QUALITY ASSURANCE PROJECT PLAN (QAPP)

for

Assessment of Toxicity of Upper Columbia River Sediments to Benthic Invertebrates

RECEIVED
By Kris McCaig at 5:47 pm, Nov 04, 2013

Submitted by:

Jeffery A. Steevens and Jacob K. Stanley

U.S. Army Engineer Research and Development Center (ERDC)

Environmental Laboratory

3909 Halls Ferry Rd.

Vicksburg, MS 39056

(601) 634-3544

September 20, 2013

SECTION A – PROJECT MANAGEMENT

A.1 – QUALITY ASSURANCE PROJECT PLAN APPROVAL PAGE

This document has been prepared in accordance with the USEPA (2001a) Requirements for Quality Assurance Project Plans, USEPA QA/R-5, for use by the USEPA, or its designated representatives. The following individuals have reviewed the QAPP and find that the procedures outlined in this document will result in data that can be used for evaluating the chemistry and toxicity of sediments collected in the Upper Columbia River (UCR), Washington (i.e., for confirmatory toxicity testing of sediment samples collected and evaluated by Teck American Incorporated (TAI) at the UCR site).

U.S. Army ERDC Senior Scientist, Jeffery A. Steevens — 9/24/13 Date

U.S. Army ERDC Project Lead, Jacob K. Stanley — 9-24-13 Date

U.S. Army ERDC Co-principal Investigator, Daniel Farrar — 9-24-13 Date

U.S. Army ERDC, Environmental Toxicology — 9-25-13 Date
Research Facility Laboratory Manager, QA Officer, James Biedenbach

USEPA Project Manager, Laura Buelow — 9.25.13 Date

USEPA Project Manager, R. Matthew Wilkening — 9/25/13 Date

USEPA Quality Assurance Manager, Ginna Grepo-Grove — 10/22/13 Date

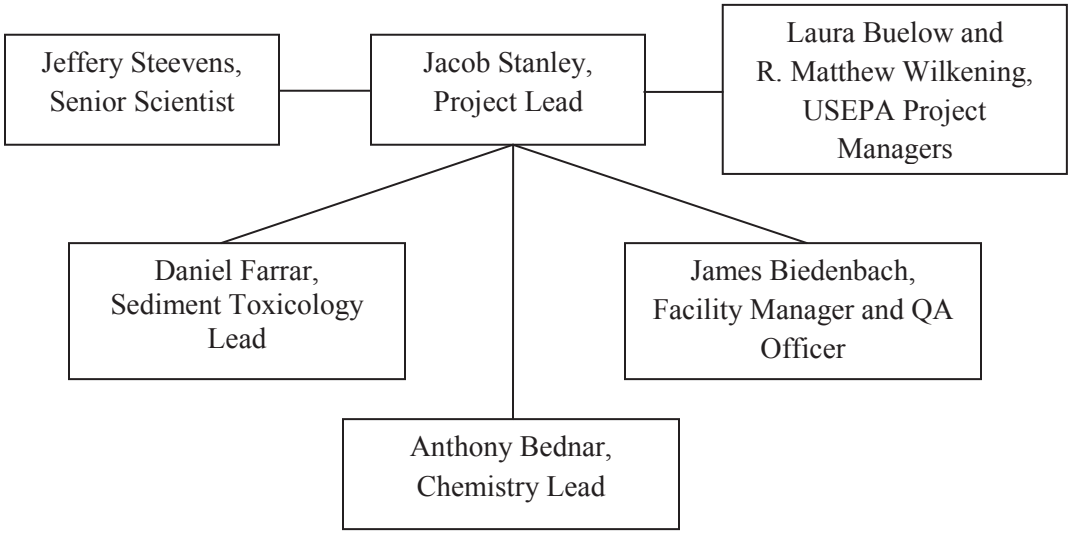
A.2 – TABLE OF CONTENTS

A.3 – DISTRIBUTION LIST

The U.S. Army ERDC Project Manager will be responsible for distribution of this QAPP. The following individuals will receive copies of this QAPP and any subsequent revisions:

Jeffery A. Steevens, U.S. ARMY ERDC
Jacob K. Stanley, U.S. ARMY ERDC
Daniel Farrar, U.S. ARMY ERDC
James Biedenbach, U.S. ARMY ERDC
Anthony J. Bednar, U.S. ARMY ERDC
Laura Buelow, USEPA Project Manager
R. Matthew Wilkening, USEPA Project Manager
Ginna Grepo-Grove, USEPA Quality Assurance Manager
Kris McCaig, Teck American Incorporated

A.4 – PROJECT/TASK ORGANIZATION



Jeffery Steevens, U.S. Army ERDC Senior Scientist	Dr. Jeffery Steevens is a U.S. Army Senior Scientist. He has over 20 years experience in sediment ecotoxicology. Provides overall direction of the project for ERDC. Ensures that all tasks are accomplished in a timely manner and within the project budget. Has direct contact with Co-principal investigators, the ERDC Quality Assurance (QA) officer, and USEPA.
Jacob Stanley, U.S. Army ERDC Project Lead	Dr. Jacob Stanley is a Research Biologist and team leader of the Ecotoxicology and Environmental Risk Team at the U.S. Army ERDC. He has over 14 years of aquatic ecotoxicology experience and 6 years of sediment ecotoxicology experience. He is a member of the Society of Environmental Toxicology and Chemistry (SETAC) *Hyalella azteca* Advisory Group (HAAG), experienced at *H. azteca* culturing, and has published four peer-reviewed journal articles on *H. azteca* research. He is also experienced at both

	short and long-term (life cycle) testing using *Chironomus dilutus*. Coordinates laboratory toxicity testing activities identified in Section B. Works with ERDC Project Manager, QA Officer, and Co-principal investigator to coordinate schedule and toxicity testing.
Daniel Farrar, U.S. Army ERDC Sediment Toxicology Lead	Mr. Daniel Farrar, M.S. is a Research Biologist with the Ecotoxicology and Environmental Risk Team at the U.S. Army ERDC. He has over 20 years of sediment ecotoxicology experience. He is a member of the Society of Environmental Toxicology and Chemistry (SETAC) *Hyalella azteca* Advisory Group (HAAG) and is experienced at ***H. azteca*** and ***C. dilutus*** culturing. He is also experienced at both short and long-term (life cycle) testing using *Chironomus dilutus*. Is responsible for toxicity testing laboratory facilities and equipment and providing direction to staff. Ensures that all testing records are complete and reviewed for completeness and accuracy.
James Biedenbach, U.S. Army ERDC Environmental Toxicology Research Facility Laboratory Manger, QA Officer	Mr. James Biedenbach, B.S.; M.B.A. is a Biologist with the Ecotoxicology and Environmental Risk Team at the U.S. Army ERDC. He has over 22 yrs of experience with sediment and porewater toxicity testing. Provides QA support to include resolving QA problems, document review, and data validation. Provides oversight to verify that project activities are being conducted in a manner consistent with requirements identified in this QAPP.
Anthony Bednar, U.S. Army ERDC	Dr. Anthony Bednar has over 15 years

Chemistry Lead Environmental Chemistry Branch	experience in low-level metals analysis and speciation, as well as method development and optimization for complex matrix interferences. Manage, direct, and provide analytical chemistry needs for the project. This includes providing in-house analysis as well as subcontracting for analyses. He will be assisted by Dr. Kristie Armstrong who has over 5 years experience with metals analysis in complex matrices. Both Drs. Bednar and Armstrong will closely monitor more junior analysts performing on this project. Ms. Charolett Hayes will perform ion chromatography analyses and has over 20 years experience with laboratory and field analysis techniques in support of ERDC and external research efforts. Mr. Andrew Bray will perform AVS, alkalinity and pH analyses. He has over 2 years experience with the ERDC laboratory and has been directly trained by Dr. Bednar and the instrument manufacturer on the analyses he is responsible for. The remaining analyses required for the project are not performed in-house by the ERDC-EP-C laboratory and therefore will be subcontracted to our NELAC BPA commercial laboratories. All data generated will be imported, stored, and reported from our LIMS by Ms. Patricia Tuminello, Mr. Michael Catt, and Ms. Charolett Hayes. They have over 8

	years experience in data reporting operations of the EP-C and customer data formatting. Ms. Tuminello is a Stanford Certified Project Manager.
Laura Buelow, USEPA Project Manager R. Matthew Wilkening, USEPA Project Manager	Has overall responsibility of project. Works closely with ERDC to provide samples, sample information, and ensure that project tasks are being met.

A.5 – PROBLEM DEFINITION/BACKGROUND

U.S. Army ERDC was contacted in February 2013 and asked to provide sediment ecotoxicology support to U.S. EPA as part of the Upper Columbia River Sediment Study. For background on the UCR site, see the Upper Columbia River Final Quality Assurance Project Plan for the Phase 2 Sediment Study (E^{x}ponent and HDR|HydroQual 2013). Briefly, TAI is conducting sediment sampling at 140 sampling stations to support the Phase 2 Sediment Sampling Study. A subset of the sediment samples collected (74 samples) will be submitted to Pacific EcoRisk (PER) for performance of the following:

- 28-day whole-sediment toxicity tests with the amphipod, *H. azteca* (endpoints of survival, weight, and biomass [USEPA 2000; ASTM 2012])

- 10-day whole-sediment toxicity tests with the midge, *C. dilutus* (endpoint of survival, weight, and biomass [USEPA 2000; ASTM 2012]).

Reproductive endpoints will be assessed by PER on a subset of 18 of these 74 samples using the following specific bioassays:

- 42-day whole-sediment toxicity tests with the amphipod, *H. azteca* (endpoints of survival, weight, biomass, and neonates/surviving female [USEPA 2000; ASTM 2012])

- 50- to 65-day whole-sediment toxicity tests with the midge, *C. dilutus* (endpoints of survival, weight, biomass, emergence, eggs/surviving female, egg hatching, and viability of young using 4 d old larvae [USEPA 2000; ASTM 2012]).

In order to support an analysis of the reliability and usability of the toxicity data generated by PER intended for risk management decisions at the UCR site, the U.S. Army ERDC will conduct confirmatory toxicity testing with splits of up to 10 of the 74

sediment samples that have been selected for short-term toxicity testing using the 28-day whole-sediment toxicity test with *H. azteca* and the 10-day whole sediment toxicity test with *C. dilutus*. These 10 samples tested by US Army ERDC will be made up of up to seven split samples from bioassay stations located upstream from the confluence of Onion Creek (RM 730) and, pending approval and agreement from the Canadian Government for EPA collection, up to three split-samples for bioassay testing from upstream reference locations. As part of the toxicity tests conducted, U.S. Army ERDC will also provide chemical analysis of sediments and porewater. Porewater will be obtained through two methods; direct centrifugation of the sediment and "peepers" placed into the sediment in the exposure chambers to passively sample the porewater and constituents. Optional chronic toxicity tests using *H. azteca* (42-day) and *C. dilutus* (50-65-day) will be conducted based on the results of the short-term toxicity test (10-d *C. dilutus* and 28-d *H. azteca*) and chemistry results.

This QAPP outlines the methods that will be used to conduct the confirmatory toxicity tests and sampling of porewater metals with splits of sediment samples to evaluate the toxicity of UCR sediments.

A.6 – TASK DESCRIPTIONS

The tasks to be conducted include:

- Toxicity testing of up to 10 sediment samples (short-term toxicity tests and optional long-term toxicity tests)
- Sampling of porewater chemistry (ions, metals, TOC) for the purpose of modeling toxicity associated with the presence of metals
- Analytical chemistry of whole sediments and porewater
- Chronic toxicity testing of samples (number TBD) (optional)
- Compilation and reporting of toxicity and analytical chemistry results

A.6.1 – Toxicity Testing of Sediments

Toxicity testing will be performed on up to 10 sediment samples using the following toxicity tests performed in basic accordance with standard methods:

- 28-day short-term whole-sediment toxicity tests with the amphipod, *H. azteca* (endpoints of survival, weight, and biomass [USEPA 2000; ASTM 2012])

- 10-day short-term whole-sediment toxicity tests with the midge, *C. dilutus* (endpoints of
  survival, weight, and biomass [USEPA 2000; ASTM 2012]).

- 42-day long-term whole-sediment toxicity tests with the amphipod, *H. azteca* (endpoints of
  survival, weight, and biomass [USEPA 2000; ASTM 2012]) (optional).

