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.441		0.696		0.441		0.764	
2-methylnaphthalene	0.524		0.765		0.337		0.565	
1-methylnaphthalene	0.484		0.626		0.232		0.478	
C2 naphthalenes	2.749		3.218		1.657		2.609	
C3 naphthalenes	2.246		2.584		1.555		2.125	
C4 naphthalenes	2.043		2.454		1.510		1.858	
acenaphthylene	0.682		0.282		0.349		0.597	
acenaphthene	0.603		0.538		0.516		0.784	
fluorene	1.103		0.833		0.918		1.100	
C1 fluorenes	2.437		1.077		0.748		1.927	
C2 fluorenes	6.796		3.229		2.545		4.279	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	11.742		4.242		4.723		10.595	
anthracene	2.555		2.039		2.292		4.840	
C1 phenanthrenes/anthracenes	4.926		3.783		2.820		5.831	
C2 phenanthrenes/anthracenes	11.949	E	10.911		7.806		14.518	E
C3 phenanthrenes/anthracenes	10.673		9.260		6.991		16.218	
C4 phenanthrenes/anthracenes	2.663		3.076		1.767		3.160	
fluoranthene	17.746		6.540		7.008		16.059	
pyrene	13.771		5.267		5.517		12.731	
C1 fluoranthenes/pyrenes	8.832		4.547		5.304		9.687	
benz[a]anthracene	6.493		1.721		1.989		5.594	
chrysene	12.002		3.705		5.038		12.091	
C1 chrysenes	13.451		6.088		5.430		16.062	
C2 chrysenes	10.148		6.387		5.050		11.658	
C3 chrysenes	5.458		3.572		2.635		4.553	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	10.871		3.426		3.469		11.176	
benzo[e]pyrene	3.759		1.341		1.249		3.888	
benzo[a]pyrene	6.426		1.944		1.736		6.135	
perylene	1.891		0.583		0.579		1.421	
indeno[1,2,3-cd]pyrene	5.024		2.003		1.881		6.675	
dibenz[ah]anthracene	1.249		0.485		0.431		1.706	
benzo[ghi]perylene	3.083		1.413		1.246		4.408	

Total NOAA PAHs	185		99		86		196	
Total EPA PAHs (16)	94		35		38		95	

Surrogate recoveries								
biphenyl-d10	106.5%		104.2%		105.0%		107.4%	
benzo[b]fluoranthene-d12	102.3%		100.0%		105.0%		105.6%	

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EERC ID	81C-1		81C-2		81C-3		81C-4	
RETEC Sample Collection Date	11/7/2007		11/7/2007		11/7/2007		11/7/2007	
EERC Sample Receipt Date	11/8/2007		11/8/2007		11/8/2007		11/8/2007	
EERC Run Date	11/27/2007		11/27/2007		11/27/2007		11/27/2007	
EERC Run Number	2682A17.D		2682A18.D		2682A19.D		2682A20.D	
AQUATOX Sample ID								
Sample Name	AR27		AR27		AR27		AR27	
Treatment	Soxhlet		Soxhlet		Soxhlet		Soxhlet	
Sample Weight, g	0.89		0.89		0.89		0.89	
Matrix	Sediment		Sediment		Sediment		Sediment	
Units	ug/g		ug/g		ug/g		ug/g	
naphthalene	0.118		0.105	J	0.111		0.118	
2-methylnaphthalene	0.101		0.090		0.104		0.138	
1-methylnaphthalene	0.058	J	0.058	J	0.060	J	0.076	
C2 naphthalenes	0.893		0.790		0.772		1.073	
C3 naphthalenes	0.616		0.729		0.656		0.864	
C4 naphthalenes	0.728		0.839		0.716		1.020	
acenaphthylene	0.382		0.210		0.207		0.433	
acenaphthene	0.096		0.108		0.120		0.115	
fluorene	0.146		0.177		0.174		0.176	
C1 fluorenes	0.375		0.422		0.416		0.506	
C2 fluorenes	1.925		1.949		1.731		2.185	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	1.303		1.800		1.623		1.485	
anthracene	0.852		0.904		0.869		0.981	
C1 phenanthrenes/anthracenes	1.352		1.513		1.356		1.533	
C2 phenanthrenes/anthracenes	3.038		3.180		3.004		4.054	
C3 phenanthrenes/anthracenes	4.875		4.928		4.524		6.352	
C4 phenanthrenes/anthracenes	1.950		1.326		1.259		1.825	
fluoranthene	2.964		4.153		3.350		3.359	
pyrene	2.561		3.411		2.866		2.953	
C1 fluoranthenes/pyrenes	2.575		2.826		2.489		3.669	
benz[a]anthracene	1.204		1.450		1.251		1.461	
chrysene	2.403		2.800		2.482		2.761	
C1 chrysenes	4.365		4.491		3.879		6.157	
C2 chrysenes	4.724		4.127		3.692		6.549	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	4.443		4.813		4.318		4.726	
benzo[e]pyrene	1.409		1.464		1.327		1.478	
benzo[a]pyrene	2.919		3.043		2.614		3.234	
perylene	0.630		0.676		0.605		0.666	
indeno[1,2,3-cd]pyrene	2.290		2.511		2.065		2.420	
dibenz[ah]anthracene	0.482		0.419		0.454		0.370	
benzo[ghi]perylene	2.208		1.959		1.644		1.888	

Total NOAA PAHs	54		57		51		65	
Total EPA PAHs (16)	24		28		24		26	

Surrogate recoveries								
biphenyl-d10	100.4%		103.9%		102.5%		91.2%	
benzo[b]fluoranthene-d12	107.9%		108.4%		108.9%		97.5%	

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EERC ID	**82C-1**		**82C-2**		**82C-3**		**82C-4**	
RETEC Sample Collection Date	**11/8/2007**		**11/8/2007**		**11/8/2007**		**11/8/2007**	
EERC Sample Receipt Date	**11/9/2007**		**11/9/2007**		**11/9/2007**		**11/9/2007**	
EERC Run Date	**11/27/2007**		**11/27/2007**		**11/27/2007**		**11/27/2007**	
EERC Run Number	**2682A21.D**		**2682A22.D**		**2682A23.D**		**2682A24.D**	
AQUATOX Sample ID								
Sample Name	**AR36**		**AR36**		**AR36**		**AR36**	
Treatment	**Soxhlet**		**Soxhlet**		**Soxhlet**		**Soxhlet**	
Sample Weight, g	**0.72**		**0.72**		**0.72**		**0.72**	
Matrix	**Sediment**		**Sediment**		**Sediment**		**Sediment**	
Units	**ug/g**		**ug/g**		**ug/g**		**ug/g**	
naphthalene	0.189		0.253		0.214		0.178	
2-methylnaphthalene	0.178		0.199		0.205		0.173	
1-methylnaphthalene	0.089		0.098		0.087		0.097	
C2 naphthalenes	1.500		1.501		1.751		1.537	
C3 naphthalenes	1.014		0.980		1.284		1.065	
C4 naphthalenes	1.213		1.260		1.280		1.177	
acenaphthylene	0.302		0.312		0.325		0.310	
acenaphthene	0.158		0.175		0.162		0.157	
fluorene	0.220		0.246		0.244		0.239	
C1 fluorenes	0.589		0.623		0.651		0.589	
C2 fluorenes	2.722		3.197		2.790		2.728	
C3 fluorenes	ND		ND		ND		ND	
phenanthrene	1.806		1.894		2.076		1.904	
anthracene	1.142		1.223		1.235		1.205	
C1 phenanthrenes/anthracenes	1.861		1.919		2.050		1.883	
C2 phenanthrenes/anthracenes	4.342		4.447		5.035		4.439	
C3 phenanthrenes/anthracenes	8.086		7.615		7.673		7.282	
C4 phenanthrenes/anthracenes	2.490		2.852		3.219		2.365	
fluoranthene	3.606		3.833		4.004		3.510	
pyrene	3.263		3.379		3.552		3.195	
C1 fluoranthenes/pyrenes	3.555		3.774		3.893		3.503	
benz[a]anthracene	1.516		1.570		1.676		1.440	
chrysene	2.912		3.037		3.216		2.851	
C1 chrysenes	5.348		5.631		5.919		5.258	
C2 chrysenes	6.556		6.301		6.838		6.222	
C3 chrysenes	ND		ND		ND		ND	
C4 chrysenes	ND		ND		ND		ND	
benzo[b+k]fluoranthene	5.367		5.527		5.994		5.125	
benzo[e]pyrene	1.596		1.643		1.769		1.517	
benzo[a]pyrene	3.386		3.339		3.774		3.152	
perylene	1.012		1.018		1.054		0.972	
indeno[1,2,3-cd]pyrene	3.114		3.067		3.316		2.953	
dibenz[ah]anthracene	0.438		0.548		0.598		0.527	
benzo[ghi]perylene	2.310		2.145		2.377		2.103	

Total NOAA PAHs	72		74		78		70	
Total EPA PAHs (16)	30		31		33		29	

Surrogate recoveries								
biphenyl-d10	99.8%		105.0%		99.3%		98.9%	
benzo[b]fluoranthene-d12	103.9%		108.8%		105.3%		103.5%	

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Pore Water and Sediment Detection Limits

CAS Numbers	PAHs	Method	Pore Water Target Detection Limits[1] (ug/L)	Pore Water Actual Detection Limits[3] (ug/L)	Sediment Target Detection Limits[1,2] (mg/Kg dry wt)	Sediment Actual Detection Limits[3] (mg/Kg dry wt)
91-20-3	Naphthalene	GC/MS	5.7	0.500	0.11	0.001
91-57-6	2-Methylnaphthalene	GC/MS	2.4	0.200	0.13	0.001
90-12-0	1-Methylnaphthalene	GC/MS	2.4	0.200	0.13	0.001
	C2 naphthalenes	GC/MS	0.89	0.600	0.15	0.005
	C3 naphthalenes	GC/MS	0.33	0.300	0.17	0.010
	C4 naphthalenes	GC/MS	0.12	0.050	0.19	0.010
208-96-8	Acenaphthylene	GC/MS	9.0	0.100	0.13	0.001
83-32-9	Acenaphthene	GC/MS	1.6	0.050	0.14	0.001
86-73-7	Fluorene	GC/MS	1.2	0.050	0.16	0.001
	C1 fluorenes	GC/MS	0.41	0.100	0.18	0.005
	C2 fluorenes	GC/MS	0.16	0.100	0.20	0.01
	C3 fluorenes	GC/MS	0.06	0.050	0.23	0.030
85-01-8	Phenanthrene	GC/MS	0.56	0.200	0.18	0.001
120-12-7	Anthracene	GC/MS	0.61	0.050	0.17	0.001
	C1 phenanthrenes/anthracenes	GC/MS	0.22	0.200	0.20	0.005
	C2 phenanthrenes/anthracenes	GC/MS	0.09	0.050	0.22	0.010
	C3 phenanthrenes/anthracenes	GC/MS	0.04	0.020	0.24	0.020
	C4 phenanthrenes/anthracenes	GC/MS	0.02	0.020	0.27	0.030
206-44-0	Fluoranthene	GC/MS	0.21	0.040	0.21	0.001
129-00-0	Pyrene	GC/MS	0.30	0.040	0.21	0.001
	C1 pyrene/fluoranthenes	GC/MS	0.14	0.050	0.23	0.005
56-55-3	Benz(a)anthracene	GC/MS	0.066	0.010	0.25	0.002
218-01-9	Chrysene	GC/MS	0.060	0.010	0.25	0.002
	C1 benz(a)anthracene/chrysenes	GC/MS	0.025	0.020	0.27	0.010
	C2 benz(a)anthracene/chrysenes	GC/MS	0.014	0.008	0.30	0.030
	C3 benz(a)anthracene/chrysenes	GC/MS	0.005	0.008	0.33	0.050
	C4 benz(a)anthracene/chrysenes	GC/MS	0.002	0.008	0.36	0.080
205-99-2 207-08-9	Benzo(b & k)fluoranthene[4]	GC/MS	0.019	0.010	0.29	0.002
50-32-8	Benzo(a)pyrene	GC/MS	0.028	0.005	0.28	0.002
192-97-2	Benzo(e)pyrene	GC/MS	0.026	0.005	0.28	0.002
198-55-0	Perylene	GC/MS	0.026	0.005	0.28	0.002
193-39-5	Indeno(1,2,3-cd)pyrene	GC/MS	0.008	0.002	0.33	0.002
53-70-3	Dibenz(a,h)anthracene	GC/MS	0.008	0.002	0.33	0.002
191-24-2	Benzo(g,h,i)perylene	GC/MS	0.013	0.002	0.32	0.002

Notes:

(1) Target detection limits for both sediment and pore water are 1/34th of the concentration of each individual PAH that corresponds to one toxic unit as described in U. S. EPA (2003) *Equilibrium Partitioning Sediment Guidelines (ESGs) for the Protection of Benthic Organisms*

(2) Sediment target detection limits were estimated assuming 1.0 % total organic carbon. Actual laboratory detection limit for sediments and pore water are based on previous research studies.

(3) Benzo[b]fluoranthene and benzo[k]fluoranthene are reported as their sum because of insufficient chromatographic resolution.

Appendix J: ERDC Laboratory Report: Aquatic Toxicity Tests

CEERD-EP-R

7 January 2007

MEMORANDUM FOR: Steve Geiger/RETEC-ENSR

SUBJECT: Chronic Sediment Toxicity Data for Anacostia River Sediment Exposed *Hyalella azteca*

1. The following is an abbreviated summary of the results from the 28-d *Hyalella azteca* study conducted with the ESTCP Anacostia River sediments. All data has passed QA/QC review and are considered final.

Please contact me if you have any questions regarding the data or methods utilized in conducting the studies.

J. Daniel Farrar

Research Biologist

Study Summary

Methods

The *H. azteca* 28-d study was conducted following the methods described in "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates" (EPA/600/R-99/064, 2000). Due to the number of sediments analyzed, the study was conducted in two phases. Sediments AR02, AR03, AR05, AR07, AR08 AR10 and AR13 were evaluated in phase I of the study. Sediments AR14, AR16, AR17, AR26, AR27 and AR36 were evaluated in phase II of the study. A performance control and reference sediments AR21 and AR25 were also evaluated in each phase of the study.

Deviations from test method.

Initial water quality was measured on 1 replicate per sediment sample at test initiation rather than on all replicates as suggested in the test method. Experience has demonstrated that when test setup occurs on day -1, water quality measurements on individual replicates of a given sediment treatment on day 0 are essentially identical therefore measurement on all replicates is not necessary. The method suggests measuring dissolved oxygen daily for both tests since the method doesn't require aeration unless dissolved oxygen measurements drops below 2.5 mg/L. Since the tests were aerated from test initiation to avoid issues related to depleted oxygen, dissolved oxygen was only measured during the course of the test if aeration was interrupted.

Statistical analyses. Statistical analyses were conducted for both studies following the methods described in "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates" and in "Evaluation of Dredged Material Proposed for Discharge in the Waters of the U.S.–Testing Manual" (EPA/823/B-98/004, 1998).

H. azteca survival. For each reference, all treatments that were not greater than 20% different from the reference were removed from the data set prior to analysis. In addition, the performance control and additional reference data were also removed. Only AR02 and AR27 had survival that was 20% less than one or both references. Percent survival for AR02 was compared to the AR21 and AR25 references using the Mann-Whitney Rank Sum Test with α=0.05. Percent survival for AR27 was compared to the AR25 reference using the T-test procedure with α=0.05.

H. azteca mass. The performance control and additional reference were removed from each data set prior to analysis. Analyses were conducted using one-way ANOVA followed by Dunnett's mean comparison to the reference or Kruskall-Wallis ANOVA on Ranks (non-parametric) followed by Dunn's mean comparison procedure.

Results

Performance metrics were met with mean control survival exceeding 80% (Tables 7 and 9). Water quality parameters fell within required ranges (Tables 1-4). Survival was significantly lower in sediment AR02 compared to the ME21 and ME25 reference sediments (Table 7).

No additional survival effects were detected in phase I or Phase II of the study (Tables 7 and 9).

Biomass for sediments AR03, AR05, AR14 and AR17 was significantly lower than biomass observed for the AR21 reference (Tables 9 and 10). Biomass for sediments AR13 and AR26 was significantly higher than biomass observed for reference sediment AR25. Biomass measurements for most of the sediments evaluated exceeded measurements obtained for the performance control. Biomass was also generally greater for site sediments compared to the AR25 reference which demonstrated increases in biomass similar to the performance control. Biomass for some sediments (e.g., AR13 and AR26) exceeded the performance control by as much as 50%. In addition, the AR21 reference biomass measurements in phase I and phase II also exceed the performance control by 50%. The higher biomass observed in AR21 likely led to the significant differences detected between this reference and the site sediments. All site sediments found to have significantly lower biomass relative to the AR21 reference had biomass measurements similar to the performance control. The higher magnitude increases in biomass observed in some sediments relative to the performance control suggests that some component of the sediment, such as a contaminant or other factor, is positively influencing organism growth. Individual replicate survival and biomass data are provided in Table 5 and 6.