- 50 to 65-day long-term whole-sediment toxicity tests with the midge, *C. dilutus* (endpoints of survival, weight, and biomass [USEPA 2000; ASTM 2012]) (optional).

A.6.2 – Sampling of Porewater Metals

Porewater metals will be sampled by centrifugation of sediment and by using peeper passive sampling devices from an extra chemistry replicate beaker set up with each treatment that also contains sediment and organisms.

A.6.3 – Compilation and Reporting of Toxicity Results

Toxicity test results from the *H. azteca* and *C. dilutus* bioassays will be summarized in a spreadsheet (Microsoft Excel format) and also provided in a written report (Microsoft Word format) and submitted to the USEPA. Data will also be provided in an electronic deliverable that will be formatted through coordination with USEPA and TAI.

A.6.4 – Analytical Chemistry

The analytical laboratory will be the ERDC Environmental Chemistry Branch, while the bioassay laboratory will be the ERDC Environmental Risk Assessment Branch. Each organization has extensive experience with analysis of complex environmental and biological samples. Standard analytical techniques will be used where applicable, although method modifications will be incorporated where needed to provide the highest quality analytical results from low-volume and complex matrix samples.

A.7 – QUALITY OBJECTIVES AND PERFORMANCE CRITERIA FOR MEASUREMENT DATA

Toxicity test performance will be determined by the ability to meet test acceptability criteria given in standard methods (ASTM 2012; USEPA 2000). For the *H. azteca* 28-day whole-sediment toxicity test, the criterion is as follows:

- Minimum Day 28 mean control survival of 80%

For the *C. dilutus* 10-day whole-sediment toxicity test the criteria are as follows:

- Minimum mean control survival must be 70%.
- Minimum mean weight per surviving control organism of 0.48 mg ash-free dry weight (AFDW).

Additional general performance-based criteria specifications and performance criteria for the long-term tests are outlined in ASTM (2012) and in USEPA (2000). The performance-based criteria used will be the same as those given in the Upper Columbia River Final Quality Assurance Project Plan for the Phase 2 Sediment Study (E^{x}ponent and HDR|HydroQual 2013).

A.8 – SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

All individuals who will be participating in toxicity testing have been appropriately trained in the toxicity tests to be performed and in the use and care of *H. azteca* and *C. dilutus* and will be following established standard operating procedures and standard methods. Thus, no additional specific training is required to complete project objectives.

All individuals who will be participating in analytical chemistry testing have been appropriately trained and have extensive experience in chemical analysis of complex matrices, including tissues and analysis of low-volume samples. Standard methods will be following except where method modifications have been developed to specifically address interferences encountered from low volume samples or complex biological matrices. Dilution as an interference reduction technique is considered the last resort for collection of analytical data. No additional specific training is required to complete project objectives.

A.9 – DOCUMENTS AND RECORDS

A.9.1 – Quality Assurance Project Plan (QAPP)

This QAPP and any associated revisions/updates will be distributed in electronic format by the U.S. Army ERDC Project Manager via e-mail to all people listed in the distribution list in Section A.3.

A.9.2 – Laboratory Documentation

A full report on all laboratory activities and results will be provided by the U.S. Army ERDC Project Manager to the USEPA Project Manager. This report will consist of the following:

- Sample receipt and testing initiation and completion dates
- Description of toxicity testing methods
- Documentation of the performance of organisms in response to reference toxicants
- The weight of a representative subsample of organisms at the start of sediment exposures will be documented
- Scanned copies of all laboratory data sheets and laboratory notes
- Summary of all toxicity and water quality results
- An evaluation of whether or not test acceptability criteria were met for each toxicity test performed
- Description of any problems encountered and corrective actions taken
- Description of any deviations from prescribed laboratory protocols

An electronic spreadsheet (Microsoft Excel format) summarizing all toxicity data will also be supplied to the USEPA Project Manager.

A.9.3 – Data Archiving

Electronic and paper copies of the laboratory report and associated data sheets and data summaries will be maintained at U.S. Army ERDC for a period of at least seven years. Electronic files, communication, records and other information will be maintained on the Army Records Information Management System (ARIMS) or on external hard drive where necessary. All analytical data produced will be stored in the U.S. Army ERDC Environmental Chemistry Branch LIMS for a period of at least seven years.

SECTION B – MEASUREMENT/DATA ACQUISITION

B.1 – Sampling Design Process (Experimental Design)

The sampling design is described in the Upper Columbia River Draft Final Quality Assurance Project Plan (E^{x}ponent and HDR|HydroQual 2013). To confirm the results and determine the reliability of sediment testing by PER, the ERDC will perform sediment toxicity testing on up to 10 split sediment samples collected by TAI and tested for sediment toxicity by the PER. Control sediments consisting of quartz sand, PER performance control sediment and a performance control sediment provided by the ERDC will also be evaluated. Chemistry replicates will be included in testing for determining pore water dissolved metal concentrations. All chemical analyses will include method specific QA/QC samples. Information obtained from sediment toxicity testing of the split samples at the ERDC will be used to determine the reliability and usability of the data generated by PER.

B.2 – Sampling Methods

Procedures for collection of sediment samples and rational for sample selection are described in the TAI study wide QAPP (E^{x}ponent and HDR|HydroQual 2013).

ERDC Sediment Volume Requirements

- *C. dilutus* 10-d short-term test – 2.75 L
- *H. azteca* 28-d short-term test - 3.5 L
- *C. dilutus* 50-d long-term test – 6.25 L (optional)
- *H. azteca* 42-d long-term test – 4.5 L (optional)
- Porewater chemistry – 2.0 L
- Sediment chemistry – 3.0 L

Total – 22 L (~5 gallons)

B.3 – Sample Handling and Custody Requirements

Chain of custody forms will be used to document the transfer of sediment samples from Columbia Analytical Services (CAS) and subsequent transportation of sediment samples to the ERDC. All COC forms related to the transfer of sediments to and from the ERDC will be maintained and archived by the ERDC. ***Samples should be shipped to: US Army Engineer Research and Development Center, 3909 Halls Ferry Road, Vicksburg, MS 39180; Attn: Jacob Stanley; Telephone: 601-634-3544***

Upon transfer of samples to the Environmental Chemistry Branch laboratory, receiving personnel will check the physical integrity of the containers and custody seals (if any), and samples will be inventoried by comparing sample labels to those on the COC forms. The laboratory will include the COC and shipping container receipt forms in the data package. Any breaks in the COC or non-conformances will be noted and reported. All samples will be stored in accordance with standard specifications, including keeping samples at 4°C, in the dark, in a secure restricted access facility. ERDC will not dispose of the samples for at least 6 months after the finalization of the data report generated.

B.4 – Analytical Methods

B.4.1 – Whole Sediment Toxicity Testing

B.4.1.1 – *Chironomus dilutus* 10-Day Short-term Test

Short-term sediment toxicity testing (10-d) with the midge, *Chironomus dilutus*, will be conducted in basic accordance with procedures outlined in ASTM (2012) and USEPA (2000). A performance control provided by ERDC consisting of a sediment collected from the University of Mississippi Field Station, Abbeville, MS (total organic carbon about 1.5%) along with a quartz sand control sediment and a performance controls sediment provided by PER will also be evaluated. Up to 10 field-collected sediments will be evaluated using the 10-day short-term midge toxicity test.

Midge exposures will be conducted in 300-ml beakers containing 175 ml of overlying water and 100 ml of sediment. To allow for sediment equilibration, sediment and overlying water will be added to exposure beakers the day before the start of the exposures at 23°C under static conditions. Overlying water renewal will be initiated at day 0 prior to the addition of organisms. The source of overlying water for all midge toxicity tests will be moderately hard reconstituted water created using methods specified in USEPA (2000).

Eleven (11) replicate toxicity beakers will be tested for each sediment (8 for biological endpoints and 3 for pore water chemistry analysis at day 7. Full-spectrum fluorescent lights providing approximately 100 to 1000 lux (at the sediment interface) and a photoperiod of 16 h light and 8 h darkness will be provided. Approximately 10-d old organisms (2^{nd} to 3^{rd} instar; approximately 50% second instar and 50% third instar) will be used to initiate the exposures with a goal to achieve a starting average dry weight of about 0.12 mg/organism. The ash-free dry weight of a representative sample of organisms at the start of the sediment exposures will be documented. Egg masses will be obtained from Environmental Consulting and Testing, (Superior, WI) and allowed to hatch at the ERDC. A total of 10 midge larvae will be added each beaker. A suspension

of Tetramin® fish food (1.5 ml containing 6 mg of Tetramin®) will be provided to each beaker daily. At the end of the sediment toxicity tests, sediment will be passed through a 425 µm sieve and surviving organisms collected and enumerated for determination of survival, weight (AFDW), and biomass. Standard bioassay conditions and other required performance criteria for the above referenced four tests can be found in Appendix A.

B.4.1.2 - *Chironomus dilutus* 50-64-Day Chronic Test (Optional)

Chronic sediment toxicity testing (50 to 64-d) with the midge, *Chironomus dilutus*, will be conducted in basic accordance with procedures outlined in ASTM (2012) and USEPA (2000). A performance control provided by ERDC consisting of a sediment collected from the University of Mississippi Field Station, Abbeville, MS (total organic carbon about 1.5%) along with a quartz sand control sediment and a performance controls sediment provided by PER will also be evaluated. A to be determined number of field-collected sediments will be evaluated using the 50-64-day midge toxicity test.

Midge exposures will be conducted in 300-ml beakers containing 175 ml of overlying water and 100 ml of sediment. To allow for sediment equilibration, sediment and overlying water will be added to exposure beakers the day before the start of the exposures at 23°C under static conditions. Overlying water addition will be initiated on day 0 prior to the addition of organisms with no water renewal for 8 hours after test organisms are added to the exposure chambers. The source of overlying water for all midge toxicity tests will be moderately hard reconstituted water created using methods specified in USEPA (2000).

Twenty-five (25) replicate toxicity beakers will be tested for each sediment (16 for biological endpoints and 3 each for chemistry analysis at day 7, sometime between days 21 and 27, and again between days 42 and 49). Four of the biological endpoint beakers will be used to provide auxiliary males only. Twice daily volume additions of overlying water will be conducted. Full-spectrum fluorescent lights providing approximately 100 to 1000 lux (at the sediment interface) and a photoperiod of 16 h light and 8 h darkness will be provided. 4 d old organisms will be used to initiate the exposures. Egg masses will be obtained from Environmental Consulting and Testing, (Superior, WI) and allowed to hatch at the ERDC. A total of 12 midge larvae (4 d old) will be added each beaker. A suspension of Tetramin® fish food (1.5 ml containing 6 mg of Tetramin®) will be provided to each beaker daily. Survival, weight and biomass will be measured on day 20. Male and female emergence, adult mortality, number of egg cases produced and number of eggs hatched will be recorded until test termination (based on cessation of emergence

in the control). Standard bioassay conditions and other required performance criteria for the above referenced four tests can be found in Appendix A.

B.4.1.3 – *Hyalella azteca* 28-Day Chronic Test

Chronic sediment toxicity testing (28-d) with the amphipod, *Hyalella azteca,* will be conducted in basic accordance with procedures outlined in ASTM (2012) and USEPA (2000). A performance control provided by ERDC consisting of a sediment collected from the University of Mississippi Field Station, Abbeville, MS (total organic carbon about 1.5%) along with a quartz sand control sediment and a performance controls sediment provided by PER will also be evaluated. Up to 10 field-collected sediments will be evaluated using the 10-day short-term midge toxicity test.

Amphipod exposures will be conducted in 300-ml beakers containing 175 ml of overlying water and 100 ml of sediment. To allow for sediment equilibration, sediment and overlying water will be added to exposure beakers the day before the start of the exposures at 23°C under static conditions. Overlying water addition will be initiated at day 0 prior to adding amphipods to exposure chambers. The source of overlying water for all toxicity tests will be reconstituted water created using methods specified in Borgmann (1996) but modified to contain 0.4 mg/L bromide.