Table 1. Anacostia River *H. azteca* Day 0 Water Quality (Phase I)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
Control	A	8.05	9.37	100	100	320	2
Control	B	8.08	9.89				
Control	C	7.80	8.42				
Control	D	8.03	9.04				
Control	E	8.10	9.41				
Control	F	8.13	9.07				
AR02	A	7.88	9.79	100	120	270	5
AR02	B	7.94	9.89				
AR02	C	7.81	8.87				
AR02	D	7.61	6.96				
AR02	E	7.74	9.02				
AR02	F	7.97	9.08				
AR03	A	7.90	8.86	100	100	310	1
AR03	B	8.02	9.13				
AR03	C	8.00	9.92				
AR03	D	8.02	9.46				
AR03	E	8.04	9.44				
AR03	F	7.99	9.29				
AR05	A	7.91	9.17	80	48	260	5
AR05	B	7.81	9.65				
AR05	C	7.78	8.90				

Table 1. Anacostia River *H. azteca* Day 0 Water Quality (Phase I)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
AR05	D	7.78	9.00				
AR05	E	7.67	8.86				
AR05	F	8.83	9.50				
AR07	A	7.90	9.80	100	108	300	7
AR07	B	7.91	9.22				
AR07	C	7.95	9.26				
AR07	D	7.68	9.68				
AR07	E	7.67	9.89				
AR07	F	7.99	8.06				
AR08	A	7.88	7.28	60	128	280	3
AR08	B	7.83	9.02				
AR08	C	7.87	8.84				
AR08	D	7.80	8.58				
AR08	E	7.83	8.97				
AR08	F	7.82	8.62				
AR10	A	7.36	7.34	80	100	260	5
AR10	B	7.33	6.88				
AR10	C	7.67	8.97				
AR10	D	7.68	9.44				
AR10	E	7.29	7.88				
AR10	F	7.28	6.84				

Table 1. Anacostia River *H. azteca* Day 0 Water Quality (Phase I)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
AR13	A	7.55	9.23	80	120	310	4
AR13	B	7.72	9.43				
AR13	C	7.75	9.45				
AR13	D	7.68	9.53				
AR13	E	7.72	9.61				
AR13	F	7.70	8.95				
AR21	A	7.65	9.30	72	120	420	8
AR21	B	7.62	9.06				
AR21	C	7.69	9.46				
AR21	D	7.65	9.33				
AR21	E	7.56	8.87				
AR21	F	7.72	9.14				
AR25	A	8.11	9.68	80	80	270	4
AR25	B	7.72	9.40				
AR25	C	7.78	9.36				
AR25	D	7.75	9.17				
AR25	E	7.65	9.17				
AR25	F	7.84	9.84				
	Min	7.28	6.84	60	48	260	1
	Max	8.83	9.92	100	128	420	8

Table 2. Anacostia River *H. azteca* Day 28 Water Quality (Phase I)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
Control	A	8.35	6.70	127	108	290	<1
Control	B	8.17	6.40				
Control	C	8.07	5.83				
Control	D	7.95	6.09				
Control	E	7.93	6.22				
Control	F	7.91	5.99				
AR02	A	7.93	5.42	100	100	350	<1
AR02	B	7.81	5.00				
AR02	C	7.79	4.73				
AR02	D	7.80	4.90				
AR02	E	7.69	4.83				
AR02	F	7.70	4.70				
AR03	A	7.87	5.80	116	128	370	3
AR03	B	7.91	5.61				
AR03	C	7.90	5.60				
AR03	D	7.93	5.55				
AR03	E	7.96	5.83				
AR03	F	8.03	6.15				
AR05	A	7.86	6.14	118	120	370	<1
AR05	B	8.00	7.04				

Table 2. Anacostia River *H. azteca* Day 28 Water Quality (Phase I)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
AR05	C	7.99	6.03				
AR05	D	7.98	5.91				
AR05	E	7.99	5.27				
AR05	F	7.96	5.41				
AR07	A	7.96	5.75	88	92	320	<1
AR07	B	7.88	5.88				
AR07	C	7.87	5.81				
AR07	D	7.84	6.35				
AR07	E	7.81	6.52				
AR07	F	7.82	5.41				
AR08	A	7.79	6.10	100	108	320	<1
AR08	B	7.75	5.23				
AR08	C	7.70	5.07				
AR08	D	7.69	5.11				
AR08	E	7.71	5.83				
AR08	F	7.72	5.97				
AR10	A	7.70	5.82	100	108	370	<1
AR10	B	7.79	5.70				
AR10	C	7.78	5.66				
AR10	D	7.76	5.94				
AR10	E	7.73	5.65				

Table 2. Anacostia River *H. azteca* Day 28 Water Quality (Phase I)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
AR10	F	7.74	5.16				
AR13	A	7.79	6.62	80	112	350	<1
AR13	B	7.77	5.63				
AR13	C	7.74	4.97				
AR13	D	7.73	5.02				
AR13	E	7.77	5.02				
AR13	F	7.86	6.74				
AR21	A	7.85	6.82	122	104	330	<1
AR21	B	7.81	6.70				
AR21	C	7.81	5.54				
AR21	D	7.78	5.42				
AR21	E	7.74	5.14				
AR21	F	8.17	4.73				
AR25	A	8.22	7.22	100	108	330	<1
AR25	B	8.10	5.80				
AR25	C	8.04	5.75				
AR25	D	8.00	5.63				
AR25	E	7.97	5.48				
AR25	F	7.94	5.98				
	Min	7.69	4.70	80	92	290	<1
	Max	8.35	7.22	127	128	370	3

Table 3. Anacostia River *H. azteca* Day 0 Water Quality (Phase II)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
Control	A	8.13	9.16	100	120	340	1
Control	B	8.15	9.07				
Control	C	8.12	9.73				
Control	D	8.63	8.36				
Control	E	8.02	9.46				
Control	F	8.38	9.41				
AR14	A	7.96	9.87	125	108	280	5
AR14	B	7.94	9.82				
AR14	C	8.03	9.89				
AR14	D	8.11	9.84				
AR14	E	7.94	9.83				
AR14	F	7.79	9.32				
AR16	A	7.85	9.84	124	100	260	2
AR16	B	7.82	9.83				
AR16	C	7.84	9.97				
AR16	D	7.86	9.59				
AR16	E	7.97	9.82				
AR16	F	7.75	9.87				
AR17	A	8.20	9.14	122	109	310	4
AR17	B	8.03	9.38				
AR17	C	8.13	9.81				

Table 3. Anacostia River *H. azteca* Day 0 Water Quality (Phase II)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
AR17	D	8.10	9.71				
AR17	E	7.97	8.98				
AR17	F	7.94	9.92				
AR26	A	7.59	6.35	144	100	330	2
AR26	B	8.12	9.68				
AR26	C	8.04	8.92				
AR26	D	8.04	9.48				
AR26	E	8.03	9.40				
AR26	F	7.92	9.20				
AR27	A	7.97	9.94	120	120	340	6
AR27	B	8.14	9.78				
AR27	C	8.09	9.60				
AR27	D	8.13	9.53				
AR27	E	8.05	8.16				
AR27	F	7.95	8.15				
AR36	A	7.88	9.00	84	100	310	7
AR36	B	7.95	8.55				
AR36	C	7.58	7.39				
AR36	D	7.32	8.79				
AR36	E	8.05	9.82				
AR36	F	8.06	9.09				

Table 3. Anacostia River *H. azteca* Day 0 Water Quality (Phase II)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
AR21	A	7.91	9.62	120	120	450	8
AR21	B	8.06	9.53				
AR21	C	8.05	8.98				
AR21	D	8.12	9.28				
AR21	E	8.28	9.86				
AR21	F	8.14	9.63				
AR25	A	7.98	9.50	80	100	270	5
AR25	B	7.94	9.80				
AR25	C	7.82	9.73				
AR25	D	7.76	9.95				
AR25	E	7.80	9.85				
AR25	F	7.80	8.95				
	Min	7.32	6.35	80	100	260	1
	Max	8.63	9.97	144	120	450	8

Table 4. Anacostia River *H. azteca* Day 28 Water Quality (Phase II)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
Control	A	8.01	6.52	80	120	470	<1
Control	B	8.06	6.63				
Control	C	7.96	7.37				
Control	D	7.90	6.55				
Control	E	7.94	6.22				
Control	F	7.85	5.96				
AR14	A	7.65	6.00	110	120	360	<1
AR14	B	7.75	5.29				
AR14	C	7.76	4.98				
AR14	D	7.73	4.78				
AR14	E	7.77	4.82				
AR14	F	7.83	5.46				
AR16	A	8.02	5.71	100	128	360	<1
AR16	B	7.93	5.05				
AR16	C	7.91	4.65				
AR16	D	7.91	4.38				
AR16	E	7.87	5.28				
AR16	F	7.92	5.22				
AR17	A	7.82	4.37	120	128	400	<1
AR17	B	7.74	4.03				
AR17	C	7.63	4.05				

Table 4. Anacostia River *H. azteca* Day 28 Water Quality (Phase II)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
AR17	D	7.68	4.35				
AR17	E	7.60	4.62				
AR17	F	7.56	4.34				
AR26	A	7.84	5.25	130	120	470	<1
AR26	B	7.81	4.76				
AR26	C	7.81	4.83				
AR26	D	7.74	4.58				
AR26	E	7.69	4.59				
AR26	F	7.83	5.70				
AR27	A	7.79	6.22	100	124	360	<1
AR27	B	7.79	5.13				
AR27	C	7.88	4.36				
AR27	D	7.87	4.60				
AR27	E	8.10	5.07				
AR27	F	8.08	5.54				
AR36	A	7.80	4.98	100	128	400	<1
AR36	B	7.80	4.61				
AR36	C	7.87	4.90				
AR36	D	7.74	5.34				
AR36	E	7.74	4.95				
AR36	F	7.83	5.01				
AR21	A	7.81	6.37		124	350	<1

Table 4. Anacostia River *H. azteca* Day 28 Water Quality (Phase II)

Sediment	Replicate	pH	D.O. (mg/L)	Hardness (PPM Ca CO_3)	Alkalinity (PPM Ca CO3)	Conductivity (µS)	Ammonia (mg/L)
AR21	B	7.82	5.54				
AR21	C	7.75	5.34				
AR21	D	7.73	5.21				
AR21	E	7.82	5.50				
AR21	F	7.83	5.61				
AR25	A	7.86	5.54	100	120	360	<1
AR25	B	7.86	5.03				
AR25	C	7.86	4.71				
AR25	D	7.82	4.84				
AR25	E	7.86	4.79				
AR25	F	7.89	5.16				
	Min	7.56	4.03	80	120	350	<1
	Max	8.10	7.37	130	128	470	<1

Table 5. Anacostia River *H. azteca* Endpoint Data (Phase I)

Sediment	Replicate	Survival	% Survival	# on pan	pan weight (g)	pan & animal weight (g)	individual dry weight (mg)
Control	A	10	1.00	10	0.03561	0.03773	0.21200
Control	B	10	1.00	10	0.03597	0.03856	0.25900
Control	C	10	1.00	10	0.04074	0.04326	0.25200
Control	D	10	1.00	10	0.04489	0.04832	0.34300
Control	E	10	1.00	10	0.04381	0.04640	0.25900
Control	F	10	1.00	9	0.04599	0.04788	0.21000
AR02	A	0	0.00	0	N/A	N/A	N/A
AR02	B	0	0.00	0	N/A	N/A	N/A
AR02	C	1	0.10	1	0.03885	0.03923	0.38000
AR02	D	4	0.40	4	0.04260	0.04341	0.20250
AR02	E	0	0.00	0	N/A	N/A	N/A
AR02	F	0	0.00	0	N/A	N/A	N/A
AR03	A	8	0.80	8	0.03860	0.03985	0.15625
AR03	B	9	0.90	9	0.04503	0.04711	0.23111
AR03	C	10	1.00	10	0.04505	0.04651	0.14600
AR03	D	8	0.80	8	0.04769	0.04954	0.23125
AR03	E	10	1.00	10	0.03791	0.04040	0.24900
AR03	F	8	0.80	8	0.03902	0.04031	0.16125
AR05	A	10	1.00	10	0.04135	0.04328	0.19300
AR05	B	10	1.00	10	0.04191	0.04551	0.36000
AR05	C	10	1.00	10	0.04231	0.04486	0.25500
AR05	D	9	0.90	9	0.04393	0.04637	0.27111

Table 5. Anacostia River *H. azteca* Endpoint Data (Phase I)

Sediment	Replicate	Survival	% Survival	# on pan	pan weight (g)	pan & animal weight (g)	individual dry weight (mg)
AR05	E	9	0.90	9	0.04188	0.04433	0.27222
AR05	F	9	0.90	9	0.05431	0.05618	0.20778
AR07	A	10	1.00	10	0.04632	0.04925	0.29300
AR07	B	10	1.00	10	0.04693	0.04950	0.25700
AR07	C	6	0.60	6	0.04022	0.04192	0.28333
AR07	D	9	0.90	8	0.05371	0.05672	0.37625
AR07	E	10	1.00	10	0.05181	0.05588	0.40700
AR07	F	8	0.80	8	0.04465	0.04692	0.28375
AR08	A	8	0.80	8	0.04762	0.05066	0.38000
AR08	B	10	1.00	10	0.04835	0.05343	0.50800
AR08	C	8	0.80	8	0.06048	0.06302	0.31750
AR08	D	10	1.00	10	0.05601	0.06019	0.41800
AR08	E	10	1.00	10	0.05070	0.05210	0.14000
AR08	F	10	1.00	10	0.05505	0.05777	0.27200
AR10	A	9	0.90	9	0.04338	0.04626	0.32000
AR10	B	8	0.80	8	0.05614	0.05831	0.27125
AR10	C	10	1.00	10	0.05851	0.06249	0.39800
AR10	D	10	1.00	9	0.04674	0.05066	0.43556
AR10	E	10	1.00	10	0.04669	0.05039	0.37000
AR10	F	8	0.80	8	0.05035	0.05328	0.36625
AR13	A	9	0.90	9	0.04822	0.05185	0.40333
AR13	B	9	0.90	9	0.05146	0.05509	0.40333

Table 5. Anacostia River *H. azteca* Endpoint Data (Phase I)

Sediment	Replicate	Survival	% Survival	# on pan	pan weight (g)	pan & animal weight (g)	individual dry weight (mg)
AR13	C	10	1.00	10	0.05725	0.06095	0.37000
AR13	D	10	1.00	10	0.05336	0.05713	0.37700
AR13	E	10	1.00	10	0.04850	0.05328	0.47800
AR13	F	9	0.90	9	0.04248	0.04554	0.34000
AR21	A	8	0.80	8	0.04319	0.04700	0.47625
AR21	B	9	0.90	9	0.04706	0.05052	0.38444
AR21	C	10	1.00	10	0.04536	0.04946	0.41000
AR21	D	9	0.90	9	0.04959	0.05448	0.54333
AR21	E	9	0.90	9	0.03715	0.04175	0.51111
AR21	F	8	0.80	8	0.05082	0.05325	0.30375
AR25	A	9	0.90	10	0.04576	0.04772	0.19600
AR25	B	10	1.00	10	0.04907	0.05184	0.27700
AR25	C	10	1.00	10	0.05409	0.05689	0.28000
AR25	D	9	0.90	10	0.05621	0.05867	0.24600
AR25	E	9	0.90	9	0.05287	0.05521	0.26000
AR25	F	10	1.00	10	0.04804	0.05101	0.29700

Table 6. Anacostia River *H. azteca* Endpoint Data (phase II)

Sediment	Replicate	Survival	% Survival	# on pan	pan weight (g)	pan & animal weight (g)	individual dry weight (mg)
Control	A	8	80%	8	0.05589	0.05805	0.27000
Control	B	7	70%	7	0.05664	0.05895	0.33000
Control	C	9	90%	10	0.06164	0.06479	0.31500
Control	D	10	100%	10	0.05816	0.06107	0.29100
Control	E	10	100%	10	0.05728	0.05991	0.26300
Control	F	9	90%	9	0.05665	0.05940	0.30556
AR14	A	10	100%	10	0.04920	0.05221	0.30100
AR14	B	10	100%	10	0.06060	0.06382	0.32200
AR14	C	9	90%	9	0.06650	0.06916	0.29556
AR14	D	9	90%	9	0.07039	0.07347	0.34222
AR14	E	9	90%	9	0.06226	0.06528	0.33556
AR14	F	9	90%	9	0.05419	0.05628	0.23222
AR16	A	9	90%	9	0.05939	0.06289	0.38889
AR16	B	10	100%	10	0.05642	0.05971	0.32900
AR16	C	10	100%	9	0.05369	0.05700	0.36778
AR16	D	9	90%	9	0.06052	0.06384	0.36889
AR16	E	9	90%	8	0.06717	0.07013	0.37000
AR16	F	9	90%	9	0.05499	0.05890	0.43444
AR17	A	10	100%	10	0.06112	0.06373	0.26100
AR17	B	9	90%	9	0.05241	0.05592	0.39000
AR17	C	9	90%	9	0.06130	0.06399	0.29889
AR17	D	10	100%	10	0.06528	0.06833	0.30500

Table 6. Anacostia River *H. azteca* Endpoint Data (phase II)

Sediment	Replicate	Survival	% Survival	# on pan	pan weight (g)	pan & animal weight (g)	individual dry weight (mg)
AR17	E	10	100%	10	0.06932	0.07172	0.24000
AR17	F	9	90%	9	0.05259	0.05585	0.36222
AR26	A	9	90%	9	0.06223	0.06711	0.54222
AR26	B	10	100%	10	0.05776	0.06184	0.40800
AR26	C	0	0%	0	N/A	N/A	N/A
AR26	D	9	90%	9	0.06544	0.06989	0.49444
AR26	E	8	80%	8	0.06770	0.07130	0.45000
AR26	F	8	80%	8	0.06604	0.06941	0.42125
AR27	A	0	0%	0	N/A	N/A	N/A
AR27	B	0	0%	0	N/A	N/A	N/A
AR27	C	10	100%	10	0.05499	0.05835	0.33600
AR27	D	10	100%	10	0.06253	0.06703	0.45000
AR27	E	8	80%	8	0.06607	0.06900	0.36625
AR27	F	10	100%	10	0.06448	0.06777	0.32900
AR36	A	9	90%	9	0.06508	0.06810	0.33556
AR36	B	10	100%	10	0.06789	0.07113	0.32400
AR36	C	10	100%	10	0.07401	0.07678	0.27700
AR36	D	10	100%	10	0.07404	0.07716	0.31200
AR36	E	9	90%	9	0.06501	0.06868	0.40778
AR36	F	6	60%	6	0.06895	0.07196	0.50167
AR21	A	8	80%	8	0.07287	0.07662	0.46875
AR21	B	10	100%	10	0.06865	0.07330	0.46500

Table 6. Anacostia River *H. azteca* Endpoint Data (phase II)

Sediment	Replicate	Survival	% Survival	# on pan	pan weight (g)	pan & animal weight (g)	individual dry weight (mg)
AR21	C	8	80%	8	0.05539	0.05785	0.30750
AR21	D	7	70%	7	0.06400	0.06702	0.43143
AR21	E	6	60%	6	0.05680	0.05971	0.48500
AR21	F	7	70%	6	0.06238	0.06460	0.37000
AR25	A	8	80%	8	0.06299	0.06537	0.29750
AR25	B	10	100%	10	0.06230	0.06588	0.35800
AR25	C	10	100%	10	0.06856	0.07145	0.28900
AR25	D	9	90%	9	0.06774	0.07077	0.33667
AR25	E	8	80%	8	0.06047	0.06302	0.31875
AR25	F	8	80%	8	0.05896	0.06185	0.36125

Table 7. Anacostia River *H. azteca* percent survival following 28-d exposure (Phase I)

Sediment		Mean	S.D.	S.E.	C.V.	n
Control		100.0%	0.0%	0.0%	0.00%	6
AR02	* #	8.3%	16.0%	7.2%	192.25%	6
AR03		88.3%	9.8%	4.4%	11.13%	6
AR05		95.0%	5.5%	2.4%	5.77%	6
AR07		88.3%	16.0%	7.2%	18.14%	6
AR08		93.3%	10.3%	4.6%	11.07%	6
AR10		91.7%	9.8%	4.4%	10.73%	6
AR13		95.0%	5.5%	2.4%	5.77%	6
AR21 (Reference)		88.3%	7.5%	3.4%	8.52%	6
AR25 (Reference)		95.0%	5.5%	2.4%	5.77%	6

Table 8. Anacostia River *H. azteca* individual dry weight following 28-d exposure (Phase I)

Sediment		Mean	S.D.	S.E.	C.V.	n
Control		0.26	0.05	0.02	18.87%	2
AR02		0.29	0.13	0.06	43.09%	2
AR03	*	0.20	0.05	0.02	23.48%	6
AR05	*	0.26	0.06	0.03	22.78%	6
AR07		0.32	0.06	0.03	18.96%	6
AR08		0.34	0.13	0.06	37.53%	6
AR10		0.36	0.06	0.03	16.08%	6
AR13	#	0.40	0.05	0.02	11.87%	6
AR21 (Reference)		0.44	0.09	0.04	20.31%	6
AR25 (Reference)		0.26	0.04	0.02	13.74%	6

* Signifcantly different from AR21 reference sediment (Dunnett's Means Comparison; α=0.05).

Signifcantly different from AR25 reference sediment (Dunnett's Means Comparison; α=0.05).