Fourteen (14) replicate toxicity beakers will be included for each sediment 8 for biological endpoints, 3 for pore water chemistry analysis at day 7 and 3 for pore water chemistry during the week prior to day 28). Ten amphipods will be added to each exposure beaker at test initiation. Twice daily volume additions of overlying water will be conducted. Full-spectrum fluorescent lights with about 100 to 1000 lux (at the sediment interface) and a photoperiod of 16 h light and 8 h darkness will be provided. 7 to 8-d old (known age) amphipods will be used to initiate the exposures with a goal of achieving starting weights in the range of about 0.02 to 0.035 mg/organism. The weight of a representative sample of organisms at the start of the sediment exposures will be documented. A mixture of yeast, Cerophyl, trout chow (YCT) food will be provided to each beaker daily. One mL YCT (from a 1800 mg solids/L stock) per day will be introduced to each test chamber during days 0 to 13 and 2 ml of YCT per day will be added to each test chamber during the remaining exposure. At the end of the sediment toxicity tests, sediment will be passed through a 425 µm sieve and surviving organisms collected. Endpoints measured at the end of the 28-d amphipod exposures will be survival, weight, and biomass. Mean weight of *H. azteca* in the control sediment on Day 28 should be greater than or equal to 0.4 mg dry/individual. Mean survival of *H. azteca* in the control sediment on Day 28 should be greater than or equal to 80%. Standard

bioassay conditions and other required performance criteria for the above referenced four tests can be found in Appendix A.

B.4.1.4 – *Hyalella azteca* 42-Day Chronic Test (Optional)

Chronic sediment toxicity testing (42-d) with the amphipod, *Hyalella azteca,* will be conducted in basic accordance with procedures outlined in ASTM (2012) and USEPA (2000). A performance control provided by ERDC consisting of a sediment collected from the University of Mississippi Field Station, Abbeville, MS (total organic arbon about 1.5%) along with a quartz sand control sediment and a performance controls sediment provided by PER will also be evaluated. A to be determined number of field-collected sediments will be evaluated using the 42-day amphipod toxicity test.

Amphipod exposures will be conducted in 300-ml beakers containing 175 ml of overlying water and 100 ml of sediment. To allow for sediment equilibration, sediment and overlying water will be added to exposure beakers the day before the start of the exposures at 23°C under static conditions. Overlying water addition will be initiated at day 0 prior to adding amphipods to exposure chambers. The source of overlying water for all toxicity tests will be reconstituted water created using methods specified in Borgmann (1996) but modified to contain 0.4 mg/L bromide.

Eighteen (18) replicate toxicity beakers will be included for each sediment (12 for biological endpoints and 3 each for porewater chemistry analysis at day 28 and sometime between days 21 and 27). Of the 12 replicates for biological endpoints, 4 replicates are for 28-d survival and growth and 8 replicates are for 35- and 42-d survival, growth, and reproduction. Ten amphipods will be added to each exposure beaker at test initiation. Twice daily volume additions of overlying water will be conducted. Full-spectrum fluorescent lights with about 100 to 1000 lux (at the sediment interface) and a photoperiod of 16 h light and 8 h darkness will be provided. 7 to 8-d old (known age) amphipods will be used to initiate the exposures. A mixture of yeast, Cerophyl, trout chow (YCT) food will be provided to each beaker daily. 1.0 ml YCT (from a 1800 mg solids/L stock) per day will be introduced to each test chamber during days 0 to 13 and 2 ml of YCT per day will be added to each test chamber during the remaining exposure. Endpoints measured for the 42-d chronic test include survival at days 28, 35 and 42, weight and biomass at days 28 and 42, reproduction at days 35 and 42, and number of males and females at day 42. A 3-cm x 3-cm piece of Nitex screen will be used as a substrate for *H. azteca* during the day 28-42 water only portion of the 42 day *H. azteca* bioassay. Mean weight of *H. azteca* in the control sediment on Day 28 should be greater than or equal to 0.4 mg dry/individual and greater than or equal to 0.5 mg dry/individual

on Day 42. Standard bioassay conditions and other required performance criteria for the above referenced four tests can be found in Appendix A.

B.4.2 – Overlying Water Quality

Overlying water quality (dissolved oxygen, pH, conductivity, hardness, ammonia and alkalinity) will be measured in a composite from each exposure chamber at the beginning and the end of each test. Temperature will be recorded daily in one replicate of each sediment. Dissolved oxygen will be measure daily in a composite sample of overlying water from each sediment treatment. pH will be measured 3 times per week and conductivity will be measured once per week in a composite overlying water sample. Dissolved oxygen will be measured more frequently if it decreases more than 1 mg/L in a treatment since the previous measurement. Aeration will be provided if DO drops below 2.5 mg/L. Light intensity will be measured and recorded at the beginning and end of each bioassay.

B.4.3 – Pore Water Sampling

Porewater will be sampled by centrifugation of sediment in 1.0 L polycarbonate centrifuge tubes (Thermo Scientific Nalgene, part # 31221010) at 2,500 x g for 30 minutes in basic accordance with methods given in USEPA (2001b). The source of the sediment for centrifugation will be the homogenized buckets of sediment with samples taken concurrently with sediment addition to bioassay test beakers. After centrifugation, the supernatant will be removed and stored in chemically-cleaned glass bottle in the dark at 4 °C until analysis. These porewater samples will be analyzed for TAL metals (except Hg) as well as DOC, pH, alkalinity, sulfide, major cations, and major anions to inform the BLM for interpreting toxicity data and for comparison to PER's results. Cations/anions to be included in these pore water analyses are Ca, Cl, Mg, K Na, and sulfate.

Porewater will also be collected using Brumbaugh type peepers using methods given in "Standard Operating Procedure 9 (SOP-9), U.S. Geological Survey – Columbia Environmental Research Center, Peeper Method For In-Situ Sampling of Sediment Porewater" found in Appendix A – Attachment 2 of the Upper Columbia River Final Quality Assurance Project Plan for the Phase 2 Sediment Study (E^xponent and HDR|HydroQual 2013). Porewater collected from the Brumbaugh type peeper will be analyzed for TAL metals except Hg. Peepers will be deployed in dedicated chemistry beakers in each sediment in each bioassay 7 days prior to each porewater retrieval. For the numbers of these beakers to be used in each bioassay and on what test days the peepers will be sampled on, see section B4.1. These chemistry beakers will contain organisms and be treated the same as biological endpoint beakers, but no organismal endpoints will be assessed in the chemistry beakers.

B.4.4 – Analytical Chemistry Methods

Guidelines for the analyses are provided by USEPA (e.g. SW-846) and ASTM standard methods. Analytical techniques used for the project are outlined below. All analyses will be performed within the holding period described in the method guidance documents. Parameters to be analyzed are listed in the following table.

Table 1. Reporting Limits

Analyte	Method	Water RL (µg/L-unless specified otherwise)	Sediment RL (mg/kg-unless specified otherwise)
Grain Size	ASTM-D422	N/A	0.1 %
pH	150.1	0.1 SU	N/A
AVS-SEM*	9030/6010/6020/7474	20(sulfide)	2(sulfide)
TOC/DOC	9060	2000	200
TAL Metals	6010/6020/7474	20/1/0.01	2/0.1/0.005
Alkalinity	310.2	10000	N/A
Hardness	6010 (Calculated)	40	N/A
Chloride/Sulfate	300 Series	20	NA

*Reporting limits for SEM are calculated considering the extraction volume and mass of the AVS procedure and dilutions, and are approximately 5 times higher than water and sediment RLs.

The analytical methods and reporting limits for the target trace metals are summarized in Table 1 above. Results are presented in units of mg/Kg and are based on an initial sample weight of approximately 0.5 grams (g) and a final digestion volume of 50 mL for ICP-AES and ICP-MS metals. Grain size requires approximately 3L of material. AVS-SEM and TOC require approximately 10 g total, resulting in a total sediment sample size of approximately 5 L of material for all analytical tests. Porewater analytical requirements are minimally 100 mL for all tests indicated in the table above.

Full laboratory data reports will be provided in electronic format to the task coordinator, who will oversee archiving the final data and data quality reports in the project file. EDDs will be prepared in spreadsheet and PDF format. Documentation requirements, to be included in the final report from the U.S. Army ERDC Project Manager to the USEPA Project Manager, for the analytical laboratory (ERDC-EP-C) include the following:

- A cover letter discussing analytical procedures and any difficulties that were encountered
- Sample receipt and analysis dates
- Final analyte concentration including reporting limit, laboratory qualifiers, and reanalysis (if any)
- Percent recovery of each compound in the matrix spike sample
- Matrix spike recovery control limits
- Relative percent difference (RPD) for all MS/MSD and/or laboratory control sample (LCS)/LCS duplicate (LCSD) results
- RPD control limits for MS/MSD and/or LCS/LCSD reports
- LCS results when analyzed
- Recovery control limits for LCS or standard reference material recoveries and relative standard deviation
- Method blank summary indicating associated samples
- Case narrative

B.4.4.1 – Overlying Water Quality

Water quality during bioassay testing will be measured using Thermo Scientific Orion 4Star meters (Thermo Scientific**,** Beverly, MA) equipped with a model 013005MD conductivity cell for measuring electrical conductivity, a model 9107WMMD automatic temperature compensating pH Triode for measuring pH, or an optical luminescence Rugged Dissolved Oxygen (RDO) probe for measuring dissolved oxygen and temperature. Total ammonia, hardness and alkalinity will be measured using LeMotte titration kits (Chestertown, MD, USA). In addition, ammonia will be measured using a 720A ion-selective electrode (ISE) meter (Thermo Orion Electron Corp., Beverly, MA) equipped with a 95-12 ammonia-sensitive electrode (Thermo Orion Electron Corp., Beverly, MA) for confirmation of concentrations exceeding 8 mg/L.

B.5 – QUALITY CONTROL REQUIREMENTS

Whole sediment toxicity testing quality control (QC) requirements are discussed in Section A.7. A 4-d water only reference toxicity test with cadmium chloride will be conducted with the same batch of animals used for testing in basic accordance with the procedures outlined in ASTM (2012) and USEPA (2000). The data generated from this test will be compared to historical control chart data to ensure organisms used in testing are healthy and appropriately sensitive. Control chart data will be provided to EPA as part of the data quality review package.

Extensive and detailed requirements for laboratory QC procedures are provided in the EPA and other standard methods that will be used for this study. Each method protocol includes descriptions of QC procedures, and many incorporate additional QC requirements by reference to separate QC sections. QC requirements include control limits and requirements for corrective action in many cases. QC procedures will be completed by the laboratories, as required in each protocol and their internal SOPs, and as indicated in this QAPP.

The frequency of analysis for LCSs, MS/MSD samples or laboratory duplicates, and method blanks will be one for every 20 samples or one per extraction or analysis batch, whichever is more frequent. Calibration procedures will be completed at the frequency specified in each method description. Equipment blanks will be subjected to the same processes as the sediment preparation.

As required for EPA SW-846 methods (USEPA 2008), performance-based control limits have been established by the laboratory. These and all other control limits specified in the method descriptions will be used by the laboratory to establish the acceptability of the data or the need for reextraction or reanalysis of the samples. All analytical data will be reviewed with respect to project-specific data quality objectives, which include attainment of adequate precision, accuracy, representativeness, comparability, and completeness (PARCC). Of the PARCC parameters, precision and accuracy will be evaluated quantitatively through the collection and analysis of QA/QC samples as listed in Table 7. Criteria for individual analyses will follow and adhere to the performance provisions appropriate to the respective contract laboratory QA/QC programs. Standard QC protocols will be followed according to method specified.

Precision

Precision is a measure of reproducibility as determined by the degree of agreement between multiple measurements of the same parameter under identical conditions. Precision is expressed quantitatively as the extent of variability of individual measurements from the mean of multiple measurements.

Table 2. QC samples for precision and accuracy.

QC Type	Precision	Accuracy	Minimum Frequency
Laboratory QC	MS/MSD RPD	MS/MSD %R	1/20 samples
	LCS/LCSD RPD	LCS/LCSD %R	1/20 samples
		Method Blank	1/20 samples

Notes:

%R = percent recovery.

LCS/LCSD = laboratory control sample/laboratory control sample duplicate.
MS/MSD = matrix spike/matrix spike duplicate.

RSD = relative standard deviation. RPD = relative percent difference.

Method guidance defines an analytical batch as 20 samples, with associated QC included at a rate of at least 1 per 20 sample batch for MS/MSD and LCS.

Precision will be evaluated by collecting and analyzing laboratory replicates, comparing the results, and then calculating the Relative Percent Difference (RPD) by using the following equation:

$$RPD = \frac{|A - B|}{(A + B) / 2}, \ 100\%$$

where:

A = primary sample concentration
B = duplicate sample concentration

Laboratory replicates will be analyzed at a rate of at least 1/20 samples during the study as required by method guidance. General precision control limits for the analytical laboratory are ±20%. Data that do not meet these precision criteria may be qualified as estimated (i.e., "J") during data review.