Table 9. Anacostia River *H. azteca* percent survival following 28-d exposure (Phase II)

Sediment		Mean	S.D.	S.E.	C.V.	n
Control		88.3%	11.7%	5.2%	13.23%	6
AR14		93.3%	5.2%	2.3%	5.53%	6
AR16		93.3%	5.2%	2.3%	5.53%	6
AR17		95.0%	5.5%	2.4%	5.77%	6
AR26		73.3%	36.7%	16.4%	50.04%	6
AR27		63.3%	49.7%	22.2%	78.42%	6
AR36		90.0%	15.5%	6.9%	17.21%	6
AR21 (Reference)		76.7%	13.7%	6.1%	17.82%	6
AR25 (Reference)		88.3%	9.8%	4.4%	11.13%	6

Table 10. Anacostia River *H. azteca* individual dry weight following 28-d exposure (Phase II)

Sediment		Mean	S.D.	S.E.	C.V.	n
Control		0.30	0.03	0.01	0.09	6
AR14	*	0.30	0.04	0.02	0.13	6
AR16		0.38	0.03	0.02	0.09	6
AR17	*	0.31	0.06	0.03	0.19	6
AR26	#	0.46	0.06	0.02	0.12	5
AR27		0.37	0.06	0.02	0.15	4
AR36		0.36	0.08	0.04	0.23	6
AR21 (Reference)		0.42	0.07	0.03	0.16	6
AR25 (Reference)		0.33	0.03	0.01	0.09	6

* Signifcantly different from AR21 reference sediment (Dunnett's Means Comparison; α=0.05).

Signifcantly different from AR25 reference sediment (Dunn's Means Comparison; α=0.05).

Appendix K: UMBC Laboratory Report: Detailed Carbon Chemistry

Assessment of sediment carbon types and nature of PAH binding to provide a mechanistic explanation of observed bioavailability and toxicity of PAHs in Anacostia River sediments.

Final Project Report
Submitted to
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1.0.Background:

The geochemistry of sediments at industrial sites is often impacted by the addition of anthropogenic organic materials such as soot, coal, coke, pitch, coal tar, and oils that alter the natural organic carbon makeup of the sediments. Exposure of sediment-bound contaminants to benthic organisms is affected by the nature of organic carbon that binds the contaminants in sediments. Thus, understanding PAH bioavailability and aqueous partitioning for MGP-impacted sediments requires: 1) the correct understanding of the types of organic carbon responsible for PAH binding in sediments, and 2) accurate description of the partitioning characteristics of the appropriate organic carbon phases (Khalil et al. 2006).

In recent work involving PAH-contaminated soils from oil-gas manufacturing facilities, Hong et al. (2003) found that site-specific measurement of partitioning for lampblack soot-impacted sediments may result in 1-2 orders of magnitude higher values of measured K_{oc} (organic carbon-water partition coefficient) compared to the values predicted using octanol-water partition coefficient (K_{ow}) -K_{oc} correlations for natural organic matter. Data compiled by Ghosh et al. (2003) show that the log partition coefficient (log K_{oc}) for phenanthrene sorbed on black carbon (BC) is 6.6 compared to a log K_{oc} of 4-4.5 for natural organic carbon. Thus, if PAHs are sorbed on BC, the aqueous availability may be much lower than PAHs associated with natural organic matter. Several recent papers have shown that elevated PAH partitioning in sediment samples could be explained based on the BC content and known high PAH sorption capacity of BC (Gustafsson et al. (1997); Jonker and Koelmans, (2001); Bucheli and Gustafsson (2000)). Working with isolated BC, Accardi-Dey and Gschwend (12) and Lohmann et al. (13) demonstrated that the observed PAH partitioning from sediment can be better predicted by a modified BC-inclusive distribution model.

Work by Khalil et al (2006) demonstrated that majority of the PAHs in Manufactured Gas Plant (MGP)-impacted sediments remain associated with weathered coal-tar pitch particles. The research demonstrated that aqueous partition coefficients for PAHs in weathered pitch samples from the field are generally an order of magnitude higher than reported for natural organic matter partitioning, and match well with theoretical predictions based on a coal tar-water partitioning model. A mechanistically sound modeling approach to PAH partitioning can improve site-specific risk assessment for MGP sites and reduce uncertainty in the process of evaluating long-term effects of remedial actions.

2.0. Research Objectives.

The overall objective of the present research was to assess the feasibility of using two site-specific measurement tools of contaminant bioavailability (porewater analysis using SPME and supercritical fluid extraction) to improve the prediction of sediment toxicity. For a new approach of predicting site-specific toxicity to be widely accepted by the scientific community, there is a need to develop a good understanding of the fundamental mechanistic underpinnings that cause elevated or reduced bioavailability of compounds. This research therefore focuses on understanding contaminant association and binding to provide a mechanistic interpretation of the results from toxicity tests and PAH porewater analysis. Four samples were selected to perform a detailed particle-scale assessment of PAH location and binding. These four samples span the range of low to high PAH concentration and PAH availability as measured by porewater analysis. Shown in Table 1 are summary results from the University of North Dakota on the PAH and SPME porewater analysis of the 17 Anacostia River sediment samples.

The four selected samples (shown in grey highlight in Table 1) included two with the highest PAH concentrations, one showing high porewater PAH toxic units and low Hyalella survival (AR02) and the other showing three orders of magnitude lower porewater PAH toxic units and high Hyalella survival (AR13). The third sample selected was AR03 which has the lowest PAH concentration and relatively high porewater PAHs, possibly caused by the low TOC in that sediment. The fourth sample selected was AR16 with 46 mg/kg PAHs in sediment and relatively high soot carbon and low porewater PAH toxic units.

The detailed assessment of contaminant association included size and density separation of particulate organic matter in sediments, particle-scale PAH measurements, and petrographic characterizations to identify the nature of the organic matter responsible for PAH binding. Identification of the geochemical nature of the soil carbon, specifically the presence of coal, coke, lampblack, pitch, and tar can help explain any differences in site-specific values of PAH partitioning from what would have been estimated using standard empirical correlations. Brief descriptions of the proposed methods to assess contaminant binding and particle-scale association are provided below.

3.0 Experimental Procedures

3.1. Size and density separation

Wet sieving was carried out to separate the sediment in four size fractions (< 63 μm, 63-250 μm, 250-1000 μm, and > 1000 μm). The size fractionized samples were further density separated using saturated cesium chloride solution (Specific gravity = 1.8). About 20 ml of wet sediment were added into 60 ml of cesium chloride solution and swirled in a beaker. The supernatant that included floating particles were drained off to a filter paper, resulting in two separate fractions defined as light and heavy. This process was repeated several times until no floating particles were observed. Both fractions were washed with DI water several times to remove cesium chloride and air-dried on watch glasses. Each of the size and density separated fractions were then analyzed for PAHs.

3.2. Separation of individual particle types

To investigate differences in PAH abundances among particle types in the sediment samples, a small quantity of sediment particles in the 250-1000 μm size fraction was manually separated under the microscope into the four most dominant particle types: wood, coal, coke, pitch or sand. Several particles of each kind were weighed and extracted in 10 mL glass vials with Teflon-lined caps using a 50:50 mixture of methylene chloride and acetone and analyzed for PAHs.

3.3. PAH extraction and analysis

PAH extraction of size and density separated samples were performed using three successive ultrasonic extraction (30 s on and 30 s off pulsing for a total 6 min) with a hexane/acetone mixture (1:1 by vol) following EPA method 3550B. The extracts were combined and cleaned using an activated silica gel column as outlined EPA method 3630C. A gas chromatograph (GC, Agilent model 6890N) equipped with A 0.25 um bonded fused silica capillary column (Agilent DB-5MS, 60 m x 0.25 mm ID) and a mass spectrometer (Agilent model 5973) was used for PAH analysis based on EPA method 8270 C for 16 parent PAHs. For PAH extraction and analysis of isolated organic particle types, the samples were weighed and placed in 10 ml glass vials which were capped and placed in a sonicator bath for three successive ultrasonic extractions (30 s on and 30 s off pulsing for a total 6 min) with a hexane/acetone mixture (1:1 by vol).

4.0. Results and Discussion

Each of the four sediment samples was composed of a mixture of sand, silt, clay, and organic debris mixed in with anthropogenic organic particles such as coal, coke, and pitch. Sediment samples AR13 and AR 16 contained a noticeably large fraction of vegetative debris along with with fine silty particles. Sediment sample AR02 was strikingly different from the others with a strong hydrocarbon odor and presence of an oil sheen observed on the surface of the overlying water. Whole sediment extraction and PAH analysis was performed in duplicate samples of each sediment to confirm results from earlier measurements and to ensure that the sediment subsamples used for the organic characterization study is not very different from the ones used for toxicity tests and porewater analysis. As shown in Table 2 (columns 2 and 3), the total PAH concentrations (16 EPA priority pollutant PAHs) in three of the sediment samples (AR02, AR03, and AR16) are very close between the two subsamples analyzed in different laboratories. However, for sample AR13, the two analyses are about an order of magnitude apart. The subsample used for organic characterization at UMBC had only 44 mg/kg PAHs compared to 504 mg/kg measured in the subsample used in porewater analysis and toxicity studies. Thus, the results of the various characterization studies for this sample will need to be interpreted carefully. In general, three and four ring PAH compounds, such as phenanthrene, fluoranthene, and pyrene, were the most abundant PAHs in each of the four samples analyzed.

4.1. Size and density separation, and analysis

Figures 1 (a) through (d) present the mass fraction of sediment in each size and density class of the four sediment samples, AR02, AR03, AR13 and AR16, respectively. Generally, the heavy density particles are distributed across the size ranges separated, with two noticeable differences: Sediment AR02 had the highest mass abundance of the heavy particles in the smallest size range (<0.063 mm) with decreasing abundance with increasing size. Sediment AR03 had the opposite trend with the highest mass abundance of heavy particles in the largest size range (> 1 mm) with decreasing abundance with decreasing size. This indicates that AR02 is dominated by the clay/silt fraction whereas AR03 is dominated by medium to coarse sand particles. For the light density sediment fraction, the largest abundance is in the higher particle size (> 1mm) which is mostly composed of vegetative debris.

The PAH mass distribution across the size and density fractions is shown in Figures 2 (a) through (d) and the concentration in each particle fraction is presented in Figures 3 (a) through (d) for the four sediment samples. As shown in Figure 2, the PAH mass in the sediment is distributed unevenly across the particle size ranges. Although the lighter density

particles have a much higher PAH concentration compared to the heavier density particles (expected based on preferential sorption of PAHs to organic matter; see Figure 3), the higher abundance of the heavier particles changes the % PAH distribution. For example, sediment samples AR02 and AR03 each have about 70% of the total PAHs associated with heavy mineral particle types. Such a distribution can be indicative of a PAH oil phase contamination where the oil phase containing PAHs is smeared on particle surfaces. This is most strikingly evident in sediment sample AR02 where both PAH abundance and PAH concentration on particle classes increases with decreasing size for the heavier density particles. Such a distribution is indicative of a surface adsorption or coating phenomena. In contrast, sediment samples AR13 and AR16 have 40 % and 25% of total PAHs associated with the heavier density fraction and the majority of the PAHs for these two sediment samples are associated with the lighter density organic particles. Among the lighter density particles, the highest PAH concentration and mass abundance was observed mostly in the 0.25-1.0 mm size range. Thus, the lighter density particles from this size fraction were used for particle-scale PAH analysis and organic petrography analysis to evaluate the PAH association with different carbon forms present in the sample.

4.2. Microscale particle separation and PAH analysis

To investigate differences in PAH abundances among individual particle types in the sediment samples, a small quantity of sediment particles in the 250-1000 μm size fraction was manually separated under the microscope into four dominant particle types: coal, coke, pitch, sand, or wood. Figure 4 shows example light microscopy images of the AR02 sediment particles in 0.25-1.0 mm size fraction.

PAH concentration on isolated sediment particles is shown in Figure 5 for the four sediment samples. As expected from high PAH partitioning to coal, coke, and pitch particles, these particle types generally have high PAH concentration in the range of several hundred to a thousand mg/kg. Comparing sediment samples AR02 and AR13, it is observed that although PAH concentrations on the coal particles are similar in these two sediments, there is a striking difference in the PAH concentration on wood particles between these sediment samples. Sample AR02 stands out with a very high PAH concentration on wood particles of about 1,100 mg/kg PAHs. Such high abundance of PAHs on wood particles is indicative of high PAH availability in this sample. Sample AR03 shows majority of the PAHs associated with coal with very little on the wood. Sample AR16 has low PAH concentrations on all isolated particle types in the tens of mg/kg PAH compared to hundreds of mg/kg in the other samples (notice differences in the concentration scales for the four sediment samples illustrated in Figure 5).

4.3. Petrography analysis of isolated organic particles

Petrography analysis was performed on the lighter density size fraction with the highest abundance of PAHs (0.25 – 1.0 mm). A summary of petrographic compositional analysis results is presented in Table 3 and sample images of the analysis identifying characteristic particles found in each sample are presented in Appendix A.

Sample AR-02 (0.25-1.0 mm lighter density fraction)

The sample AR-02 (0.25-1.0 mm lighter density fraction) has 66.0% by volume of total industry related carbons which consist of coal, coke and byproduct carbons (coal tar pitch). Bituminous coal is the most abundant industrial related carbon in the current sample at 27.4% and it appears to be metallurgical grade coal which range from high to low in volatile content. By far, this sample had the highest volume fraction of coal/coke particles which is also corroborated by the highest soot carbon fraction measured in this whole sediment sample (3.7%; see Table 2). The AR-02 sample contains 9.4% of anthracite coal, followed by 2.2% of thermally softened coal and 2.2% of oxidized coal, as listed in Table 1 and shown in Figures 1 through 5. Most of the oxidized coal appears to be related to weathering rather than thermal alteration. There is 14.0% of total carbonized material which consist mostly of metallurgical coke at 9.2%, with 1.8% coked coal inerts, 1.2% of carbon black, 1.0% of charcoal and 0.4% of burnt coke and depositional carbon. The depositional carbon occur as coatings on the metallurgical coke and are a product of cracked vapor and gas phase hydrocarbons during the coking process. There is 10.2% of total by-product related material from coke making, which consists of 3.6% low to medium QI coal tar pitch, with 3.0% of high QI pitch, 2.6% of pitch mixed with other materials and 1.0% of coked cenospheres. The coal tar pitch is a by-product of the coking process in which the tar has been converted to a pitch by a distillation process. There is 9.0% of other carbon related materials which consist of 6.2% green organic plant material with cellular structure and 2.8% of woody plant material. The AR-02 sample contains 25.0% of total mineral matter which consists mostly (14.4%) of fine grained groundmass minerals which resemble clay or sediment, with 5.4% of slag and slag with metallic inclusions, 1.6% of diatoms, 0.8% of pyrite and 0.4% of quartz. Approximately (8.4%) half of the groundmass minerals occur as coatings on the various carbon materials which occur in this sample. The petrographic composition data is listed in Table 1 and most of the materials are illustrated in Figures 1 through 5.

The most abundant industrial related carbon in the AR-02 sample is bituminous coals at 27.4%, followed by anthracite coal at 9.2% and metallurgical coke at 9.2%. The AR-02 sample contains the most coal, coke and by-product related carbons and the least amount of plant material and mineral matter in the current group of samples.

Sample AR-03 (0.25-1.0 mm lighter density fraction)

The sample AR-03 (0.25-1.0 mm lighter density fraction) has 32.6% by volume of total industry related carbons which consist of coal, coke and byproduct carbons (coal tar pitch). Anthracite coal is the most abundant industrial related carbon in the current sample at 14.0%, followed by 4.6% of oxidized coal, with 2.2% of thermally softened coal and 2.2% of bituminous coal, as listed in Table 1 and shown in Figures 6 through 10. Anthracite is much higher in rank or maturity than bituminous and its specific place in the ranking system can be determined by vitrinite reflectance. There is 6.8% of total carbonized material which consists mostly of metallurgical coke at 5.4%, with lesser amounts of graphite, depositional carbon and coked coal inerts. There is 2.6% of total by-product related material from coke making, which consists of 1.6% tar or gum and 1.0% of high QI coal tar pitch. There is 22.4% of other carbon related materials which consist of 17.0% green plant material with cellular structure and 5.4% of woody plant material. The AR-03 sample contains 45.0% of total mineral matter which consists mostly (23.4%) of fine grained groundmass minerals, with 5.5% of a white grainy mineral material which might be slag, 7.6% of diatoms, 6.6% of a material in which the cellular structure has been replaced with minerals and 0.8% of iron oxide. The petrographic data is listed in Table 1 and most of the materials are illustrated in Figures 6 through 10. The AR-03 sample has the highest amount of diatoms and the current group of samples and the material in which the cellular structure has been replaced with minerals may also be some form of diatomaceous earth since both appear as fossilized remains.

Sample AR-13 (0.25-1.0 mm lighter density fraction)

The sample AR-13 (0.25-1.0 mm lighter density fraction) has only 3.6% by volume of total industry carbon related materials. The AR-13 sample contains a very small amount of low volatile bituminous coal at 0.4% and a small amount of oxidized coal at 0.6%. There is no microscopic evidence of any carbonized material such metallurgical coke in the current sample. There is 2.6% of by-product related material which consists of 1.0% of low to medium QI coal tar pitch, with 0.8% of high QI pitch, 0.6% of gum or tar like material and 0.2% of coked cenospheres. The most abundant material in the AR-13 sample is related to organic plants which consist of 47.8% green plant material with cellular structure and 12.2% of woody plant material, as listed in Table 1 and shown in Figures 11 through 14. The AR-13 sample contains 36.4% of total mineral matter which consists mostly (21.6%) of fine grained groundmass minerals, with 5.4% of a white grainy mineral material with different colored mineral inclusions, 4.6% of diatoms, 1.8% iron oxide, 2.2% quartz and other transparent minerals and 0.2% of pyrite. The petrographic data is listed in Table 1 and most of the materials are illustrated in Figures 11 through 14. The AR-13 sample has the lowest amount of coal and coked constituents and the highest amount of plant related material in the

current group of samples.

Sample AR-16 (0.25-1.0 mm lighter density fraction)

The sample AR-16 (0.25-1.0 mm lighter density fraction) has 22.4% by volume of total industrial related carbons which consist of coal, coke and byproduct carbons (coal tar pitch). Bituminous coal and metallurgical coke are the two most abundant industrial related carbons in the current sample with 6.6% of bituminous coal and 8.0% of metallurgical coke and coked coal inerts, as listed in Table 1 and shown in Figures 15 through 18. In addition the AR-16 sample contains 1.8% of anthracite coal, 1.2% of oxidized coal, 1.6% of coal inerts and 1.2% of charcoal or charred wood. There is a relatively small amount of by-product related carbon which consists of 1.0% tar or gum and 0.8% of cenospheres. The cenospheres are associated with the by-product related material since their circular shape and porous microstructure indicate the coal particles were carbonized rapidly in unconfined space while metallurgical coke contains thicker coke walls and has a much denser microstructure since the coal particles are carbonized under confinement in the coke oven chamber. There is 44.4% of other carbon related materials which consist of 32.6% green plant material with cellular structure and 11.8% of woody plant material. The AR-16 sample contains the second highest amount of organic plant material in the current group of samples. There is 33.2% of total mineral matter which consists mostly of fine grained groundmass minerals and coatings at 9.6% and a milky white slag like mineral with grainy texture at 10.2%. There is also a small amount of transparent minerals at 2.8% and 1.8% of diatoms. The petrographic data is listed in Table 1 and most of the materials are illustrated in Figures 15 through 18.

4.4. Overall findings and conclusions

Summary findings and conclusions based on the organic particle characterization for the four sediment samples are provided below.