Accuracy

Accuracy is a measure of the deviation (or agreement) between an analytical measurement and the true or accepted value for a standard reference material. The accuracy of a measurement system can be affected by errors introduced by cross-contamination in the field sampling process, sample preservation, sample handling, matrix sample preparation, analytical techniques, and cross-contamination in the laboratory. A program of sample spiking will be conducted to evaluate laboratory accuracy. This program includes analysis of the MS/MSD samples, LCS/LCSD samples, and method blanks. MS/MSD and LCS/LCSD samples are analyzed at a frequency of one per batch; a batch of samples is limited to 20 samples. The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy.

Accuracy is expressed as the percent recovery of an analyte that has been added (spiked) to an environmental sample in a known concentration before extraction/analysis. Accuracy is calculated using the following equation:

$$\text{Percent Recovery} = \frac{(C + S) - C}{T} \times 100$$

where:

S = measured spike sample concentration
C = sample concentration
T = true or actual concentration of the spike

Concentrations and recovery limits for standard reference materials are based on the type of sample being analyzed and the certified values provided by the supplier. Appropriate spike and reference standard compounds and concentration levels are specified in the analytical methods. If the spiking levels for MS/MSD and LCS/LCSD are not provided, the spiking will be conducted

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an environmental condition.

- Were samples handled correctly?
- Were samples collected from appropriate locations?
- Were an appropriate number of samples collected and analyzed?
- Did other factors bias the results?

Comparability

Comparability is a qualitative parameter that expresses the degree of confidence with which one data set can be compared to another. Comparability of data will be achieved by consistently following procedures for subsampling and analysis activities, by using the same types of sampling equipment, and by using standard measurement units in reporting analytical data. Laboratory data will be reported in consistent units for each analytical test (i.e., mg/kg).

Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures and when none of the QC criteria that affect data usability are exceeded. Data that are validated and qualified as estimated will not be counted against the completeness goal because they are considered usable. Only rejected data or data not collected will be counted against the completeness goal. When all data validation is completed, the percent completeness will be calculated by dividing the number of valid sample results by the total number of sample results planned for this investigation. The following equation is used to determine completeness:

$$\text{Completeness } (\%C) = \frac{V}{T} \cdot 100$$

where:

%C = percent completeness
V = number of valid samples
T = total number of planned samples

Although a quantitative number can be calculated for each analyte, the data user must use this qualitatively to assess whether the investigation objectives can be met with the data obtained. As a guideline, data completeness should be approximately 90% for each analyte for all samples.

Data that do not meet completeness goals may suggest the need for resampling and analysis or, at a minimum, may suggest that the data set should be used with caution, depending on the effect of the incomplete data on the data quality objectives. Data that were planned but not collected should count against the completeness goal, unless they were omitted for a valid reason and corrective actions documented.

B.6 – INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Instrumentation will be maintained in a manner established by the manufacturer and communicated to the end user through standard operating procedures. Established performance criteria (e.g., instrument slope, etc.) will be utilized to determine the functional state of instrumentation where appropriate. Instrumentation used for chemical

analysis within the Environmental Chemistry Branch is maintained under an Instrumentation Service Contract.

B.7 – INSTRUMENT CALIBRATION AND FREQUENCY

Instrument logs will be established to allow linking of data generated to the instrument used to obtain the measure. Instruments will be calibrated prior to use according to manufacturer and standard method guidance using standards that bracket the expected target concentration of the measured value. Where applicable, a traceable standard, manufactured following procedures from a nationally recognized standard organization such as NIST, will be utilized. Calibration results and frequency will be logged for each instrument. Where commercial standards are used a record of standard certification will be kept with the project documentation. Where required by method guidance, a traceable second source calibration verification standard will be used.

B.8 – INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

All supplies, consumables and equipment that come into contact with sediment will be cleaned following established decontamination procedure, where appropriate, to ensure no sample cross contamination occurs. The quality of the water used during testing will documented. The appropriate grade and purity of chemicals will be utilized where required. Certificates of analysis or other documents of certification for chemicals and containers will be maintained with project documentation.

B.9 – DATA MANAGEMENT

All hard copies of data documents will be maintained in a secure location with access control measures in place. All documents and other information supporting the research will be maintained at the ERDC for 7 years following the delivery of the final report. Electronic files, communication, records and other information will be maintained on the Army Records Information Management System (ARIMS) or on external hard drive where necessary. All analytical data produced will be stored in the U.S. Army ERDC Environmental Chemistry Branch LIMS for a period of at least seven years.

SECTION C – ASSESSMENT/OVERSIGHT

This task will rely on the knowledge and expertise of the U.S. Army ERDC project delivery team. The U.S. Army ERDC Project Manager, QA officer, and Co-principal investigators will remain in close communication throughout the testing and reporting phases of this project. This level of communication will serve to keep the management team appraised of activities and events, and will allow for informal but continuous task oversight.

C.1 – ASSESSMENTS AND RESPONSE ACTIONS

Assessments that will be performed as part of this project include:

- An assessment by the PIs before the start of testing that all technicians working on the project have received all required training on methods utilized in this project.
- An assessment by the QA officer at the end of testing that all QA requirements have been met.
- An assessment by the PIs at the end of testing of all water quality and toxicity data for completeness and accuracy.

In the event of non-conforming conditions or any deviations from prescribed laboratory protocols the USEPA Project Manager or her designee will be consulted about what corrective action should be taken to ensure data quality issues are minimized. However, if experimental conditions require an immediate decision on how to handle a deviation, the US Army ERDC co-principal investigators and/or the U.S Army ERDC Project manager, will use their best professional judgment on the appropriate corrective action. The US EPA Project Manager will then be notified, via a corrective action report, about the corrective action taken as soon as possible (not to exceed 3 business days) after the corrective action is taken. The non-conforming condition, corrective options, and correction will be documented via a deviation and corrective action report provided to the USEPA Project Manager. A deviation and corrective action report will be created and included in the final report.

C.2 – REPORTS TO MANAGEMENT

Toxicity test results from the *H. azteca* and *C. dilutus* bioassays as well as analytical chemistry results will be summarized in a spreadsheet (Microsoft Excel format) and also provided in a written report (Microsoft Word format) and submitted to the USEPA Project Manager, Laura Buelow by the U.S. Army ERDC Senior Scientist, Jeff Steevens or Program Manager, Jacob Stanley, after completion of testing. Electronic data from both bioassays and chemistry analyses will be provided in appropriate Excel spreadsheet format to the UCR Project Database manager (Cristy Kessel, E^{x}ponent) for upload to the

project database. Additional progress reports will be provided to management upon request. Deviation and corrective action reports, if needed, will be submitted to the USEPA Project Manager as described in Section C.1.

SECTION D – DATA VALIDATION AND USABILITY

D.1 – DATA REVIEW, VERIFICATION, AND VALIDATION

U.S. Army ERDC will evaluate data using the performance criteria listen in Section A.7. If the reviewers identify data that is considered suspect, the data will be investigated further in order to ensure that the data is accurate and does not represent an error. A narrative of the data investigation will be documented. If the data are determined to be in error, the erroneous value will be replaced with the correct value. If the investigation concludes that the data are suspect but a correct value cannot be determined, then the data will be flagged to indicate its suspect status and a decision will be made in conjunction with the USEPA Project Manager on whether the data should be accepted, rejected, or otherwise qualified.

D.2 – VERIFICATION AND VALIDATION METHODS

A series of reviews will be performed by technical personnel to ensure the data generated meet the data quality objectives and will include the following:

- All data entered into spreadsheets will be checked for accuracy through a 100% comparison with the hard-copy data listing. If errors are discovered, they will be corrected.
- At the end of each work day, data collected that day will be reviewed by laboratory personnel for completeness and to identify potential errors.
- At the completion of an experiment, all data will be reviewed for potential errors by a co-principal investigator and the QA officer.

D.3 – RECONCILIATION WITH USER REQUIREMENTS

The goal of data validation is to determine the quality of each data result and to identify those that do not meet data quality objectives. Non-conforming data may be qualified as estimated (i.e., a “J” qualifier will be applied to the result) or rejected as unusable (i.e., an “R” qualifier will be applied to the result) during data validation if criteria for data quality are not met. Rejected data will not be used for any purpose. A summary of the qualified data and the reasons for qualification will be included in the report.

Data qualified as estimated will be used for all intended purposes and will be appropriately qualified in the final project database. However, these data are less precise or less accurate than unqualified data. Data users are responsible for assessing the effect of the inaccuracy or imprecision of the qualified data on statistical procedures and other data uses.

SECTION E – REFERENCES CITED IN TEXT OF QAPP

American Society for Testing and Materials (ASTM). 2012. Standard test method for measuring the toxicity of sediment-associated contaminants with freshwater invertebrates (ASTM 1444 E1706-05). Annual Book of ASTM Standards Volume 11.06, West Conshohocken, PA.

Borgmann U. 1996. Systematic analysis of aqueous ion requirements of Hyalella azteca: A standard artificial medium including the essential bromide ion. *Archives of Environmental Contamination and Toxicology* 30:356-363.

E^{x}ponent and HDR|HydroQual. 2013. Upper Columbia River final quality assurance project plan for the phase 2 sediment study.

USEPA. 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates, second edition. EPA 600/R-99/064, Duluth, MN and Washington, DC.

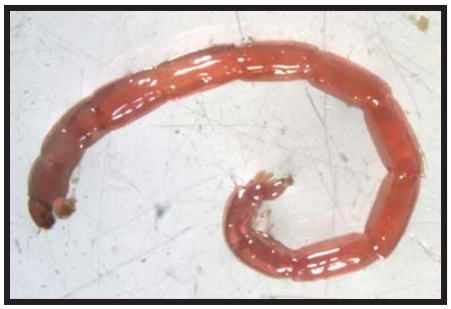
USEPA. 2001a. USEPA Requirements for Quality Assurance Project Plans, USEPA QA/R-5.

USEPA. 2001b. Methods for collection, storage and manipulation of sediments for chemical and toxicological analyses: Technical manual. EPA-823-B-01-002, Washington D.C.

SECTION F-APPENDICES

Appendix A - Biological Testing Standard Operating Procedures

Appendix A.1 - Protocol for Conducting 10-d Sediment Toxicity Tests Using *Chironomus dilutus*



Testing Protocol Provided by:

US Army Engineer Research and Development Center (ERDC)

Environmental Laboratory, EP-R

3909 Halls Ferry Rd

Vicksburg, MS 39180

Ecotoxicology and Environmental Risk Team

Team Leader:

Dr. Jacob Stanley
Tel: 601-634-3544
Jacob.K.Stanley@usace.army.mil

Mr. J. Daniel Farrar
Tel: 601-634-2118
Daniel.Farrar@usace.army.mil

Mr. Alan J. Kennedy
Tel: 601-634-3344
Alan.J.Kennedy@usace.army.mil

1. Testing Procedure Overview

The protocol to follow provides guidance for conducting 10-d sediment toxicity tests using the freshwater midge *Chironomus dilutus.* The protocol is in basic accordance with the guidance provided in "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates" (EPA-600-R99-064) and "Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates" (ASTM, 1706-05 (2010)).