(a) **AR02**: Total PAH concentration in this sample is 886±73 mg/kg dry. This sediment is toxic to Hyalella resulting in less than 10 % mean survival of the organism in a 28-day exposure test. The TU_{34} is more than an order of magnitude higher than one indicating acute toxicity of the sediment due to elevated PAHs. This sediment sample is odorous and a sheen of oil was visible on the top of the overlying water indicating that an excess free phase of tar/oil was present that causes high porewater toxic units and Hyalella mortality. Clays and silt are the most abundant component of the sediment and a majority of the PAHs are associated with this fine fraction as expected for a sediment with a free oil phase that can coat particle surfaces and be

enriched in the finer size mineral fraction. It is important to note that this sediment also had the highest TOC and soot carbon fractions. Petrography analysis also revealed an abundance of coal and coke derived particles in the organic fraction. However, in the presence of a free oil phase, the black carbon particles are inactivated and behave no different from wood particles as evidenced by similar PAH concentrations on wood and coal particles in this sediment. Thus, interpretation of the role of black carbon on PAH partitioning in sediment needs to be interpreted carefully when there is free oil phase present as discussed in detail by Hong et al. (2003).

(b) **AR03**: This sediment is made up of coarse sandy particles and contains low PAHs (15±2 mg/kg dry) compared to other samples tested in this report. More than 59% of the sediment constitutes heavy and larger than 1 mm particles and the sum of light sediment mass is less than 1%. Majority of the PAHs in this sediment is associated with the heavy density mineral particles which may explain why in this sediment with the lowest PAH concentration, the porewater toxic units (0.427) is higher than some of the other sediments with higher PAH concentrations. Although this sample contains some coal and coke particles in the organic fraction, the overall abundance of organic particles is small evidenced also by the low TOC value of 0.88%. Hyalella survival in this sediment was 88% which may indicate some low level of toxicity even at this low PAH concentration in sediment.

(c) **AR13:** As described earlier, the subsample used for organic characterization at UMBC had only 44 mg/kg PAHs compared to 504 mg/kg measured in the subsample used in porewater analysis and toxicity studies. Thus, the results of the various characterization studies for this sample will need to be interpreted with caution. This sediment was a mixture of various sized particles with a large fraction of wood debris. More than 50 % of total PAH in this sediment is associated with the light density 0.25-1.0 mm sized particles which comprises only 4 % of total sediment mass. Within this size fraction, coal and weathered pitch particles had the highest PAH concentration (600-1000 mg/kg) which was about 2 orders of magnitude higher than the PAH concentration on wood particles. This sediment sample exemplifies a case where the coal and pitch particles are exhibiting high sorption capacity for PAHs as expected and are the primary reservoirs of PAHs in the sediment. As expected, this sediment also demonstrates low porewater PAHs and toxic units, and is found to be non toxic to Hyalella (95% survival).

(d) **AR16**: This sediment is similar to AR13 in PAH concentration and distribution

among particle classes. Majority of the PAHs in this sediment are associated with the light density 0.25-1.0 mm sized particles which comprises only 5% of total sediment mass. However, the total PAH concentration on individual particle types in this sediment is much lower compared to that in sample AR13. Wood particles in this sediment appear to have similar PAH concentration compared to coal and coke particles (Figure 5). However, petrography analysis reveals that some of the wood particles are partially charred to form charcoal that can have a high affinity for PAHs. As expected based on the low PAH concentration and abundance of strongly sorbing coal, coke, and charcoal particles, this sediment demonstrates low porewater PAHs and toxic units and is found to be non toxic to Hyalella (93% survival).

Generally, the results from particle separation and organic characterization of sediment samples from the Anacostia River support the observations of strong sorption of PAHs to sediment and reduced toxicity to Hyalella. The strong sorption is explained by the association of PAHs with coal, coke, charcoal, and weathered pitch particles found in the sediment when the sorption capacity of the geosorbents is not attenuated by the presence of a free oil phase. Presence of an excess oil phase in sediment can complicate interpretations using black carbon analysis as the sole tool to assess PAH bioavailability and toxicity in sediments. However, porewater PAH analysis and toxic unit calculation provides an accurate assessment of PAH availability in sediments even within the complex interactions between different geosorbent forms and a free oil phase in sediment.

Table 1. Anacostia River Sediment and porewater PAH concentrations.

Sample Type	RETEC ID	Total PAH_{16} (mg/kg)	SPME Porewater TU_{34}
TEST	AR03	19.2	0.427
TEST	AR08	29.1	0.007
REFERENCE	AR25	29.3	0.010
REFERENCE	AR25	29.3	0.010
TEST	AR07	31.3	0.021
TEST	AR27	34.3	0.007
TEST	AR10	35.9	0.013
TEST	AR17	40.0	0.066
TEST	AR36	40.6	0.012
TEST	AR16	45.6	0.007
TEST	AR14	47.4	0.017
TEST	AR05	47.8	0.010
REFERENCE	AR21	51.4	0.010
REFERENCE	AR21	51.4	0.010
TEST	AR26	87.2	0.062
TEST	AR13	514	0.020
TEST	AR02	800	62.1
CONTROL	AR_Control	NA	NA
CONTROL	AR_Control	NA	NA

Table 2. Sediment properties and toxicity test results of the four selected sediment samples

Sample ID	Total PAH16 (mg/kg) UMBC	Total PAH16 (mg/kg) (UND)	SPME porewater TU34	TOC Wt % dry	SOC Wt % dry	Hyalella Azteca (28 day)			
						Survival		Growth	
						mean	n	mean	n
AR02	886±73	800	62.2	9.4± 0.12	3.70± 0.13	8.3± 16	6	0.29± 0.13	6
AR03	15±2	19.2	0.427	0.88± 0.14	0.18± 0.03	88± 10	6	0.20± 0.05	6
AR13	44±4	514	0.020	4.89± 0.26	0.67± 0.30	95± 5.5	6	0.40± 0.05	6
AR16	45± 8	45.6	0.007	6.64± 0.32	1.64± 0.28	93± 5.2	6	0.38± 0.03	6

Table 3. Petrographic compositional analysis of the sediment organic particle fraction in the size range of 0.25 – 1.0 mm.

SAMPLE NAME	AR-02	AR-03	AR-13	AR-16
	Volume %	*Volume %*	*Volume %*	*Volume %*
Carbons				
Softened Coal	2.2	2.2	---	---
High Vol. Bituminous Coal	14.8	0.4	---	4.6
Medium Vol. Bituminous Coal	7.8	0.8	---	0.6
Low Vol. Bituminous Coal	4.8	1.0	0.4	1.4
Anthracite Coal	9.4	14.0	---	1.8
Oxidized Coal	2.2	4.6	0.6	1.2
Coal Inerts	0.6	0.2	---	1.6
Total Coal	41.8	23.2	1.0	11.2
Metallurgical Coke	9.2	5.4	---	6.0
Depositional Carbon on Coke	0.4	0.4	---	---
Burnt Coke	0.4	---	---	---
Coked Inert	1.8	0.2	---	2.0
Carbon Black	1.2	---	---	---
Charcoal	1.0	---	---	1.2
Graphite	---	0.8	---	0.2
Total Coke/Carbon	14.0	6.8	0.0	9.4
Coal Tar Pitch Low to Medium QI	3.6	---	1.0	---
Coal Tar Pitch High QI	3.0	1.0	0.8	---
Coal Tar Pitch Coating/Mixed	2.6	---	---	---
Cenosphere	1.0	---	0.2	0.8
Gum or Tar	---	1.6	0.6	1.0
Total Byproduct Related	10.2	2.6	2.6	1.8
Woody Plant Material (cellular structure)	2.8	5.4	12.2	11.8
Green Plant Material (cellular structure)	6.2	17.0	47.8	32.6
Total Plant Material	9.0	22.4	60.0	44.4
Mineral Matter				
Groundmass Minerals	8.4	20.8	18.8	8.2
Groundmass Mineral Coating Plant/Carbon	5.0	1.4	2.2	1.2
Groundmass Mineral Mixed - Plant/Carbon	3.4	1.2	1.2	0.2
Glassy Slag	3.2	---	---	8.4
Slag with Metallics	2.2	---	---	---
Quartz	0.4	---	1.2	---
Transparent Mineral	---	1.4	1.0	2.8
White Grainy Mineral w/ red & green Mineral Inclusions	---	---	5.4	---
White Mineral with Milky to Grainy Texture	---	5.8	---	10.2
Pyrite	0.8	---	0.2	---
Iron Oxide	---	0.8	1.8	0.4
Plant Like Structure w/Mineral Matter Walls	---	6.6	---	---
Diatom	1.6	7.0	4.6	1.8
Total Mineral Matter	25.0	45.0	36.4	33.2
Grand Total	100.0	100.0	100.0	100.0

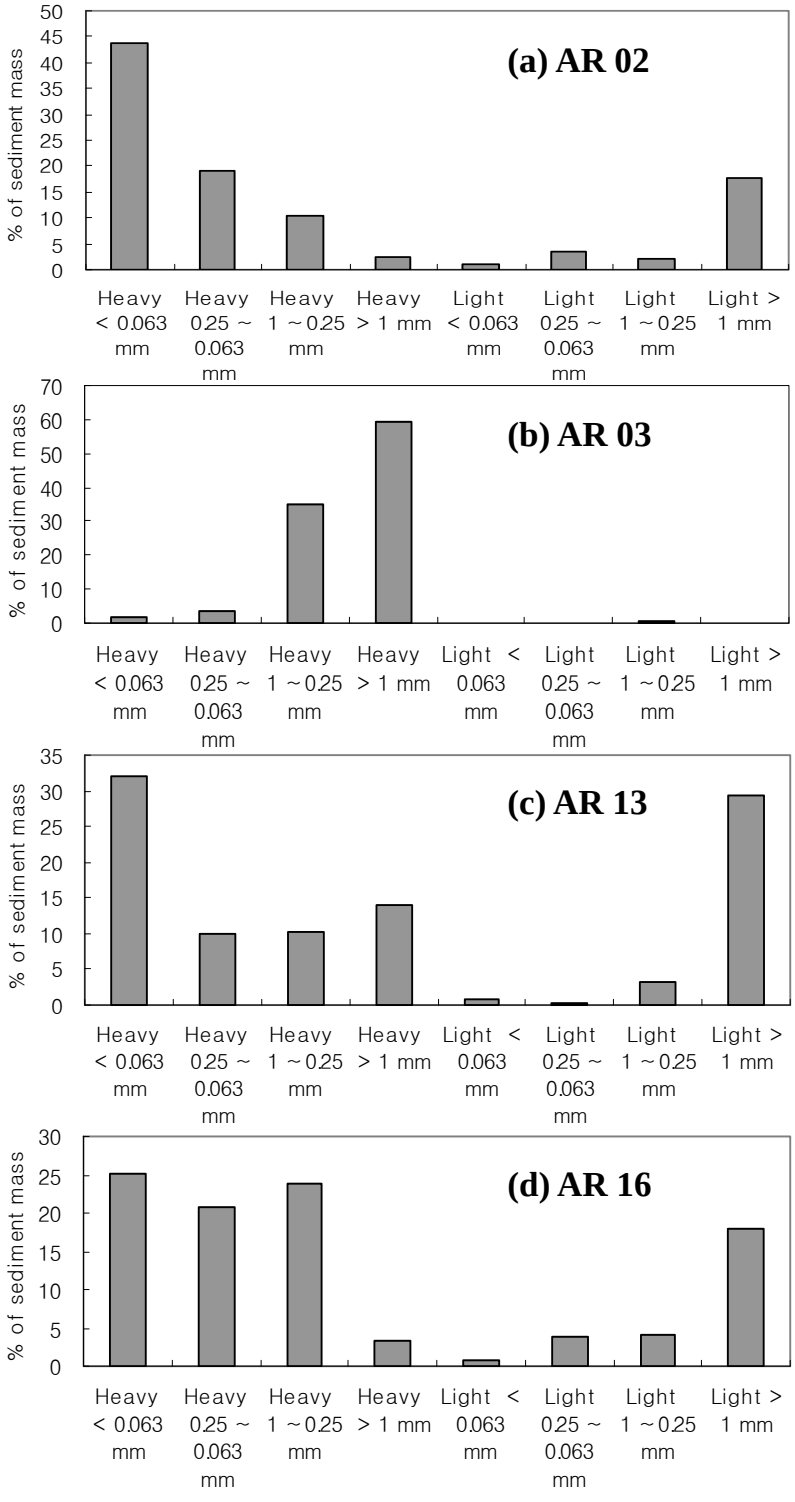


Figure 1. Mass distribution of sediments by size and density fractions

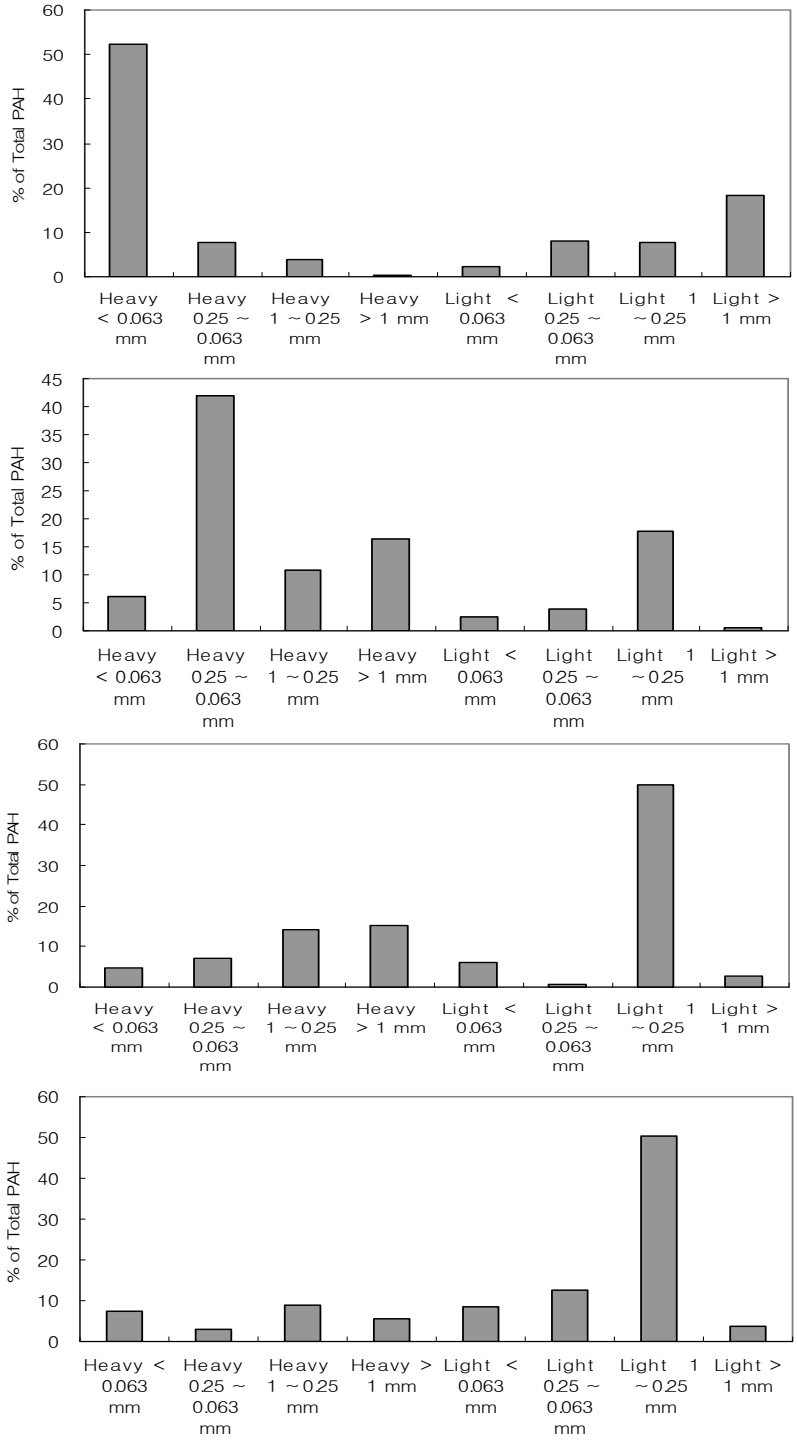


Figure 2. Total PAH distribution in sediments by size and density fractions

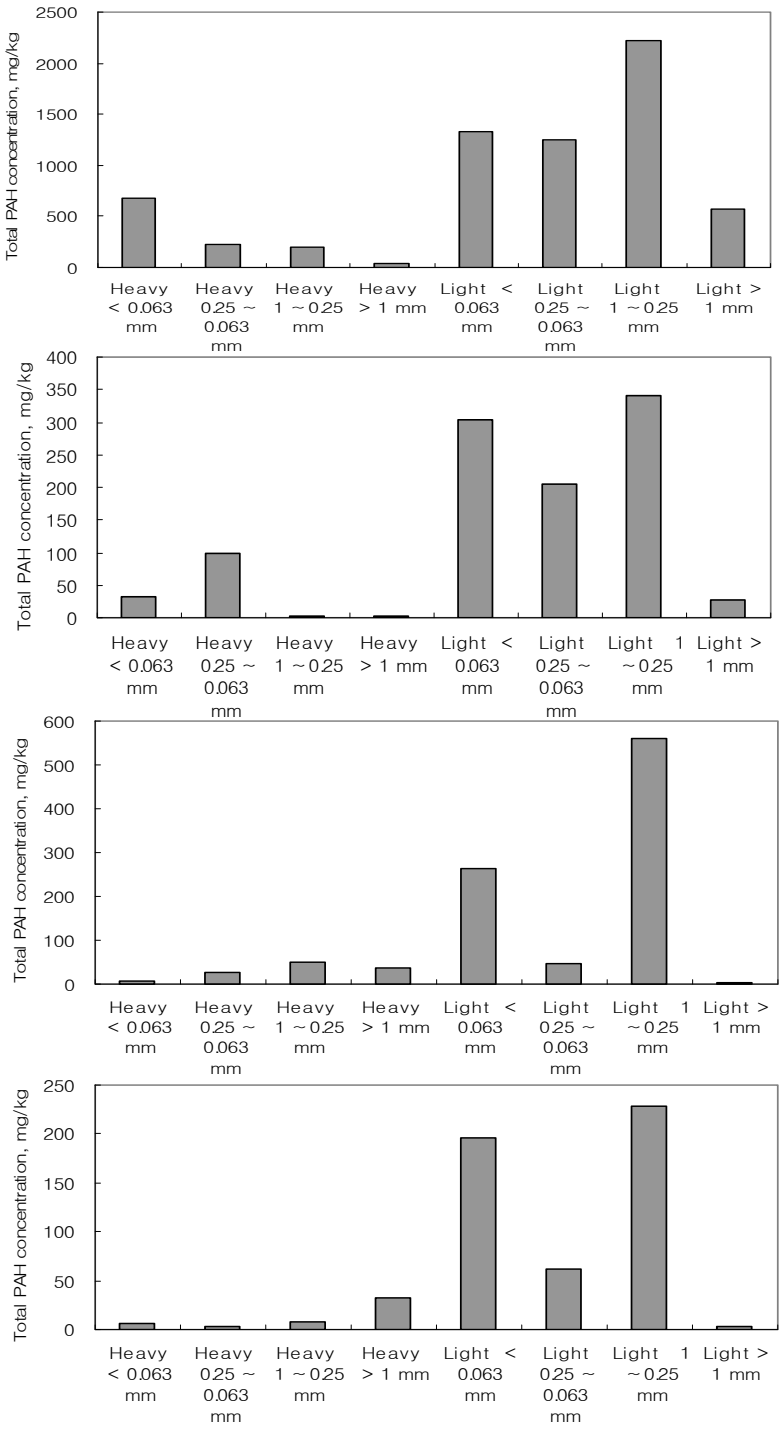


Figure 3. PAH concentration in sediment fractions by size and density

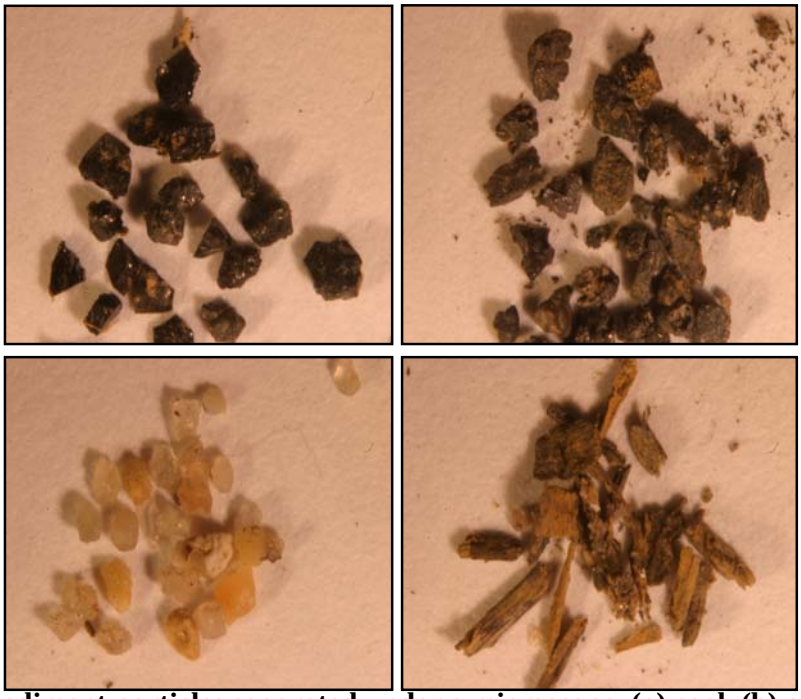


Figure 4. The sediment particles separated under a microscope, (a) coal, (b) coke, (c) sand and (d) wood: AR 02 at 250 – 1000 μm size fraction.