2. Required Materials for Preparation and Testing

- Biological testing material
  - ✓ *Chironomus dilutus* larvae (2nd and 3rd instar; approximately 50% in second instar and 50% in third instar)
  - ✓ Control, reference and site sediment
  - ✓ Reconstituted moderately hard fresh water prepared as described in USEPA (2000)
  - ✓ Environmental chamber with temperature and photoperiod control
  - ✓ Tetramin® Fish Flakes (ground to 500 µm particle size)
- Glassware and accessories
  - ✓ Testing chambers (11 300 mL beakers per treatment, 8 for assessment of biological endpoints and 3 chemistry-only beakers) prepared for use in a Zumwalt or modified Brunson-Zumwalt water exchange system (US EPA 2000)
  - ✓ Test initiation/maintenance/breakdown materials
    - Transfer pipettes
    - Turbulence reducers (e.g., nylon, Teflon or polyethylene disks attached to a rod)
    - Eight inch diameter #40 sieves (425 µm mesh sizes)
    - Plastic wash bottles
    - Reconstituted moderately hard water for probe rinsing, etc.
    - Talley counters (optional)
    - Pans or bowls for counting
    - Heavy duty aluminum foil pans for initial and final weight determination
- Instruments and equipment
  - ✓ Electric drill/mixer with stainless steel mixing rod
  - ✓ Zumwalt or modified Brunson-Zumwalt water exchange system (US EPA 2000)
  - ✓ Stainless steel or high density polyethylene (HDPE) spoon/mixing rods
  - ✓ Centrifuge to separate pore water from sediment
  - ✓ Centrifuge tubes
  - ✓ Photoperiod timers
  - ✓ Temperature controllers
  - ✓ Dissolved oxygen (D.O.) meter
  - ✓ Thermometer (°C)
  - ✓ pH meter
  - ✓ Light boxes
  - ✓ Desiccators for holding dried organisms

- ✓ Drying oven (capable of 60 °C temperature)
- ✓ Muffle furnace (capable of 550 °C temperature)
- ✓ Balance (0.01 mg resolution)
- ✓ Aeration system (if required due to low dissolved oxygen)
- ✓ Salinity/conductivity meter or refractometer
- ✓ ISE meter (Orion 720A or similar)
  - Ammonia sensitive electrode (Orion 95-12 or similar)
  - 0.1 M or 1000 ppm NH_3 standard, ionic strength adjuster

- Chemicals
  - ✓ Liquid soap (e.g., Liqui-Nox)
  - ✓ 10% nitric acid
  - ✓ Acetone
  - ✓ Ammonium chloride (reference test)
- Miscellaneous
  - ✓ Data sheets: live animal acclimation, water quality, survival, growth and reproduction
  - ✓ Labels for beakers
  - ✓ Safety equipment (e.g., gloves, lab coats, safety glasses, safety showers and eye fountains)

3. Health, Safety and Waste Management

3.1. Personal safety equipment should be used by all participating technicians due to the unknown nature of test sediment contamination.

3.1.1. Use gloves, laboratory coats, safety glasses and respirators where applicable

3.1.2. Manipulate sediment (as needed) and other chemicals (e.g., acids, volatile organic solvents) under fume hood

3.2. Laboratory safety equipment such as first aid kits, emergency showers, eye fountains must be available to workers

3.3. All sediment wastes should be captured in a labeled five gallon bucket (or similar container) during the breakdown and sieving of the toxicity test.

3.4. All containers (chemical and waste) must be properly labeled.

3.5. Sediment and chemical wastes should be held and disposed of in consultation with the customer and in compliance with the participating laboratory's applicable state and federal laws.

4. Receipt of sediment

4.1. Sediment will be shipped to the ERDC in plastic buckets or other container as deemed appropriate based on the chemical class of the contaminant of concern. Sediment will be shipped at 4.0 ± 2°C. The temperature and other pertinent information (COC, etc.) should be completed on arrival, sediment logged into sediment storage record book and placed into a locked cold room at 4.0 ± 2°C until required for testing.

5. Receipt and Hatching of Egg Masses (Day -10 to -12)

5.1. Egg Masses are obtained from a commercial vendor (e.g., Environmental Consulting and Testing (superior, WI)) and shipped overnight to the ERDC. Egg masses should be shipped in the appropriate water quality conditions (temperature 23 ± 5°C, D.O. ≥ 4.0 mg/L). An organism receipt and acclimation form should be completed upon receipt of the egg masses and any deviations in the water quality ranges noted.

5.2. The temperature of the egg masses are brought to 23°C and are placed in plastic tote tubs containing fresh water. The tubs are placed in an environmental chamber at 23°C and under 16:8 Light:Dark cycle. Aeration with an airstone is provided.

5.3. Eggs will begin to hatch in approximately 48 hours. When hatching is observed, 300 mg of Tetramin® is provided to each tub by mixing the food with fresh water and delivering as a slurry. Organisms are then water exchanged (50%) and fed 200 mg of Tetramin® every three days or as required based on observed food consumption. Larvae will build tubes out of the food provided.

5.4. Larvae will reach the appropriate size for testing approximately 10 days after hatching. Organism instar and instar ratio should be determined as describe in the above QAPP.

6. Test chamber and sediment preparation (day –1sediment addition)

6.1 Glass soap water/acetone/acid-washed 300 mL beakers containing mesh screen overflow holes will serve as test chambers. The number required is eight replicates/beakers per treatment plus 3 chemistry-only beakers per treatment. Each beaker should be labeled with the treatment and replicate (e.g., A, B, C, D and E) designation.

6.2 Prior to test initiation, sediment should be homogenized thoroughly within the bucket or other suitable container using an electric drill or mixer equipped with a stainless steel shaft and prop/agitator. After ~5 minutes of thorough mixing, the sediment in the buckets should be manually mixed with a stainless steel spatula or HDPE scoop, involving complete turnover of the sediment (i.e., bringing the bottom to the top). Utilize the power mixer again and repeat until the sediment is thoroughly mixed, homogenous in texture/color and has an even distribution of water. Repeat this process for all sediments following decontamination of equipment (Section 5.3).

6.3 Decontamination: The homogenization equipment (e.g., mixer, spatula) should be thoroughly decontaminated between the mixing of each sediment. This includes a (1) water rinse, (2) soap water scrubbing, (3) rinse with 10% nitric acid, (4) rinse with acetone[1] and (5) deionized water wash. Rinses must be thorough to ensure all acid/solvent is removed and soaking glassware overnight in deionized water is an option.

6.4 Beakers will be filled with 100 mL (~ 2 cm) of homogenized sediment (as described in section 4.2) from each site, control and reference using a decontaminated stainless steel or HDPE scoop/spatula. The sediment should be added carefully to minimize contact with the sides of the beakers and leveled by tapping the side of the beakers.

6.5 Slowly add the overlaying filtered/dechlorinated fresh water using a turbulence reducer to minimize resuspension of the sediment. Beakers should be filled to the overflow holes on the beakers.

6.6 Arbitrarily place all treatment replicates in the specified Zumwalt or modified Brunson-Zumwalt water exchange system. The water exchange system should be in a controlled

[1] Attention should be given to whether all equipment is stainless steel or HDPE before using acetone rinses due to concern regarding chemical release (e.g., phthalates from plastics).

environmental chamber at 23 ± 1°C. No aeration is provided unless DO drops below minimum requirements (2.5 mg/L) during testing.

6.7 Porewater characterization is conducted on day -1 or shortly after sediment receipt. Centrifuge at 2500 g for 30 minutes. Decant supernatant and measure temperature, pH, and total ammonia on the supernatant. Porewater for chemistry analysis is collected in an identical manner at day-1 using centrifuge bottles of appropriate volume.

7. Test initiation (day 0)

7.1. A full water exchange should be conducted on test day 0 prior to measuring parameters and adding organisms.

7.2. Water quality measurements (temperature, pH, and dissolved oxygen (DO), hardness, alkalinity, conductivity, and total ammonia) will be taken on a composite sample collected from the replicates of each sediment and recorded. If parameters are within the specified ranges (US EPA 2000), the test may be initiated. If DO drops below 2.5 mg/L then aeration should be implemented.

7.3. *Chironomus dilutus* larvae (2^{nd} and 3^{rd} instar) will be counted directly into each exposure beaker. Beakers can be removed from the water exchange system to facilitate organism addition. Ten organisms should be randomly counted into each exposure beaker by replicate (i.e., count into all replicate A beakers, then replicate B beakers, etc.). Organism transfer should be carried out using plastic disposable pipettes with the non-bulb end cut to the appropriate size to pipette larvae and food tubes. Fully submerge the pipettes when transferring larvae. Effort should be made to transfer larvae within their food tubes to minimize injury (forceps are not to be used). Only apparently healthy individuals should be selected for testing. Any organisms that are dropped or contact a dry surface cannot be used in testing. Special attention should be paid to floating larvae, which should be submerged using a drop of test water from the test beaker using a pipette. Count out the required number of larvae for initial weight measurements and reference toxicity testing. The ash-free dry weight of a representative sample of 80 organisms used to start the bioassay will be documented (e.g., 4 replicates of 20 organisms/replicate).

7.4. Upon addition of test organisms, the time of test initiation must be recorded on the appropriate data sheet.

7.5. Organisms in each beaker are provided 1.5 ml of water/food slurry containing 6 mg of Tetramin®.

7.6. Approximately one hour following the addition of test organisms, chambers should be observed for injured or unhealthy larvae. At this time, injured or unhealthy individuals or individuals may be replaced if the response does not appear to be specific to the particular sediment treatment. Floating larvae should be submerged using a drop of test water from the test beaker using a pipette.

8. Test monitoring and maintenance (days 1 – 9)

8.1. Test chambers should be observed twice daily for floating larvae during the first week. After the first week they should be observed once daily. Any larvae observed floating should be submerged using water droplets dropped gently from a pipette.

8.2. Water additions (2 volume additions of overlying water per day) will be conducted twice daily using the automated Brunson-Zumwalt system or manually using the Zumwalt delivery system.
8.3. Water quality parameters (temperature and DO) will be measured daily in a replicate of each treatment prior to water exchange. Dissolved oxygen will be measured more frequently if it decreases more than 1 mg/L in a treatment since the previous measurement. Aeration will be provided if DO drops below 2.5 mg/L.
8.4. Organisms in each beaker are provided 1.5 ml of water/food slurry containing 6 mg of Tetramin® daily following water quality parameters.
8.5. Record exposure system temperature daily.
8.6. Check light cycle and ensure that each test chamber is adequately aerated (if required) daily.

9. Test termination/breakdown (day 10)

9.1. Water quality: (temperature, pH, DO, alkalinity, hardness, , conductivity, and total ammonia) is measured in a composite sample collected from all replicates of a sediment
9.2. Survival endpoint determination
9.2.1. Gently pour off all but 50 mL of overlying water through an 8 or 12-inch diameter 425 µM ASTM testing sieve to isolate larvae. Swirl and suspend sediment in the remaining overlying water for easier passing of sediment through the sieves. If using an 8 inch sieve, a 12-inch diameter sieve (1 mm mesh) can be placed over the bucket receiving the waste to support the 8 inch sieve.
9.2.2. Transfer small amounts of the material retained on each sieve to a counting bowl or tray for examination. Count surviving larvae. All missing individuals or larvae that fail to move following close observation (e.g., under a dissection microscope) and gentle prodding with a blunt probe are considered dead. Individuals exhibiting any movement are counted as alive.
9.2.3. Organisms that have pupated are counted as live but are not used for weight and biomass determination.
9.2.4. Surviving larvae from each replicate beaker are placed on ashed pre-weighed pans and placed in a drying oven at 60°C for 24 hours. After 24 hours, pans are placed in a desiccator and allowed to cool (4 hours). Dry weights are recorded for each pan. Pans are then placed into a muffle furnace at 550 °C for 2 hours. After 2 hours of ashing, pans are placed in a desiccator to cool (4 hours). Once cool pans are weighed and ash weight is recorded. Ash-free dry weight is then determined by the following formula:(Dry weight – pan weight) - (Ash-free weight – pan weight)
9.2.5. Biomass will be reported as average final mass of each sediment replicate. Weight is reported as the average individual ash-free dry weight for a sediment by dividing total biomass of each replicate by the number of surviving organisms within the replicate then calculating the mean value.

10. Acceptability

10.1. The test is not acceptable if any of the following do not occur.

10.1.1. Tests must be started with second- to third-instar larvae (about 10-d-old larvae), with a goal to achieve a starting average weight of about 0.12 mg/organism.
10.1.2. 70% or greater average survival in the control
10.1.3. Average size of *C. dilutus* in the control sediment must be at least 0.48 mg AFDW at the end of the test.
10.1.4. Hardness, alkalinity, and ammonia in the overlying water typically should not vary by more than 50% during the test, and dissolved oxygen should be maintained above 2.5 mg/L in the overlying water.

11. Recording data

11.1. All specified data should be recorded in full on the appropriate data sheets (i.e., organism acclimation, water quality, survival, reproduction).
11.2. If any parameters are not within the specified range, record on a datasheet, make a note in the comments and contact study coordinator.

References

ASTM International. 2010. Standard test method for measuring the toxicity of sediment-associated contaminants with estuarine and marine invertebrates. ASTM, 1706-05. ASTM International, West Conshohocken, PA

US EPA. 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates. EPA-600-R99-064. US EPA. Washington, DC.

USEPA/USACE 1998. US Environmental Protection Agency/US Army Corps of Engineers. Evaluation of Material Proposed for Discharge to Waters of the U.S. - Testing Manual (Inland Testing Manual). EPA-823-B-98-004, Office of Water, Washington DC

Table A.1-1 Summary of Conditions for 10-d sediment toxicity test using *Chironomus dilutus*

Description	Condition
Test Type	Static renewal
Test Duration	10 days
Temperature	23.0 ±1.0°C
pH	6.5-9
Light Quality	Wide spectrum fluorescent lights
Light Intensity	About 100-1000 lux
Photoperiod	16:8 L:D
Test Chamber Size	300 mL (with screens)
Sediment Depth/volume	2 cm (100 mL)
Overlying Water Volume	Fill to bottom of screens
Sediment settling time	Overnight
Renewal of Overlying water	Two 100% water volume additions daily. Water is moderately hard reconstituted water created using methods specified in USEPA (2000)
Age of Test Organisms	~10 day old larvae (2^{nd} and 3^{rd} instar; approximately 50% in second instar and 50% in third instar) with a goal to achieve an average starting weight of about 0.12 mg/organism
Organisms/chamber	10
Replicates/treatment	11 (8 replicates for biological analysis and 3 replicates for chemistry analysis)
Organisms/treatment	110 (80 in replicates for biological analysis and 30 in replicates for chemistry analysis)
Feeding Regime	1.5 ml of water/food slurry containing 6 mg of Tetramin® daily into each chamber
Test Chamber Cleaning	Clean screens as required with small brush
Test Solution Aeration	None unless DO drops below 2.5 mg/L; Trickle flow (<100 bubbles/min); >60% saturation

Dilution Water	Moderately hard reconstituted water created using methods specified in USEPA (2000)
Test Concentrations	Site sediment, reference, control
Endpoint(s)	Survival, biomass and ash-free dry weight

Table A.1-2. Summary of Tasks for 10-d sediment toxicity test using *Chironomus dilutus*

Day	Task
-12	Receive egg masses and place in culture tubs
-10	Eggs should begin hatching; provide food as described in protocol
-1	Homogenize sediment in shipping bucket or other container appropriate for contaminant(s). Collect pore water samples for chemical analysis via centrifugation. Measure porewater water quality characteristics if not performed earlier. Fill test chambers with sediment and add overlying water.
0	Test initiation: Record temperature, pH, D.O. alkalinity, hardness, conductivity and total ammonia from a composite overlying water sample from all replicates of a sediment treatment. Transfer 10 larvae to each test chamber. Feed 6 mg of Teftrafin® in a water slurry per replicate. Collect 80 larvae for day 0 ash-free dry weight determination. Place peepers in chemistry replicates.
1-9	Record temperature and DO daily. Aerate if DO drops below 2.5 mg/L. Provide each chamber with 6 mg of Tetramin® in a water slurry. Perform two water volume additions per day.
7	Collect peepers and sediment porewater from chemistry replicates
10	Test termination: Record temperature, pH, D.O. alkalinity, hardness, conductivity and total ammonia. Sieve sediment for amphipods and record survival. Place surviving adults on pans and place in drying oven at 60 °C for 24 hours.
11	Place pans in desiccator to cool and record pan dry weights. Place pans in muffle furnace and ash for 2 hours at 500 °C. Place pans in dessicator and allow to cool for 4 hours. Record ashed dry weights.

Appendix A.2 - Protocol for Conducting 28-d Sediment Toxicity Tests Using *Hyalella Azteca*

Testing Protocol Provided by:

US Army Engineer Research and Development Center (ERDC)

Environmental Laboratory, EP-R

3909 Halls Ferry Rd

Vicksburg, MS 39180

Ecotoxicology and Environmental Risk Team

Team Leader:

Dr. Jacob Stanley
Tel: 601-634-3544
Jacob.K.Stanley@usace.army.mil

Mr. J. Daniel Farrar
Tel: 601-634-2118
Daniel.Farrar@usace.army.mil

Mr. Alan J. Kennedy
Tel: 601-634-3344
Alan.J.Kennedy@usace.army.mil

Testing Procedure Overview

The protocol to follow provides guidance for conducting 28-d sediment toxicity tests using the freshwater amphipod *Hyalella azteca.* The protocol is in basic accordance with the guidance provided in "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates" (EPA-600-R99-064) and "Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates" (ASTM, 1706-05 (2010).

1. Required Materials for Preparation and Testing

- Biological testing material
  - ✓ *Hyalella azteca* (7-8 days old)
  - ✓ Control, reference and site sediment
  - ✓ Reconstituted water prepared as described in Borgmann (1996) modified to contain 0.4 mg/L bromide.
  - ✓ Environmental chamber with temperature and photoperiod control
  - ✓ YCT (yeast, Cerophyl® and trout chow) prepared as described in Appendix B of EPA600/R/R-99/064 (US EPA 2000).
- Glassware and accessories
  - ✓ Testing chambers (14 300 mL beakers per treatment, 8 for assessment of biological endpoints and 6 chemistry-only beakers) prepared for use in a Zumwalt or modified Brunson-Zumwalt water exchange system (US EPA 2000)
  - ✓ Test initiation/maintenance/breakdown materials
    - Transfer pipettes
    - Turbulence reducers (e.g., nylon, Teflon or polyethylene disks attached to a rod)
    - 12 inch diameter #25 sieves (710 µm mesh size size) for collection of known age neonates
    - 12 or 8 inch diameter #25 sieves (425 µm mesh size) for test termination
    - Plastic wash bottles
    - Reconstituted Borgmann water for probe rinsing, etc.
    - Talley counters (optional)
    - Pans or bowls for counting
    - Aluminum pans for initial and final weight determination

- Instruments and equipment
  - ✓ Electric drill/mixer with stainless steel mixing rod
  - ✓ Zumwalt or modified Brunson-Zumwalt water exchange system (US EPA 2000)
  - ✓ Stainless steel or high density polyethylene (HDPE) spoon/mixing rods
  - ✓ Centrifuge to separate pore water from sediment
  - ✓ Centrifuge tubes
  - ✓ Photoperiod timers
  - ✓ Temperature controllers
  - ✓ Dissolved oxygen (D.O.) meter
  - ✓ Thermometer (°C)

- ✓ pH meter
- ✓ Light boxes
- ✓ Dessicators for holding dried organisms
- ✓ Drying oven (capable of 60°C temperature)
- ✓ Balance (0.01 mg resolution)
- ✓ Aeration system (if required due to low dissolved oxygen)
- ✓ Salinity/conductivity meter or refractometer
- ✓ ISE meter (Orion 720A or similar)
    - Ammonia sensitive electrode (Orion 95-12 or similar)
    - 0.1 M or 1000 ppm NH_3 standard, ionic strength adjuster

- Chemicals
    - ✓ Liquid soap (e.g., Liqui-Nox)
    - ✓ 10% nitric acid
    - ✓ Acetone
    - ✓ Ammonium chloride (reference test)
- Miscellaneous
    - ✓ Data sheets: live animal acclimation, water quality, survival, growth and reproduction
    - ✓ Labels for beakers
    - ✓ Safety equipment (e.g., gloves, lab coats, safety glasses, safety showers and eye fountains)

3.0 **Health, Safety and Waste Management**

3.1 Personal safety equipment should be used by all participating technicians due to the unknown nature of test sediment contamination.

3.1.1 Use gloves, laboratory coats, safety glasses and respirators where applicable

3.1.2 Manipulate sediment (as needed) and other chemicals (e.g., acids, volatile organic solvents) under fume hood

3.2 Laboratory safety equipment such as first aid kits, emergency showers, eye fountains must be available to workers

3.3 All sediment wastes should be captured in a labeled five gallon bucket (or similar container) during the breakdown and sieving of the toxicity test.

3.4 All containers (chemical and waste) must be properly labeled.

3.5 Sediment and chemical wastes should be held and disposed of in compliance with the participating laboratory's applicable state and federal laws.

4.0 **Receipt of sediment**

4.1 Sediment will be shipped to the ERDC in plastic buckets or other container as deemed appropriate based on the chemical class of the contaminant of concern. Sediment will be shipped at 4.0 ± 2°C. The temperature and other pertinent information (COC, etc.) should be recorded on arrival, sediment logged into the sediment storage log book and placed into the cold room at 4.0 ± 2°C until required for testing.

5.0 **Collection of 7-8 day old amphipods**

5.1 *Hyalella azteca* can be ordered at the correct age from a commercial vendor or collected from in-house cultures. If ordered from a commercial vendor, an organism receipt and acclimation form should be completed upon receipt of the amphipods and any deviations in the water quality ranges noted.

5.2 If temperature on arrival is greater than 3° C from the holding/testing temperature then the amphipods should be slowly acclimated to the correct temperature. Amphipods should be evaluated 24 hours after receipt for dead organisms. If mortality exceeds 10% then the amphipods should not be used for testing. Amphipods should be held for 24-48 hours before use in testing to ensure that injured and unhealthy organisms are not used. Amphipods should be fed the normal culture or testing food ration during holding.

5.3 If amphipods are collected from in-house cultures, amphipods to be used in testing are collected by placing adult amphipods on a #25 (710 μm) sieve containing pre-conditioned leaves. Place sieves in a larger holding container filled with reconstituted water. Provide gentle aeration. The following day, collect the neonates passing through the sieve into the holding container by gently shaking amphipods off the leaves. Count the neonates produced into a bowl/container containing fresh reconstituted water. Based on the count determine if enough neonates have been collected to start testing. A goal of 125% of total required should be collected. If not enough are produced in a single day, the process can be repeated and the neonates produced over a two day period can be combined. Adults are returned to the culture once enough neonates are collected. Starting weight of amphipods must be in the range of about 0.02 to 0.035 mg/organism. Amphipods should be fed the normal culture or testing food ration during holding. Water exchanges should be conducted every 3 days during holding.

6.0 **Test chamber and sediment preparation (day –1)**

6.1 Glass soap water/acetone/acid-washed 300 mL beakers containing mesh screen overflow holes will serve as test chambers. The number required is fourteen (14) replicate beakers: eight (8) replicates/beakers per treatment for biological endpoints, 3 for pore water chemistry analysis at day 7, and 3 for pore water chemistry during the week prior to day 28. All replicate beakers will contain *H. azteca*. Each beaker should be labeled with the treatment and replicate (e.g., A-N) designation.

6.2 Prior to test initiation, sediment should be homogenized thoroughly within the bucket or other suitable container using an electric drill or mixer equipped with a stainless steel shaft and prop/agitator. After ~5 minutes of thorough mixing, the sediment in the buckets should be manually mixed with a stainless steel spatula or HDPE scoop, involving complete turnover of the sediment (i.e., bringing the bottom to the top). Utilize the power mixer again and repeat until the sediment is thoroughly mixed, homogenous in texture/color and has an even distribution of water. Repeat this process for all sediments following decontamination of equipment (Section 4.3).

6.3 Decontamination: The homogenization equipment (e.g., mixer, spatula) should be thoroughly decontaminated between the mixing of each sediment. This includes a (1) water rinse, (2) soap water scrubbing, (3) rinse with 10% nitric acid, (4) rinse with acetone and (5) deionized water wash. Rinses must be thorough to ensure all acid/solvent is removed and soaking glassware overnight in deionized water is an option.

6.4 Beakers will be filled with 100 mL (~ 2 cm) of homogenized sediment (as described in section 6.2) from each site, control and reference using a decontaminated stainless steel or HDPE scoop/spatula. The sediment should be added carefully to minimize contact with the sides of the beakers and leveled by tapping the side of the beakers.

6.5 Slowly add the overlaying reconstituted water created using methods specified in Borgmann (1996) but modified to contain 0.4 mg/L bromide using a turbulence reducer to minimize resuspension of the sediment. Beakers should be filled to the overflow holes on the beakers.

6.6 Arbitrarily place all treatment replicates in the specified Zumwalt or modified Brunson-Zumwalt water exchange system. The water exchange system should be in a controlled environmental chamber at 23 ± 1°C. No aeration is provided unless DO drops below minimum requirements (2.5 mg/L) during testing.

6.7 Porewater characterization is conducted on day -1 or shortly after sediment receipt. Place 50 mLs of sediment into a 50 mL centrifuge tube. Centrifuge at 2500 g for 20 minutes. Decant supernatant and measure temperature, pH, and total ammonia on the supernatant. Porewater for chemistry analysis is collected in an identical manner at day-1 using centrifuge bottles of appropriate volume.