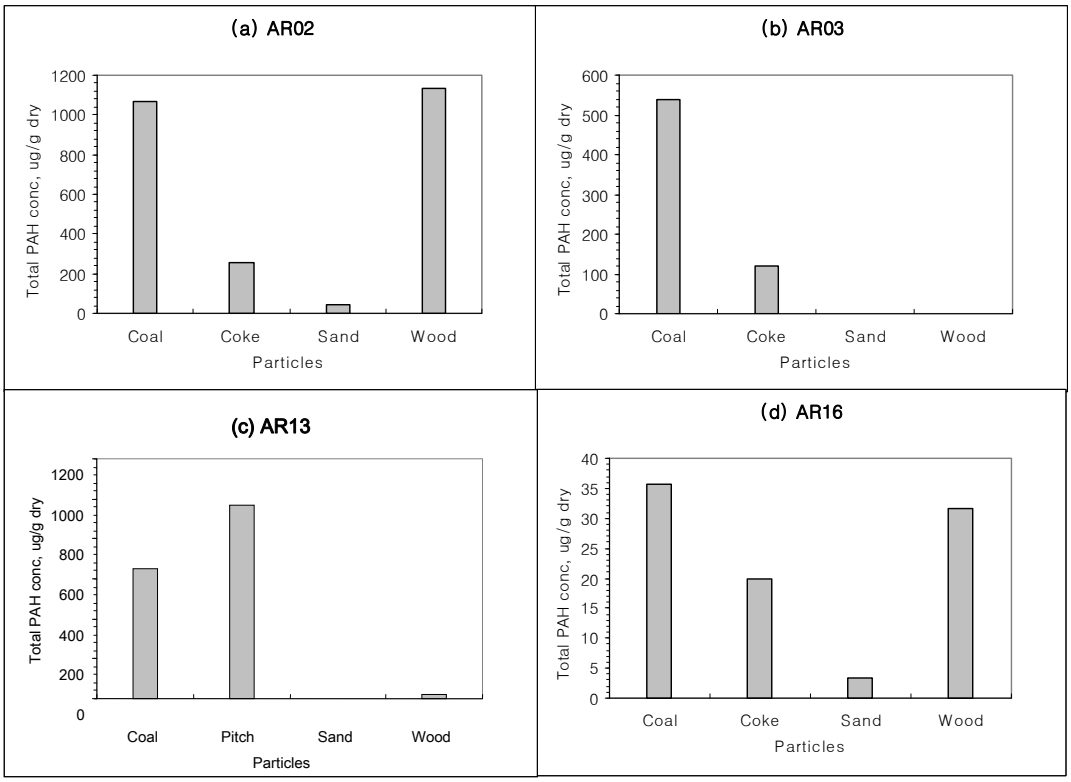


Figure 5. Microscale PAH concentration of the four dominant particle types: coal, coke, pitch, sand, and wood.

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APPENDIX L:

DETAILED PETROGRAPHY IMAGES OF 0.25-1.0 mm LIGHTER DENSITY SEDIMENT FRACTIONS

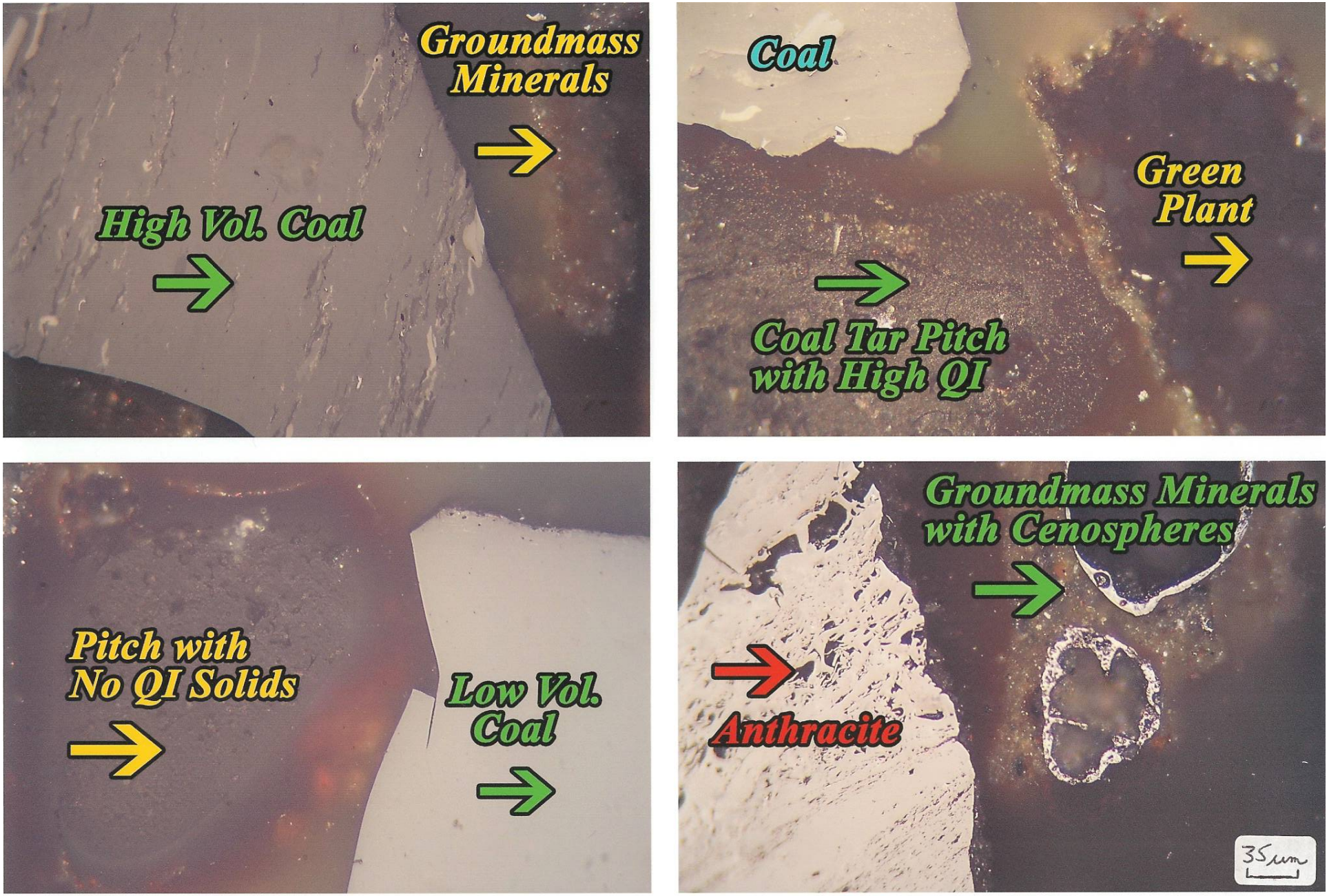


Figure A1 Photomicrographs of Materials in the UMBC Soil Sample AR-02 Showing: High and Low Volatile Bituminous Coal, Anthracite Coal, Coal Tar Pitch with Normal QI Solids, Pitch with No QI Solids, Groundmass Mineral Matter and Green Plant Material. Reflected Light in Oil, 400X.

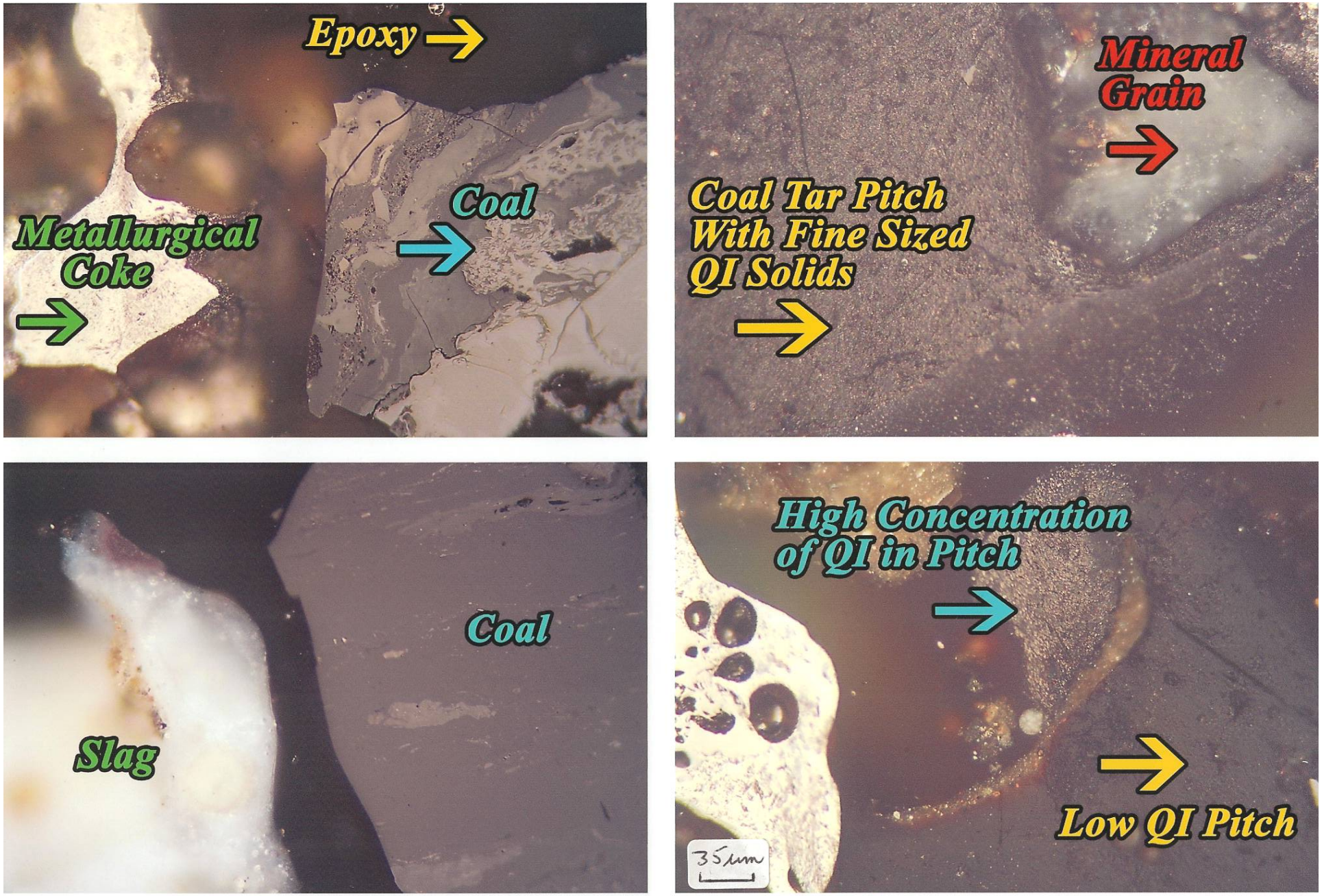


Figure A2 Photomicrographs of Materials in the UMBC Soil Sample AR-02 Showing: Bituminous Coal, Metallurgical Coke, Coal Tar Pitch with Normal QI Solids, Coal Tar Pitch with High and Low Content of QI Solids, Slag, Mineral Grain and Epoxy. Reflected Light in Oil, 400X.

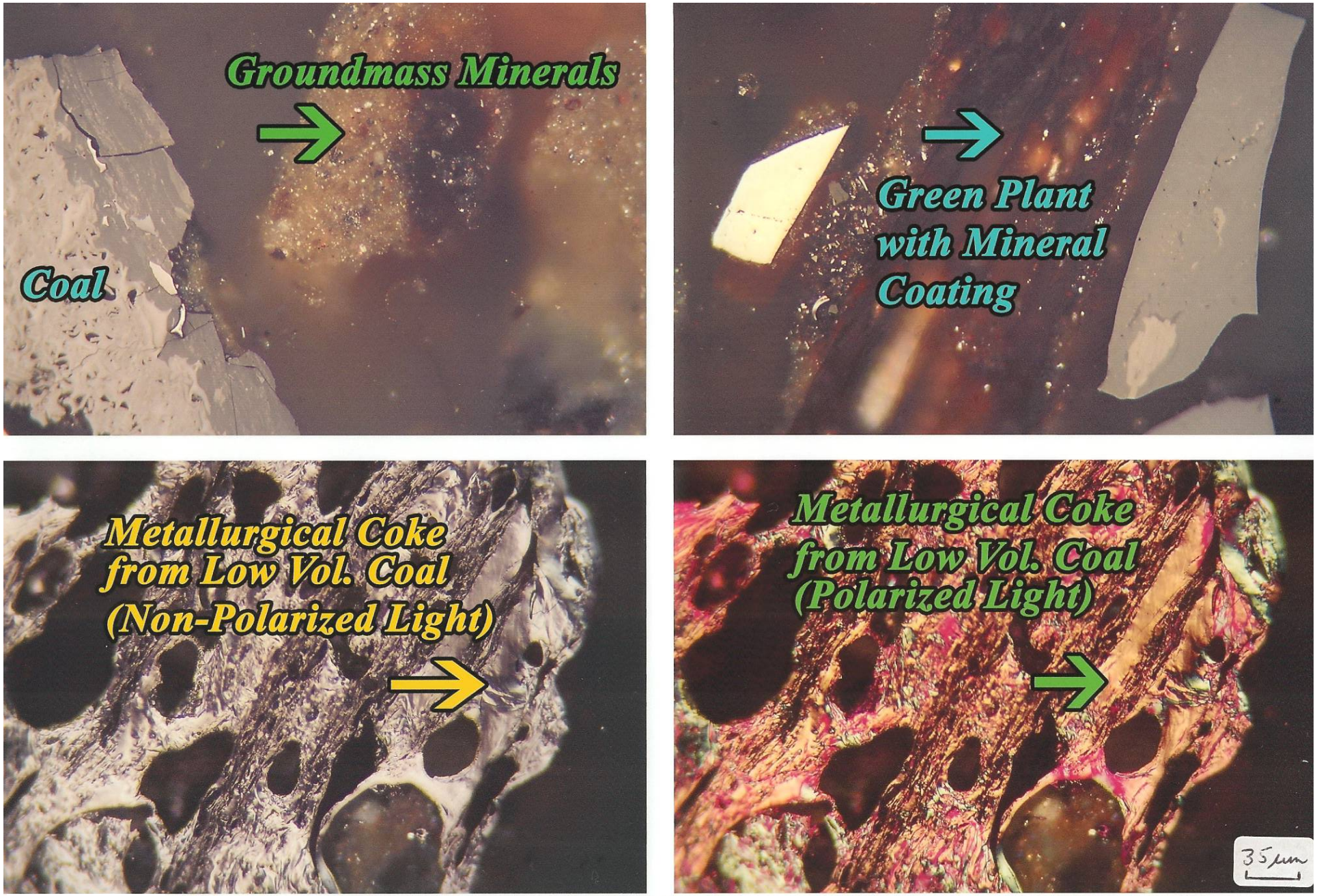


Figure A3 Photomicrographs of Materials in the UMBC Soil Sample AR-02 Showing: Bituminous Coal, Metallurgical Coke from Low Vol. Coal shown in Polarized and Non-Polarized Light, Groundmass Minerals and Green Plant Material with Groundmass Mineral Coating. Reflected Light in Oil, 400X.

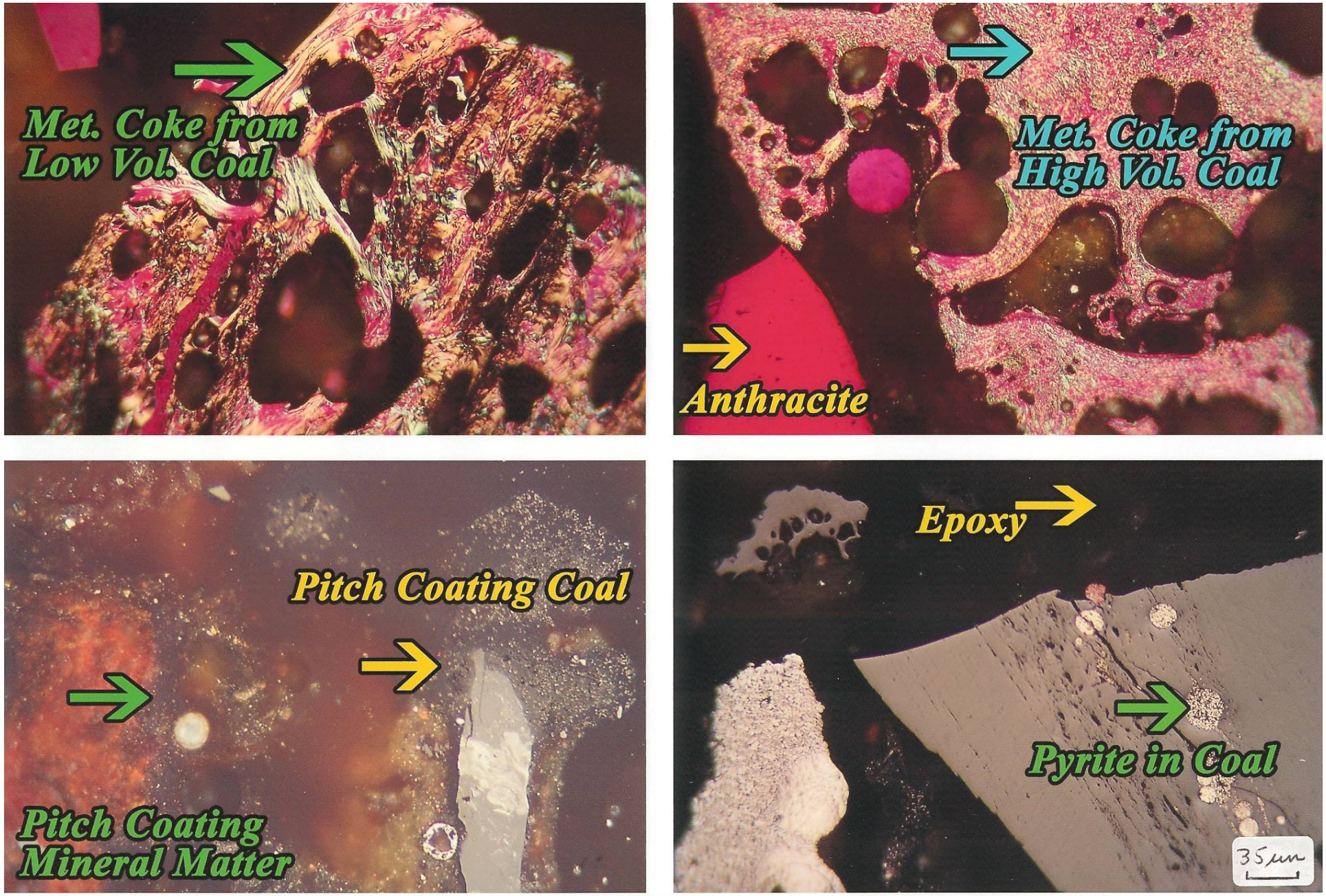


Figure A4 Photomicrographs of Materials in the UMBC Soil Sample AR-02 Showing: Pyrite in Bituminous Coal, Metallurgical Coke from High and Low Vol. Coal shown in Polarized Light, Anthracite, Pitch Coating Coal & Mineral Matter and Epoxy Mounting Media. Reflected Light in Oil, 400X.