7. Test initiation (day 0)

7.1 A water addition should be conducted on day 0 prior to measuring parameters and adding organisms.

7.2 Water quality measurements (temperature, pH, dissolved oxygen (DO), hardness, alkalinity, conductivity, and total ammonia) will be taken in all chambers and recorded. Overlying water total ammonia will be taken in one replicate per treatment and recorded. If parameters are within the specified ranges (US EPA 2000), the test may be initiated.

7.3 *Hyalella azteca* (7-8 days old) will be placed in a culture bowl or counting tray. Ten organisms should be randomly counted into HDPE cups or glass 50 mL beakers so that initial and QA-QC counts are manageable. Organism transfer should be carried out using wide-bore pipettes, fully submerging the pipettes to minimize injury (forceps are not to be used). Only apparently healthy individuals should be selected at random for testing. Any organisms that are dropped or contact a dry surface cannot be used in testing. After the appropriate number of counting-containers is filled, a technician should check to ensure each container has 10 individuals. Include an additional 8 counting chambers (80 organisms) for initial weight measurements. A second technician should be present to ensure that only 10 organisms are added to each chamber and that all individuals are removed from the counting beakers. Squirt bottles containing fresh filtered/dechlorinated water can be used to dislodge amphipods that are stuck to the sides of the counting chambers.

Special attention should be paid to floating amphipods, which should be submerged using a drop of test water from the test beaker using a pipette. Load all A replicates first with the sediment treatments randomized. Repeat the process for the remaining replicates.

7.4 Upon addition of test organisms, the time of test initiation must be recorded on the appropriate data sheet.

7.5 Organisms in each beaker are provided fed 1.0 mL of YCT (1800 mg/L stock) to each test chamber.

7.6 Approximately one hour following the addition of test organisms, chambers should be observed for injured or unhealthy amphipods. At this time, injured or unhealthy individuals or individuals may be replaced if the response does not appear to be specific to the particular sediment treatment. Floating amphipods should be submerged using a drop of test water from the test beaker using a pipette.

8. Test monitoring and maintenance (days 1 – 27)

8.1 Test chambers should be observed twice daily for floating amphipods during the first week. After the first week they should be observed once daily. Any amphipods observed floating should be submerged using water droplets dropped gently from a pipette.

8.2 Two overlying water additions/day will be conducted using the automated Brunson-Zumwalt system or manually using the Zumwalt delivery system. Water quality parameters (temperature and DO) will be measured daily in a replicate of each treatment prior to water exchange. Dissolved oxygen will be measured more frequently if it decreases more than 1 mg/L in a treatment since the previous measurement. Aeration will be provided if DO drops below 2.5 mg/L.

8.3 Organisms in each beaker are fed 1 ml of YCT/day (from a 1800 mg solids/L stock) on days 0-13, and 2 ml/day will be added daily to each test chamber during the remainder of the exposure

8.4 Record exposure system temperature daily.

8.5 Check light cycle and ensure that each test chamber is adequately aerated daily.

9. Test termination/breakdown (day 28)

9.2 Water quality: temperature, pH, and DO, alkalinity, hardness, conductivity, and total ammonia are measured in a composite sample collected from all replicates of a sediment treatment and recorded. Survival endpoint determination

9.2.1 Gently pour off all but 50 mL of overlying water through an 8 or 12-inch diameter 425 μM ASTM testing sieve to isolate larvae. Swirl and suspend sediment in the remaining overlying water for easier passing of sediment through the sieves. If using an 8 inch sieve, a 12-inch diameter sieve (1 mm mesh) can be placed over the bucket receiving the waste to support the 8 inch sieve.

9.2.2 Transfer small amounts of the material retained on each sieve to a counting bowl or tray for examination. Count surviving amphipods. All missing individuals or amphipods that fail to move following close observation (e.g., under a dissection

microscope) and gentle prodding with a blunt probe are considered dead. Individuals exhibiting any movement are counted as alive.

9.2.3 Surviving amphipods from each replicate beaker are placed on pre-dried, pre-weighed pans and placed in a drying oven at 60 °C for 24 hours. After 24 hours, pans are placed in a desiccator and allowed to cool. Dry weights are recorded for each pan. Dry weight is then determined by the following formula:

(amphipod and pan dry weight – pan dry weight) = amphipod dry weight

9.2.4 Biomass will be reported as average final mass of each sediment replicate. Weight is reported as the average individual dry weight for a sediment by dividing total biomass of each replicate by the number of surviving organisms within the replicate then calculating the mean value.

10. **Acceptability**

10.1 Test acceptability requirements include:

10.1.1 Tests must be started with 7-8 day old amphipods.

10.1.2 80% or greater average survival in the control

10.1.3 Hardness, alkalinity, and ammonia in the overlying water typically should not vary by more than 50% during the test, and dissolved oxygen should be maintained above 2.5 mg/L in the overlying water.

10.1.4 Mean weight of *H. azteca* in the control sediment on Day 28 should be greater than or equal to 0.4 mg dry/individual.

11. Recording data

11.1 All specified data should be recorded in full on the appropriate data sheets (i.e., organism acclimation, water quality, survival, reproduction).

11.2 If any parameters are not within the specified range, record on a datasheet, make a note in the comments and contact study coordinator.

References

ASTM International. 2010. Standard test method for measuring the toxicity of sediment-associated contaminants with estuarine and marine invertebrates. ASTM, 1706-05. ASTM International, West Conshohocken, PA.

Borgmann 1996. Systematic analysis of aqueous ion requirements of *Hyalella Azteca*: A standard artificlial medium including the essential bromide ion. *Arch. Environ. Contam. Toxicol.* 30:356-363.

US EPA. 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates. EPA-600-R99-064. US EPA. Washington, DC.

USEPA/USACE 1998. US Environmental Protection Agency / US Army Corps of Engineers. Evaluation of Material Proposed for Discharge to Waters of the U.S. - Testing Manual (Inland Testing Manual). EPA-823-B-98-004, Office of Water, Washington DC.

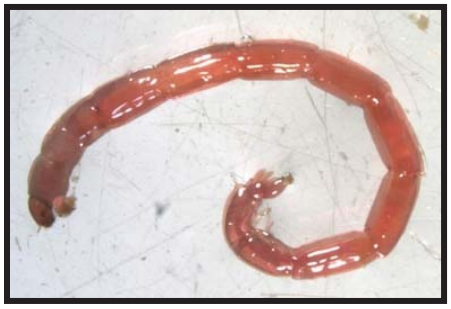
Table A.2-1. Summary of Conditions for 28-d sediment toxicity test using *Hyalella azteca*

Description	Condition
Test Type	Static renewal
Test Duration	28 days
Temperature	23.0 ±1.0°C
pH	6.5-9
Light Quality	Wide spectrum fluorescent lights
Light Intensity	About 100-1000 lux
Photoperiod	16:8 L:D
Test Chamber Size	300 mL (with screens)
Sediment Depth/volume	2 cm (100 mL)
Overlying Water Volume	Fill to bottom of screens
Sediment settling time	Overnight
Renewal of Overlying water	Two volume additions daily. Water is reconstituted water created using methods specified in Borgmann (1996) but modified to contain 0.4 mg/L bromide
Age of Test Organisms	7-8 days old (starting weight must be in the range of about 0.02 to 0.035 mg/organism)
Organisms/chamber	10
Replicates/treatment	8 (plus 6 chemistry only replicates)
Organisms/treatment	80
Feeding Regime	1 ml of YCT/day (from a 1800 mg solids/L stock) on days 0-13, and 2 ml/day during the remainder of the exposure
Test Chamber Cleaning	Clean screens as required with small brush
Test Solution Aeration	None unless DO drops below 2.5 mg/L; Trickle flow (<100 bubbles/min); >60% saturation
Dilution Water	Reconstituted water created using methods specified in Borgmann (1996) but modified to contain 0.4 mg/L bromide
Test Concentrations	Site sediment, reference, control
Endpoint(s)	Survival, biomass, and dry weight

Table A.2-2. Summary of Tasks for 28-d sediment toxicity test using *Hyalella azteca*

Day	Task
-7	Collect neonates for testing. Maintain at test temperature and feed YCT during holding period
-1	Sample pore water by centrifugation for porewater water quality analysis and chemistry analysis. Homogenize sediment in shipping bucket or other container appropriate for contaminant(s). Fill test chambers with sediment and add overlying water.
0	Test initiation: Record temperature, pH, D.O., alkalinity, hardness, conductivity, and total ammonia on a composite sample of all replicates from a sediment treatment. Add 10 amphipods per chamber. Collect 80 amphipods for initial weight determination. Place peepers in sediment chemistry replicates.
1-9	Perform exposure chamber observations. Measure temperature and DO daily in a composite sample collected from replicates of a sediment treatment. Provide each chamber with 1 mL of YCT (1800 mg/L stock solution). Perform twice daily water renewals.
7	Collect day 7 peepers and sediment pore water from chemistry replicates.
14	Increase feeding rate to 2 mL YCT per replicate
14-20	Place peepers in second set of chemistry replicates.
21-27	Collect day 14-20 peepers and sediment pore water from chemistry replicates.
28	Test termination: Record temperature, pH, D.O. alkalinity, hardness conductivity and total ammonia from a composite sample collected from all replicates of a sediment treatment.
29	Place pans in desiccator to cool and record pan weights.

Appendix A.3 - Protocol for Conducting Life Cycle Sediment Toxicity Tests Using *Chironomus dilutus*



Testing Protocol Provided by:

US Army Engineer Research and Development Center (ERDC)

Environmental Laboratory, EP-R

3909 Halls Ferry Rd

Vicksburg, MS 39180

Ecotoxicology and Environmental Risk Team

Team Leader:

Dr. Jacob Stanley
Tel: 601-634-3544
Jacob.K.Stanley@usace.army.mil

Mr. J. Daniel Farrar
Tel: 601-634-2118
Daniel.Farrar@usace.army.mil

Mr. Alan J. Kennedy
Tel: 601-634-3344
Alan.J.Kennedy@usace.army.mil

1. Testing Procedure Overview

The protocol to follow provides guidance for conducting chronic (~64 day) sediment toxicity tests using the freshwater midge *Chironomus dilutus.* The protocol is in basic accordance with the guidance provided in "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates" (EPA-600-R99-064) and "Standard Test Method for Measuring the Toxicity of Sediment-Associated
Contaminants with Freshwater Invertebrates" (ASTM, 1706-05 (2010)).

2. Required Materials for Preparation and Testing

- Biological testing material
  - ✓ *Chironomus dilutus* larvae (4-d old)
  - ✓ Control, reference, and site sediment
  - ✓ Reconstituted fresh water prepared as described in USEPA (2000)
  - ✓ Environmental chamber with temperature and photoperiod control
  - ✓ Tetramin® Fish Flakes (ground to 500 µm particle size)
- Glassware and accessories
  - ✓ Twenty-five (25) replicate toxicity beakers per sediment (16 for biological endpoints and 3 each for chemistry analysis at day 7, sometime between days 21 and 27, and again between days 42 and 49) prepared for use in a Zumwalt or modified Brunson-Zumwalt water exchange system (US EPA 2000)
  - ✓ Test initiation/maintenance/breakdown materials
    - Transfer pipettes
    - Turbulence reducers (e.g., nylon, Teflon or polyethylene disks attached to a rod)
    - Eight inch diameter #40 sieves (425 µm mesh sizes)
    - Plastic wash bottles
    - Type I water for probe rinsing, etc.
    - Talley counters (optional)
    - Pans or bowls for counting
    - Heavy duty aluminum foil pans for initial and final weight determination.
- Instruments and equipment
  - ✓ Electric drill/mixer with stainless steel mixing rod
  - ✓ Zumwalt or modified Brunson-Zumwalt water exchange system (US EPA 2000)
  - ✓ Stainless steel or high density polyethylene (HDPE) spoon/mixing rods
  - ✓ Centrifuge to separate pore water from sediment
  - ✓ Centrifuge tubes/bottles
  - ✓ Photoperiod timers
  - ✓ Temperature controllers
  - ✓ Dissolved oxygen (D.O.) meter
  - ✓ Thermometer (°C)

- ✓ pH meter
- ✓ Light boxes
- ✓ Dessicators for holding dried organisms
- ✓ Drying oven (capable of 60 °C temperature)
- ✓ Emergence traps
- ✓ R/O chambers (reproduction/oviposition)
- ✓ Dissecting microscopes
- ✓ Muffle furnace (capable of 550 °C temperature)
- ✓ Balance (0.01 mg resolution)
- ✓ Aeration system (if required due to low dissolved oxygen)
- ✓ Salinity/conductivity meter or refractometer
- ✓ ISE meter (Orion 720A or similar)
  - Ammonia sensitive electrode (Orion 95-12 or similar)
  - 0.1 M or 1000 ppm NH_3 standard, ionic strength adjuster

- Chemicals
  - ✓ Liquid soap (e.g., Liqui-Nox)
  - ✓ 10% nitric acid
  - ✓ Acetone
  - ✓ Ammonium chloride (reference test)
- Miscellaneous
  - ✓ Data sheets: live animal acclimation, water quality, survival, growth and reproduction
  - ✓ Labels for beakers
  - ✓ Safety equipment (e.g., gloves, lab coats, safety glasses, safety showers and eye fountains)

3. Health, Safety and Waste Management

3.1. Personal safety equipment should be used by all participating technicians due to the unknown nature of test sediment contamination.