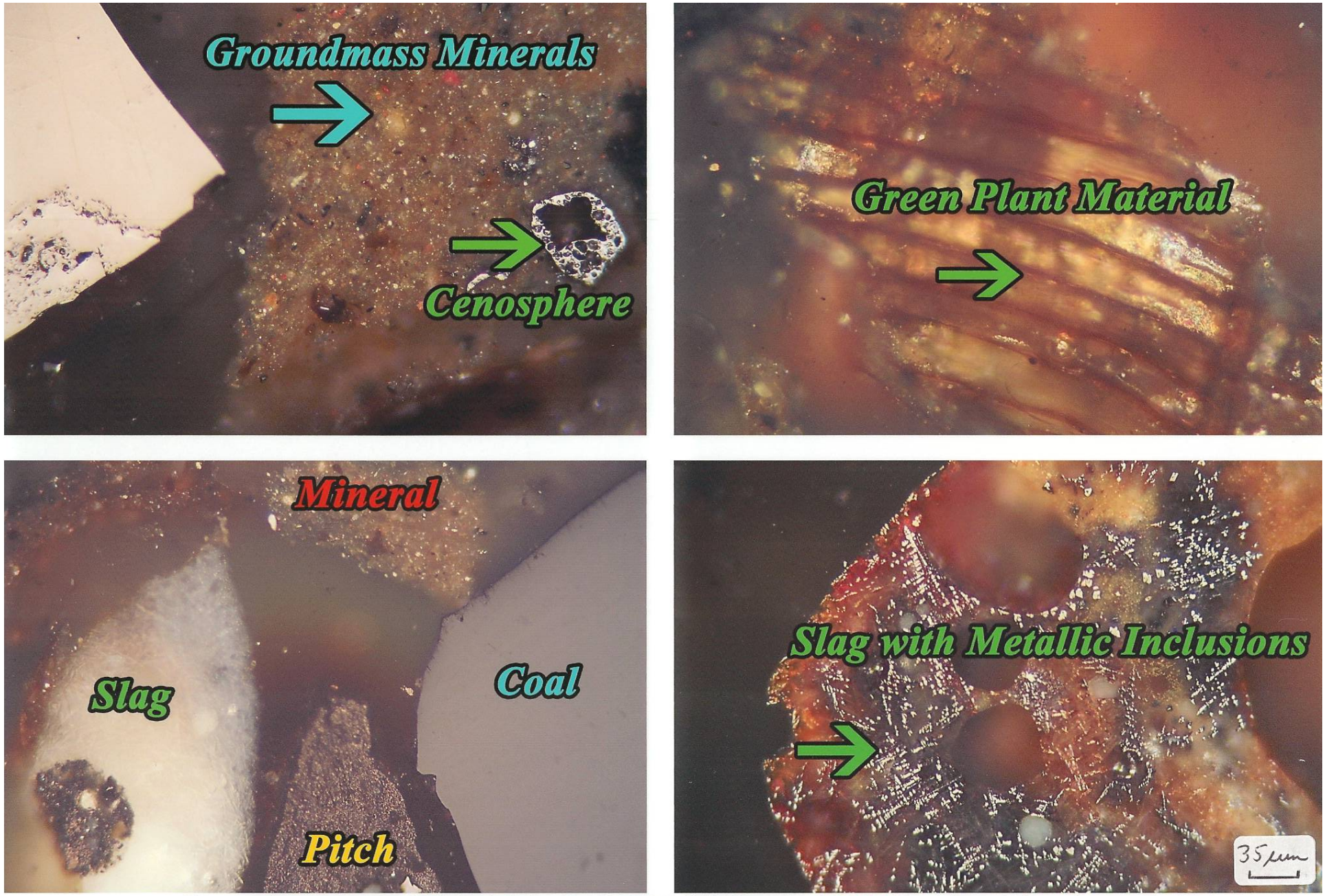


Figure A5 Photomicrographs of Materials in the UMBC Soil Sample AR-02 Showing: Bituminous Coal, Cenosphere, Coal Tar Pitch, Groundmass Minerals, Slag, Slag with Metallic Inclusions and Green Plant Material. Reflected Light in Oil, 400X.

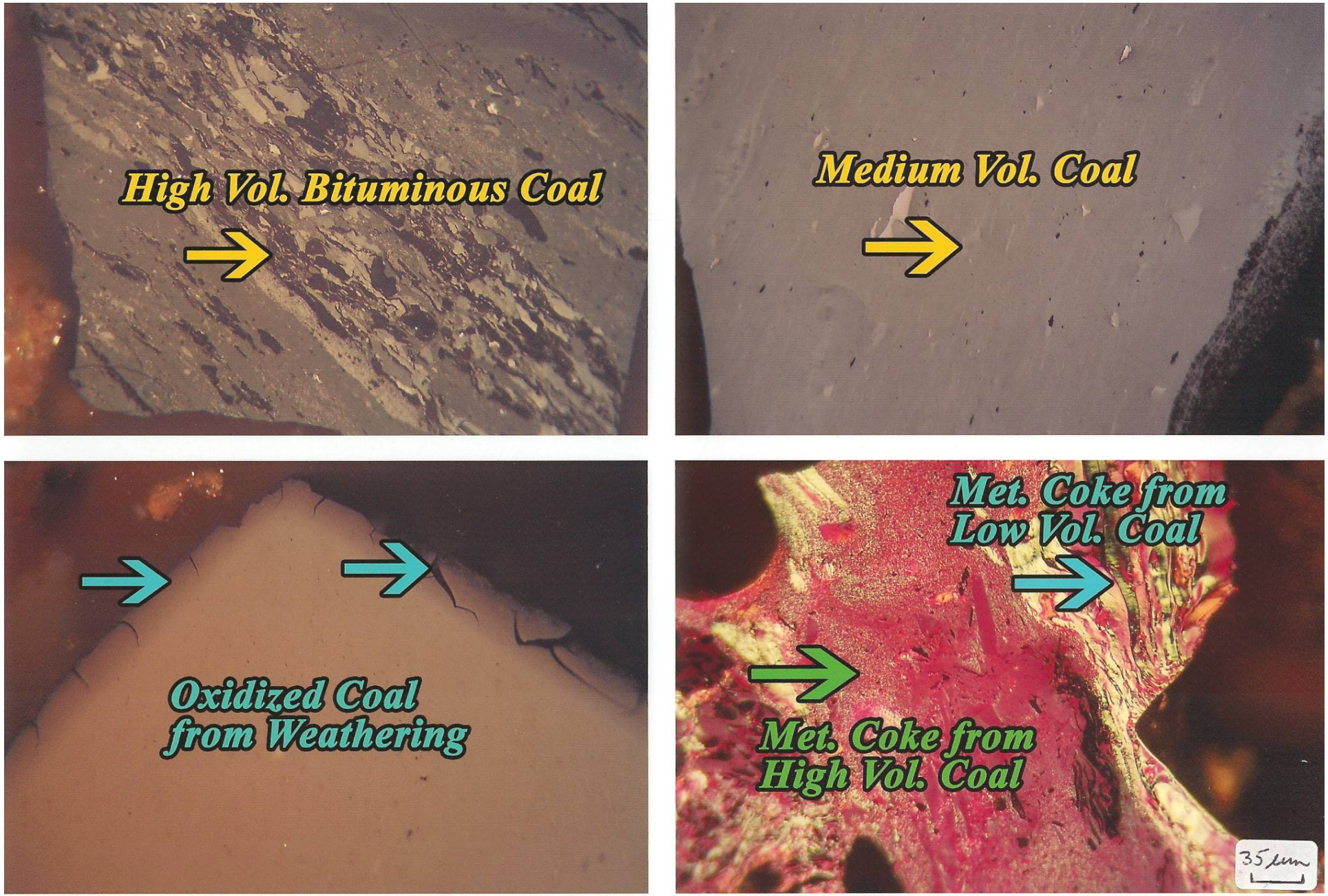


Figure A6 Photomicrographs of Materials in the UMBC Soil Sample AR-03 Showing: High & Medium Volatile Bituminous Coals, Oxidized Coal from Weathering and Metallurgical Coke from High and Low Volatile Coals. Reflected and Polarized Light in Oil, 400X.

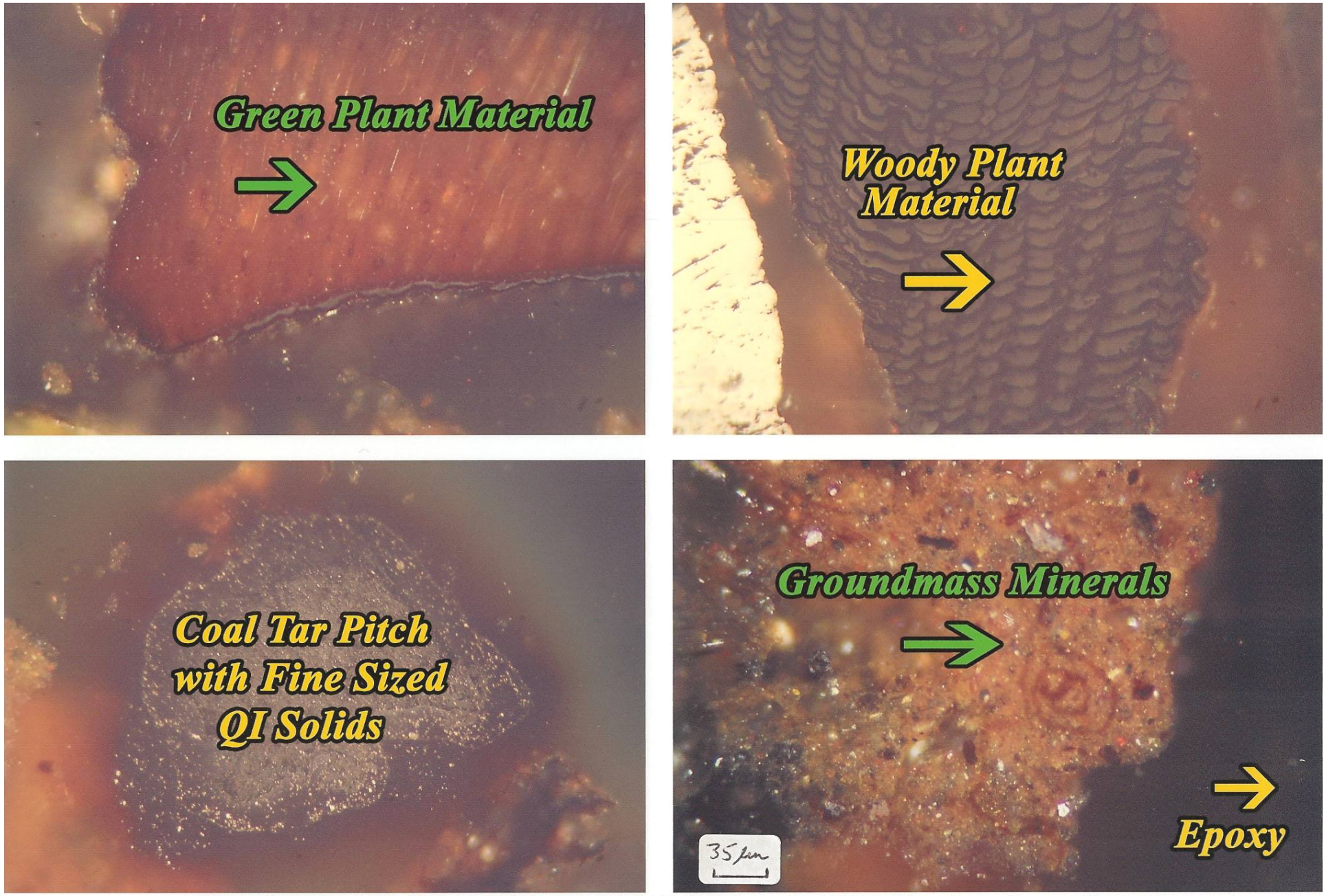


Figure A7 Photomicrographs of Materials in the UMBC Soil Sample AR-03 Showing: Green Plant Material, Woody Plant Material, Coal Tar Pitch with Normal QI, Groundmass Minerals and Epoxy Mounting Media. Reflected Light in Oil, 400X.

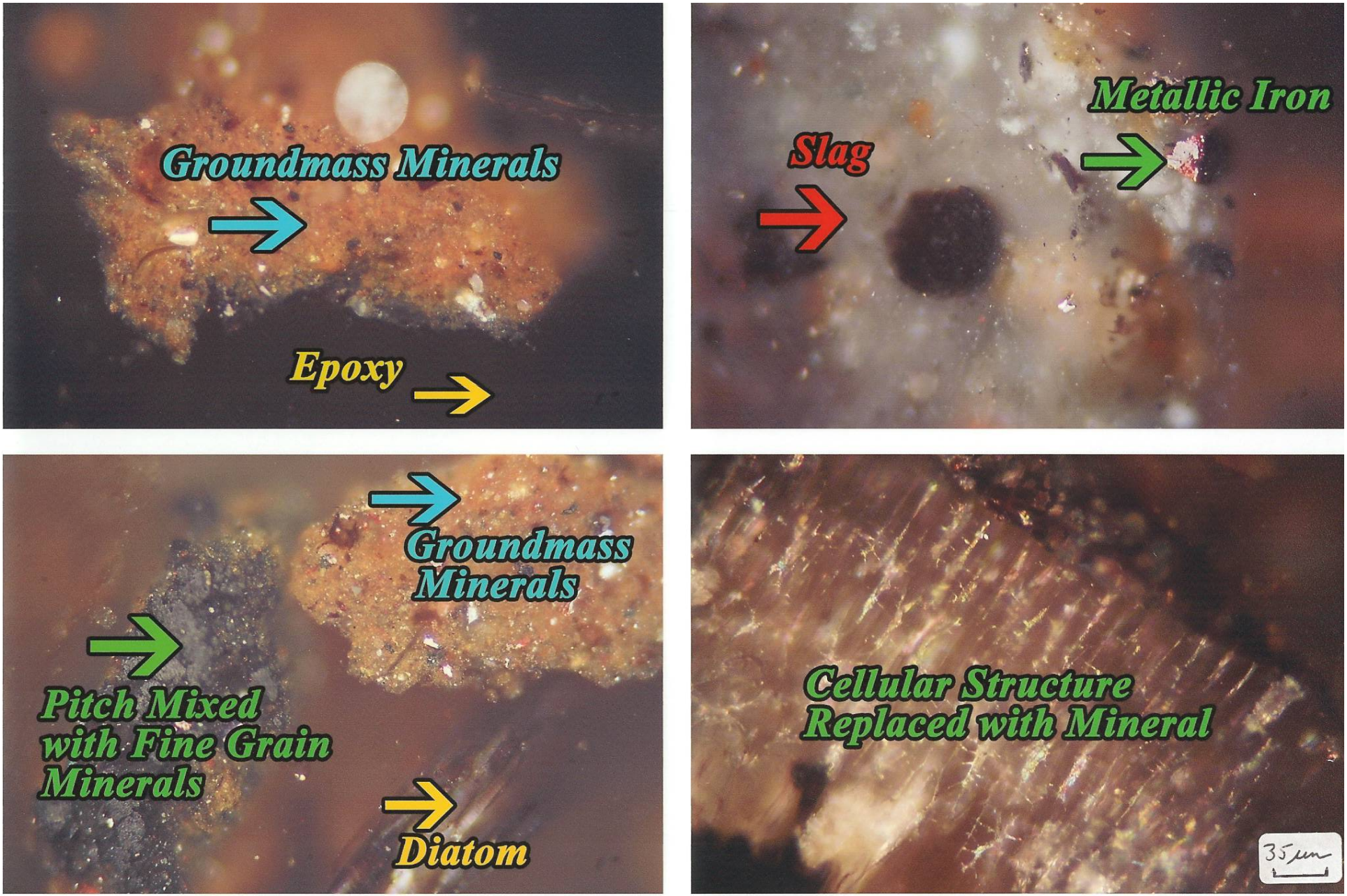


Figure A8 Photomicrographs of Materials in the UMBC Soil Sample AR-03 Showing: Coal Tar Pitch Mixed with Fine Grain Minerals, Groundmass Minerals, Slag, Metallic Iron, Diatom, Cellular Structure Replaced with Mineral and Epoxy Mounting Media. Reflected Light in Oil, 400X.

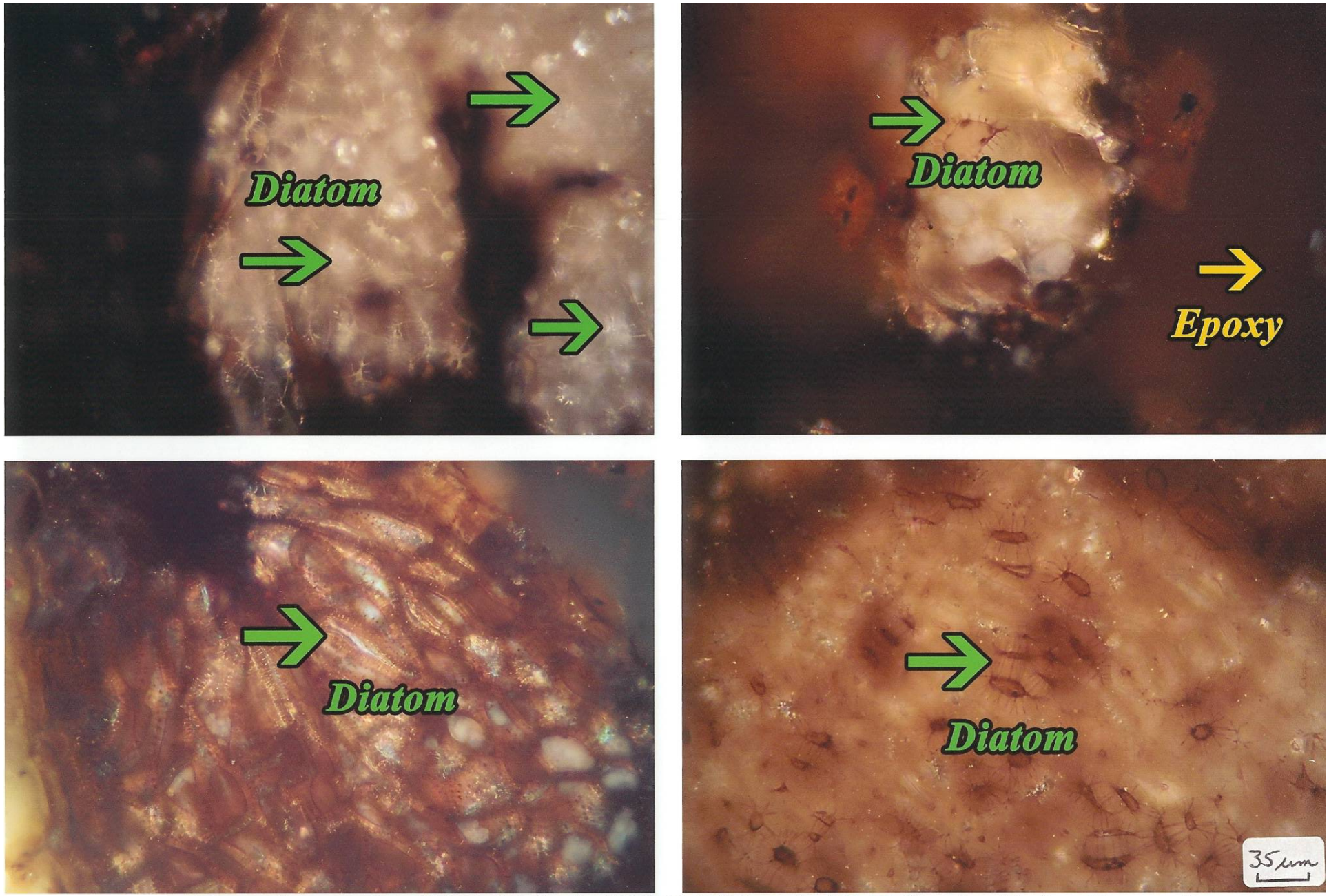


Figure A9 Photomicrographs of Materials in the UMBC Soil Sample AR-03 Showing Various Forms of Diatomaceous Earth and Epoxy Mounting Media. Reflected Light in Oil, 400X.

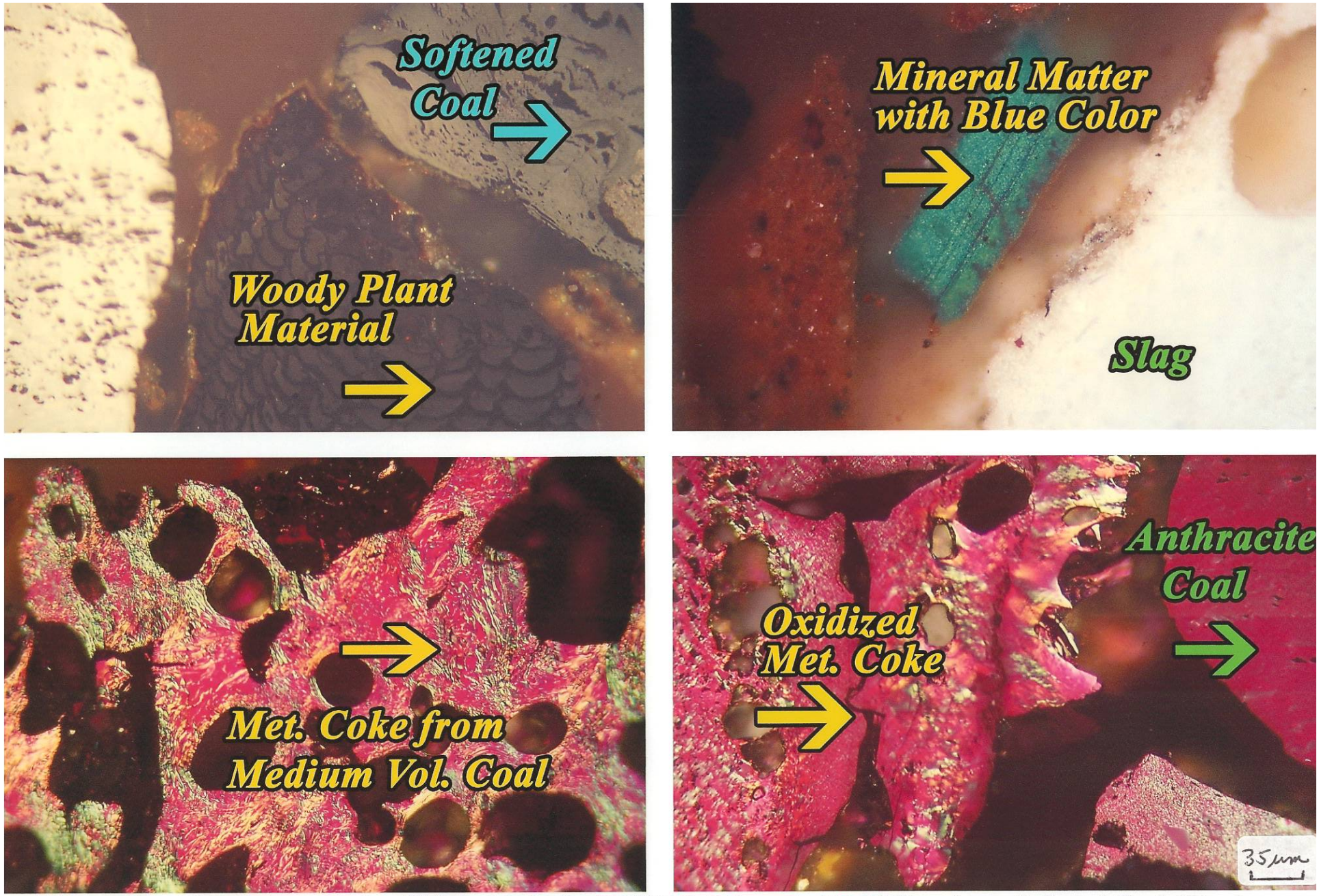


Figure A10 Photomicrographs of Materials in the UMBC Soil Sample AR-03 Showing: Softened Coal, Woody Plant Material, Slag, Blue Mineral Matter, Anthracite Coal, Metallurgical Coke from Medium Vol. Coal and Oxidized Metallurguical Coke. Reflected Light in Oil, 400X.

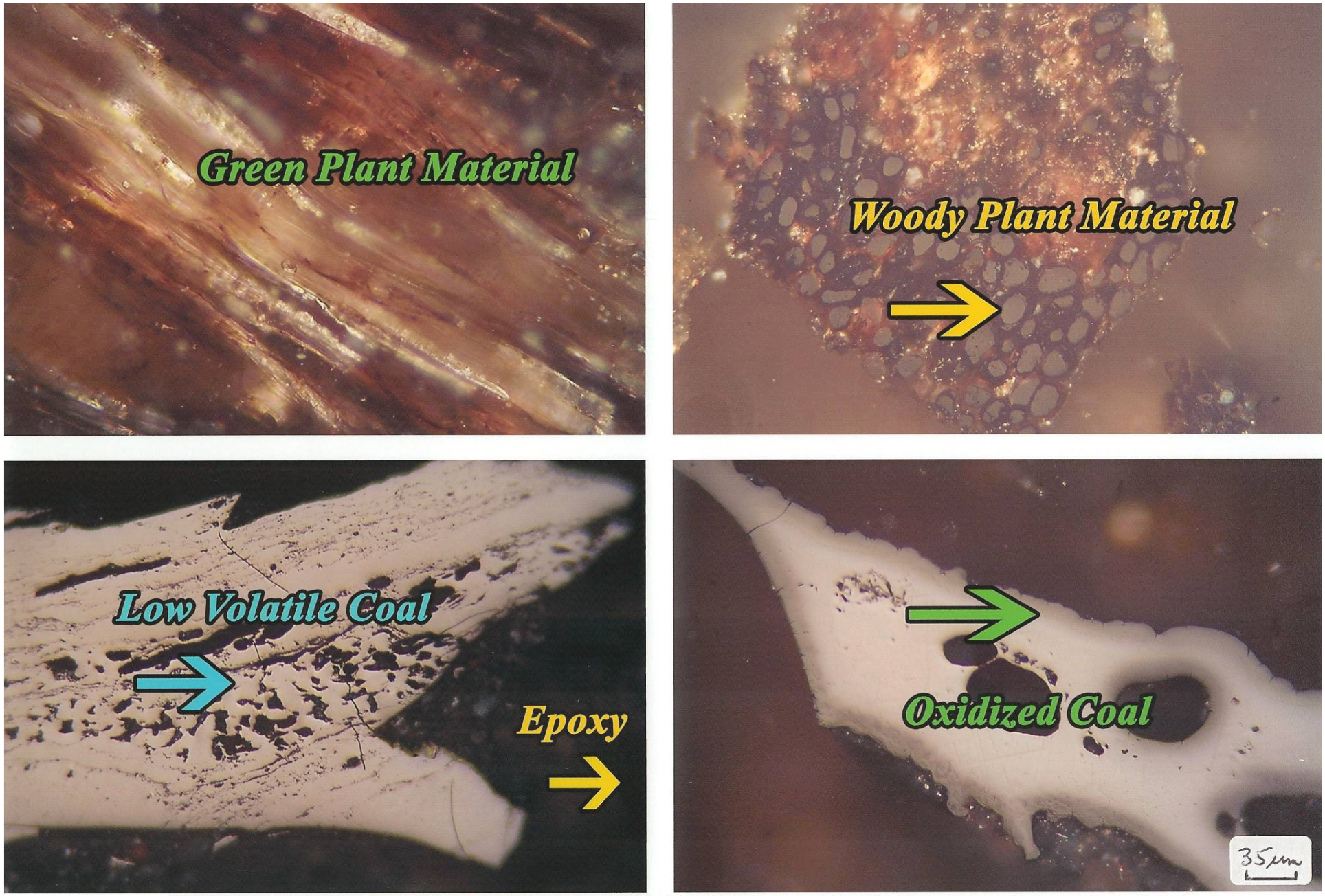


Figure A11 Photomicrographs of Materials in the UMBC Soil Sample AR-13 Showing: Green Plant Material, Woody Plant Material, Low Volatile Bituminous Coal, Thermally Oxidized Coal and Epoxy Mounting Media. Reflected Light in Oil, 400X.

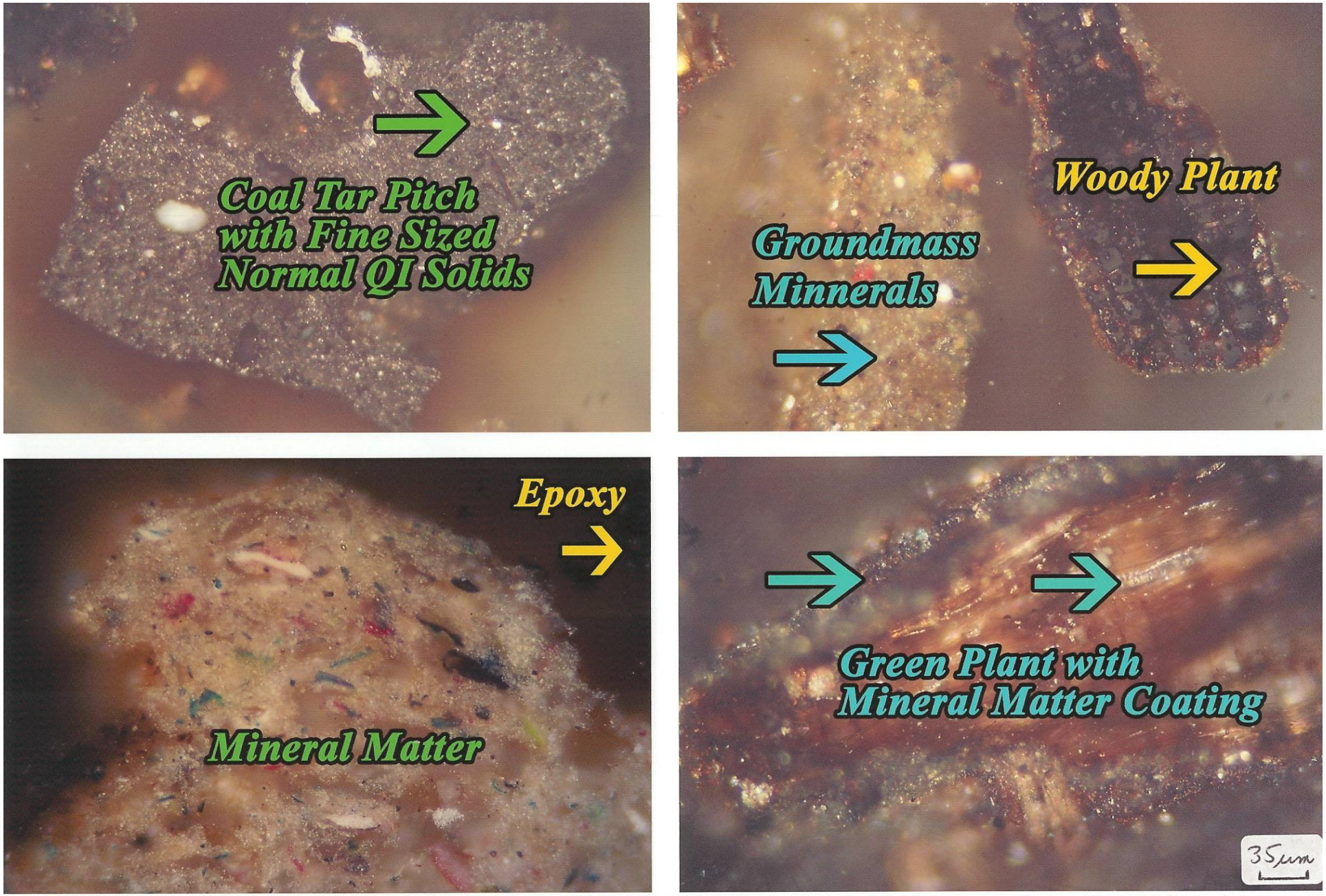


Figure A12 Photomicrographs of Materials in the UMBC Soil Sample AR-13 Showing: Green Plant Material, Woody Plant Material, Coal Tar Pitch with Fine Sized Normal QI Solids, Groundmass Minerals, Mineral Matter with Different Colored Minerals and Epoxy. Reflected Light in Oil, 400X.

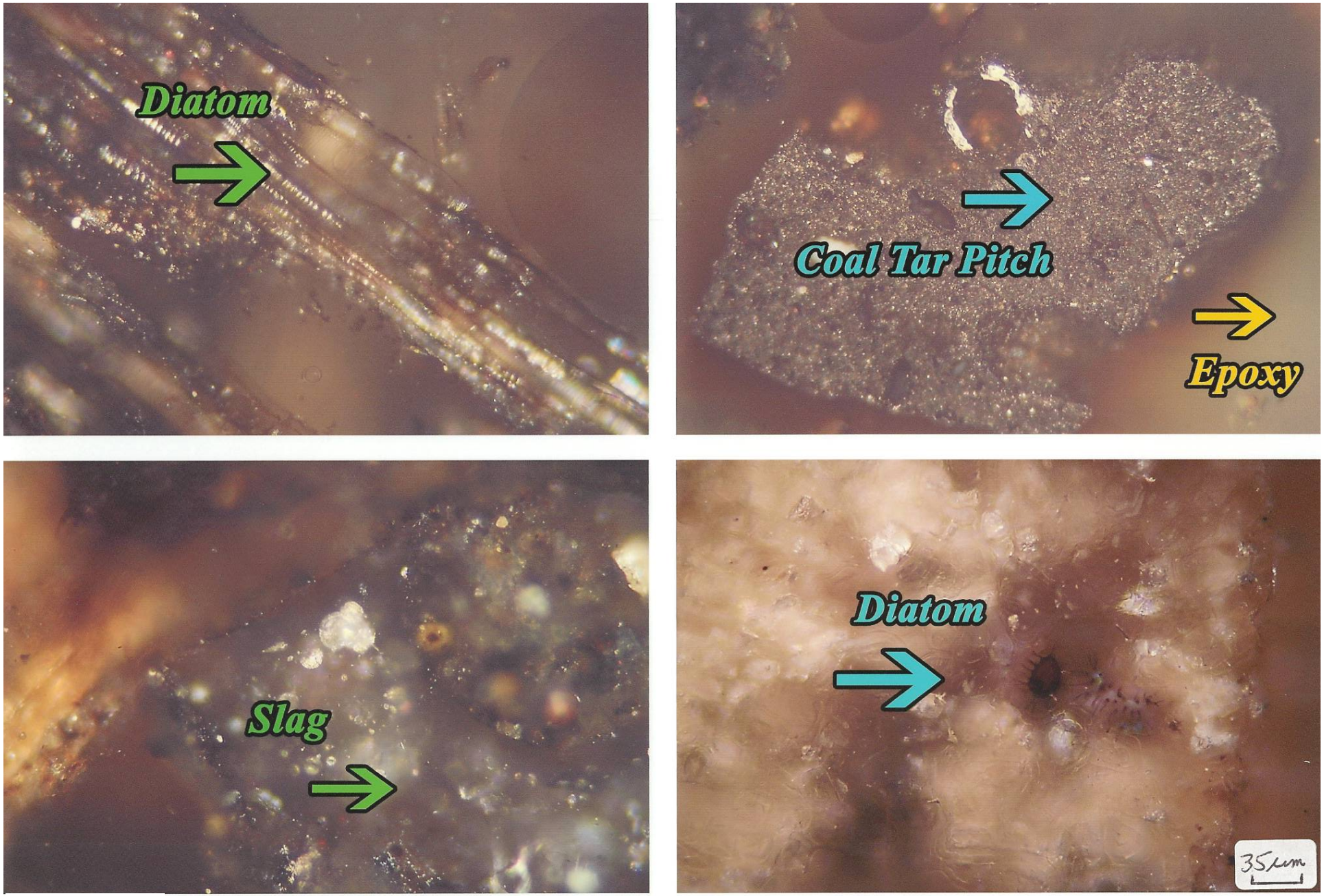


Figure A13 Photomicrographs of Materials in the UMBC Soil Sample AR-13 Showing: Coal Tar Pitch with Fine Sized Normal QI Solids, Slag, Diatomaceous Earth and Epoxy. Reflected Light in Oil, 400X.

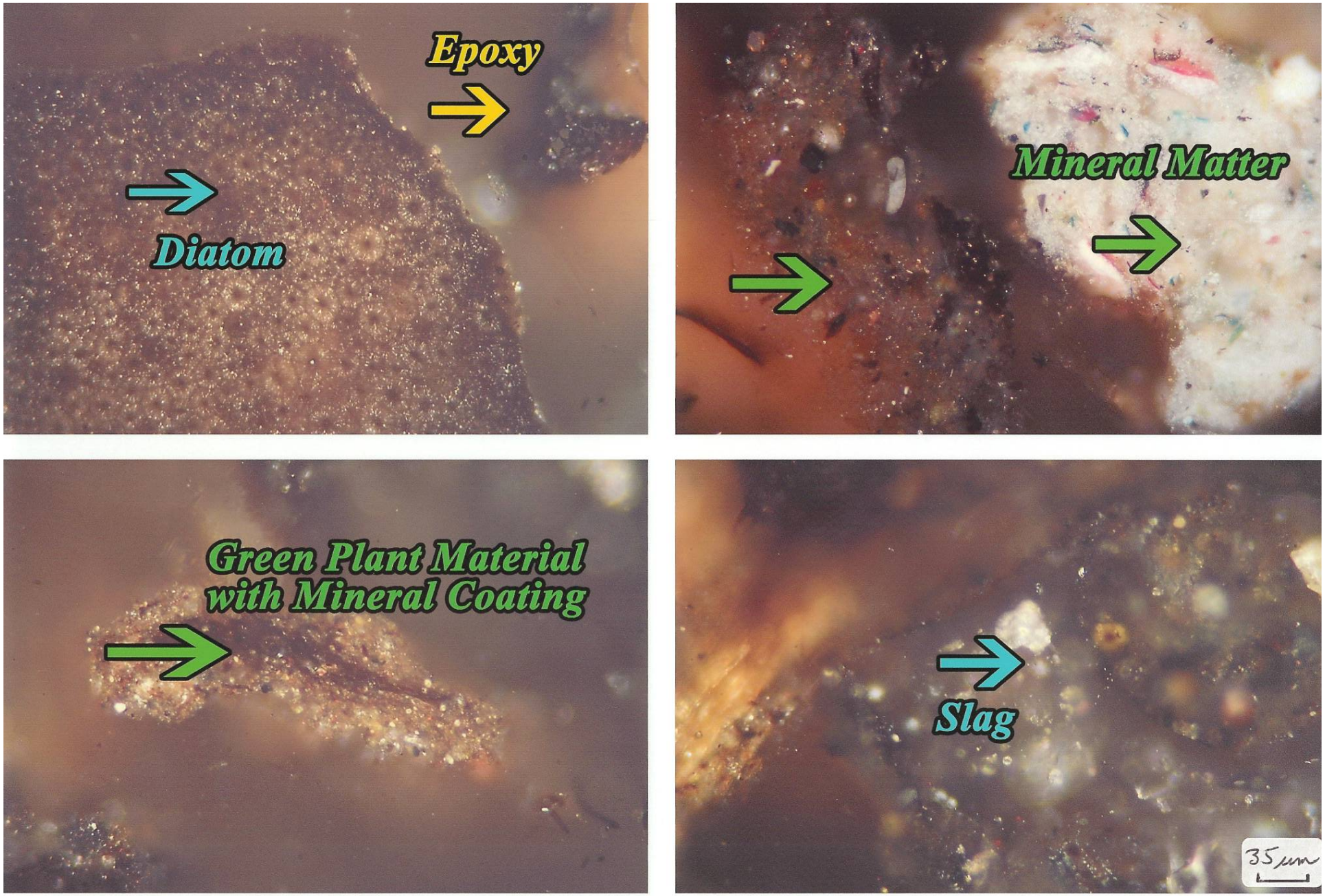


Figure A14 Photomicrographs of Materials in the UMBC Soil Sample AR-13 Showing: Green Plant Material with Mineral Coating, Slag, Diatom, Other Mineral Matter and Epoxy Mounting Media. Reflected Light in Oil, 400X.

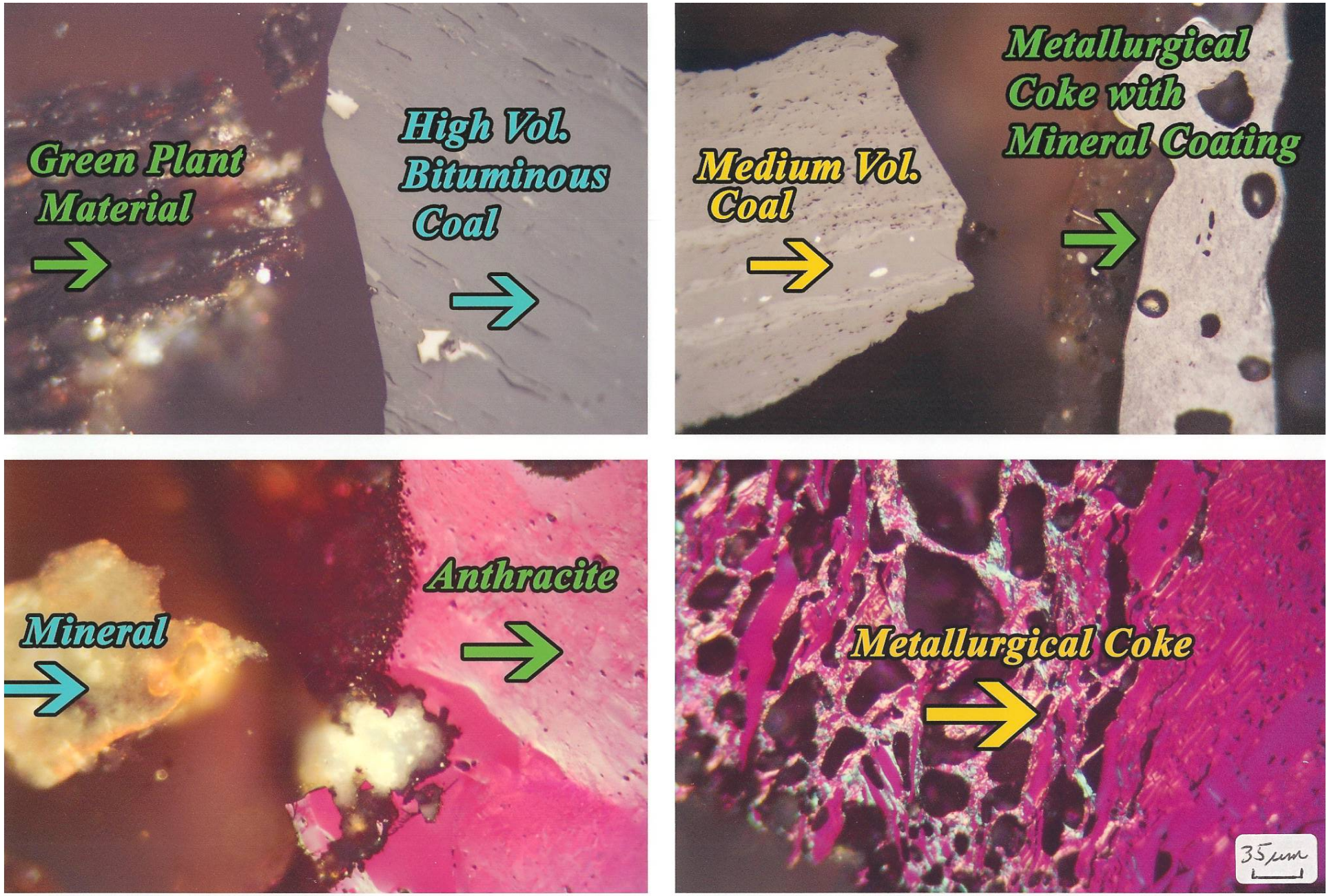


Figure A15 Photomicrographs of Materials in the UMBC Soil Sample AR-16 Showing: High and Medium Volatile Bituminous Coal, Anthracite Coal, Metallurgical Coke, Green Plant Material, Mineral Matter Coating and Other Mineral Matter. Reflected Light in Oil, 400X.

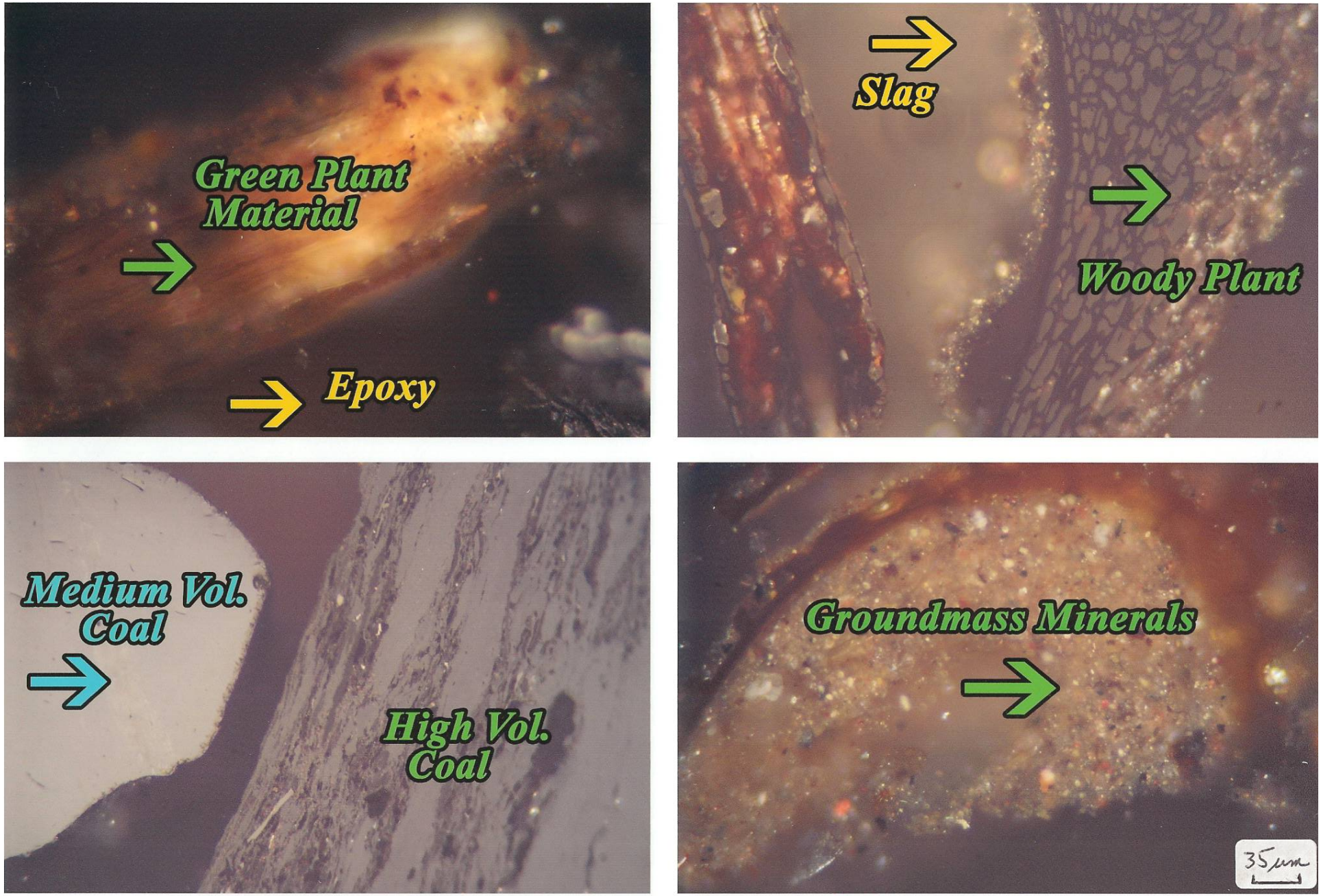


Figure A16 Photomicrographs of Materials in the UMBC Soil Sample AR-16 Showing: High and Medium Volatile Bituminous Coal, Green Plant Material, Woody Plant Material, Groundmass Minerals, Slag and Epoxy Mounting Media. Reflected Light in Oil, 400X.

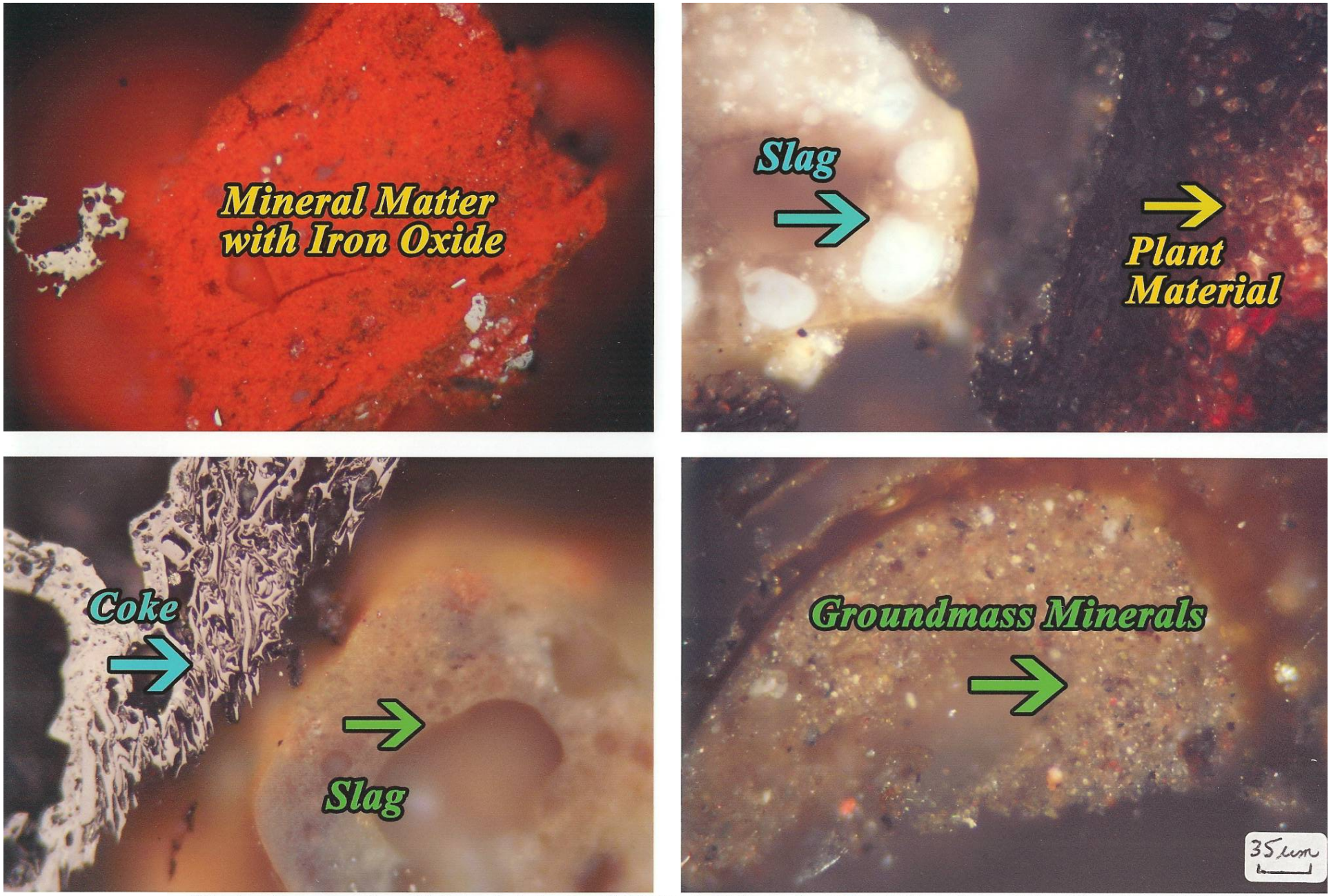


Figure A17 Photomicrographs of Materials in the UMBC Soil Sample AR-16 Showing: Metallurgical Coke, Plant Material, Groundmass Minerals, Slag and Mineral Matter Mixed with Iron Oxide. Reflected Light in Oil, 400X.

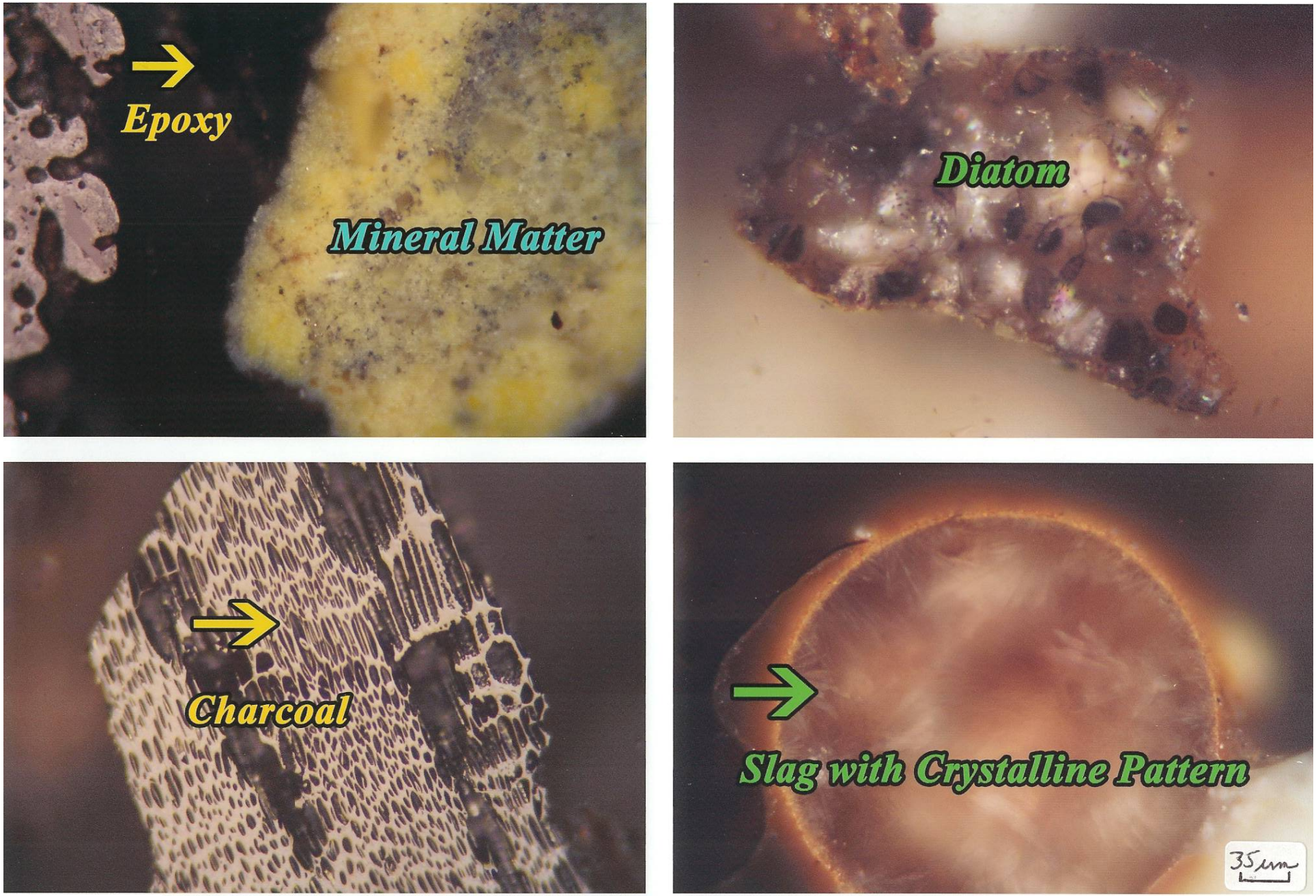


Figure A18 Photomicrographs of Materials in the UMBC Soil Sample AR-16 Showing: Charcoal, Mineral Matter, Slag with Crystalline Pattern, Diatom and Epoxy Mounting Media. Reflected Light in Oil, 400X.