3.1.1. Use gloves, laboratory coats, safety glasses and respirators where applicable

3.1.2. Manipulate sediment (as needed) and other chemicals (e.g., acids, volatile organic solvents) under fume hood

3.2. Laboratory safety equipment such as first aid kits, emergency showers, eye fountains must be available to workers

3.3. All sediment wastes should be captured in a labeled five gallon bucket (or similar container) during the breakdown and sieving of the toxicity test.

3.4. All containers (chemical and waste) must be properly labeled.

3.5. Sediment and chemical wastes should be held and disposed of in consultation with the customer and in compliance with the participating laboratory's applicable state and federal laws.

4. Receipt of sediment

4.1. Sediment will be shipped to the ERDC in plastic buckets or other container as deemed appropriate based on the chemical class of the contaminant of concern. Sediment will be shipped at 4.0 ± 2°C. The temperature and other pertinent information (COC, etc.) should be completed on arrival, sediment logged into sediment storage record book and placed into a locked cold room at 4.0 ± 2°C until required for testing.

5. Receipt and Hatching of Egg Masses (Day -3 and Day -6)

5.1. Order the required number of egg masses to conduct the test 2 days prior to testing. Order approximately half that number of egg masses to arrive 6 days later to be used as auxiliary males.

5.2. Egg masses are obtained from a commercial vendor (e.g., Environmental Consulting and Testing (Superior, WI) and shipped overnight to the ERDC. Egg masses should be shipped in the appropriate water quality conditions (temperature 23 ± 5°C, D.O. ≥ 4.0 mg/L). An organism receipt and acclimation form should be completed upon receipt of the egg masses and any deviations in the water quality ranges noted.

5.3. The temperature of the egg masses are brought to 23 °C and are placed in 4 inch culture bowls or crystallizing dishes containing fresh water. The dishes are placed in an environmental chamber at 23 °C and under 16:8 Light:Dark cycle.

5.4. Eggs will begin to hatch in approximately 48 hours with most larvae leaving the egg case in 24 hours. The test is initiated with 4-d old larvae. When hatching is observed, transfer the egg case to a new dish so know age animals can be collected. Tetramin® is provided to each dish by mixing the food with fresh water (300 mg/mL). The slurry is allowed to settle and the fine food material is delivered to each dish just enough to lightly cover the bottom of the dish. The larvae will use the food as a tube building substrate which will facilitate the addition of larvae to test replicates. This process is repeated for the auxiliary male egg masses received later.

6. Test chamber and sediment preparation (day –1 sediment addition)

6.1. Glass soap water/acetone/acid-washed 300 mL beakers containing mesh screen overflow holes will serve as test chambers. The number required is 16 replicates/beakers per treatment plus 9 chemistry-only beakers per treatment. Four replicates will be used for auxiliary males. Each beaker should be labeled with the treatment and replicate (e.g., A-Y) designation.

6.2. Prior to test initiation, sediment should be homogenized thoroughly within the bucket or other suitable container using an electric drill or mixer equipped with a stainless steel shaft and prop/agitator. After ~5 minutes of thorough mixing, the sediment in the buckets should be manually mixed with a stainless steel spatula or HDPE scoop, involving complete turnover of the sediment (i.e., bringing the bottom to the top). Utilize the power mixer again and repeat until the sediment is thoroughly mixed, homogenous in texture/color and has an even distribution of water. Repeat this process for all sediments following decontamination of equipment (Section 5.3).

6.3. Decontamination: The homogenization equipment (e.g., mixer, spatula) should be thoroughly decontaminated between the mixing of each sediment. This includes a (1) water rinse, (2) soap water scrubbing, (3) rinse with 10% nitric acid, (4) rinse with acetone[2] and (5) deionized water wash. Rinses must be thorough to ensure all acid/solvent is removed and soaking glassware overnight in deionized water is an option. Beakers will be filled with 100 mL (~ 2 cm) of homogenized sediment (as described in section 5.2) from each site, control and reference using a decontaminated stainless steel or HDPE scoop/spatula. The sediment should be added carefully to minimize contact with the sides of the beakers and leveled by tapping the side of the beakers.

6.4. Slowly add the overlaying reconstituted water using a turbulence reducer to minimize resuspension of the sediment. Beakers should be filled to the overflow holes on the beakers.

6.5. Arbitrarily place all treatment replicates in the specified Zumwalt or modified Brunson-Zumwalt water exchange system. The water exchange system should be in a controlled environmental chamber at 23 ± 1°C. No aeration is provided unless DO drops below minimum requirements (2.5 mg/L) during testing.

6.6. Porewater characterization is conducted on day -1 or shortly after sediment receipt. Centrifuge at 2500 g for 30 minutes. Decant supernatant and measure temperature, pH, and total ammonia on the supernatant. Porewater for chemistry analysis is collected in an identical manner at day-1 using centrifuge bottles of appropriate volume.

7. Test initiation (day 0)

7.1. A 100% water volume addition should be conducted on test day 0 prior to measuring parameters and adding organisms.

7.2. Water quality measurements (temperature, pH, and dissolved oxygen (DO), hardness, alkalinity conductivity and total ammonia) will be taken in on a composite sample collected from the replicates of each sediment and recorded. If parameters are within the specified ranges (US EPA 2000), the test may be initiated. If DO drops below 2.5 mg/L, then aeration should be implemented.

7.3. *Chironomus dilutus* larvae (4 d old) will be counted directly into each exposure beaker. A dissecting microscope should be used to ensure no larvae are injured during the transfer process. Beakers can be removed from the water exchange system to facilitate organism addition. Twelve organisms should be randomly counted into each exposure beaker by replicate (i.e., count into all replicate A beakers, then replicate B beakers, etc.) Organism transfer should be carried out using plastic disposable pipettes with the tip cut to the appropriate size to pipette larvae and food tubes. Fully submerge the pipettes near the sediment surface when transferring larvae. Effort should be made to transfer larvae within their food tubes, ensuring that only 1 larvae is in the food tube, to minimize injury. Only apparently healthy individuals should be selected for testing. Any

[2] Attention should be given to whether all equipment is stainless steel or HDPE before using acetone rinses due to concern regarding chemical release (e.g., phthalates from plastics).

organisms that are dropped or that contact a dry surface cannot be used in testing. Special attention should be paid to floating larvae immediately after all animals have been added to exposure chambers. Floating larvae should be removed and replaced with a healthy larvae. No water exchanges should be conducted for 8 hours following larvae addition to test replicates.

7.4. Upon addition of test organisms, the time of test initiation must be recorded on the appropriate data sheet.

7.5. Organisms in each beaker are provided 1.5 ml of water/food slurry containing 6 mg of Tetramin®.

7.6. Approximately one hour following the addition of test organisms, chambers should be observed for floating larvae. At this time floating individuals may be replaced if the response does not appear to be specific to the particular sediment treatment.

8. **Auxiliary Male Production**

8.1. For each test treatment, prepare the remaining 4 replicates to provide auxiliary males, repeating the steps described in Sections 6.0, above.

8.2. On Day 6 of the test, incubate new egg cases as in Section 5, above.

8.3. On Days 7-10 of the test, allocate new larval chironomids into each of the "Auxiliary Male" replicates, repeating the steps described in Sections 7, above.

9. Test Monitoring and Maintenance (Day 1-20)

9.1. Test chambers should be observed twice daily for floating larvae during the first week. After the first week they should be observed once daily. Any larvae observed floating (24 hours after test initiation) should be submerged using water droplets dropped gently from a pipette.

9.2. Water additions (2 volume additions of overlying water per day) will be conducted twice daily using the automated Brunson-Zumwalt system or manually using the Zumwalt delivery system.

9.3. Water quality parameters (temperature and DO) will be measured daily in a replicate of each treatment prior to water exchange. Ever Monday, Wednesday and Friday pH should be measure on the composite. Conductivity should be measured once weekly. Dissolved oxygen will be measured more frequently if it decreases more than 1 mg/L in a treatment since the previous measurement. Aeration will be provided if DO drops below 2.5 mg/L.

9.4. Organisms in each beaker are provided 1.5 ml of water/food slurry containing 6 mg of Tetramin® daily following water quality parameters.

9.5. Record exposure system temperature daily.

9.6. Check light cycle and ensure that each test chamber is adequately aerated (if required) daily.

10. Day 20

10.1. Survival, mean dry weight and ash-free dry weight (AFDW) are determined for 4 replicates at day 20.

10.2. Measure temperature in one randomly selected replicate of each sediment treatment.

10.3. Survival endpoint determination

10.3.1. Gently pour off all but 50 mL of overlying water through an 8 or 12-inch diameter 425 µM sieve to isolate larvae. Swirl and suspend sediment in the remaining overlying water for easier passing of sediment through the sieves. If using an 8 inch sieve, a 12-inch diameter sieve (1 mm mesh) can be placed over the bucket receiving the waste to support the 8 inch sieve.

10.3.2. Transfer small amounts of the material retained on each sieve to a counting bowl or tray for examination. Count and record surviving larvae. All missing individuals or larvae that fail to move following close observation (e.g., under a dissection microscope) and gentle prodding with a blunt probe are considered dead. Individuals exhibiting any movement are counted as alive.

10.3.3. Organisms that have pupated are counted as live but are not used for weight and biomass determination.

10.3.4. Surviving larvae from each replicate beaker are placed on ashed pre-weighed pans and placed in a drying oven at 60 °C for 24 hours. After 24 hours, pans are placed in a desiccator and allowed to cool (4 hours). Dry weights are recorded for each pan. Pans are then placed into a muffle furnace at 550 °C for 2 hours. After 2 hours of ashing, pans are placed in a desiccator to cool (4 hours). Once cool pans are weighed and ash weight is recorded. Ash-free dry weight is then determined by the following formula:(Dry weight – pan weight) - (Ash-free weight – pan weight)

10.3.5. Biomass will be reported as average final mass of each sediment replicate. Weight is reported as the average individual ash-free dry weight for a sediment by dividing total biomass of each replicate by the number of surviving organisms within the replicate then calculating the mean value.

10.3.6. Place emergence traps on remaining exposure chambers.

11. **Test Monitoring and Maintenance (Day 21 to test end)**

11.1. Prepare a reproduction/oviposition (R/O) chamber for each of the 8 remaining initial test replicates.

11.2. On a daily basis, record emergence of males and females, pupal and adult mortality, and time-to-death for previously collected adults. Two categories are recorded for emergence: complete emergence and partial emergence. Complete emergence occurs when an organism has shed the pupal exuviae completely. Partial emergence occurs when an organism only partially sheds the pupal exuviae. Partially-emerged adults will eventually die. Some adults will get trapped in the surface tension of the water and die. Record time-to-death for both partial emerged and trapped individuals. Trapped organisms usually die within 24 hours so 24 hour time to death is generally recorded.

11.3. On a daily basis transfer the completely emerged adults from each replicate to the corresponding R/O chamber for that replicate. These adults will begin to mate and produce egg cases.

11.4. Observe each R/O chamber daily for the presence of egg cases and dead adults. Record time to death for dead adults.

11.5. Female chironomids are capable of laying multiple egg cases: a primary egg case (typically large and C-shaped) and an occasional secondary egg case which is much smaller. Only primary egg cases are evaluated. Transfer each egg case from the R/O chamber to a corresponding labeled (so as to identify the egg transfer date) 60- x 15-mm plastic Petri dish containing ~15 mL of test water to monitor incubation and hatch.

11.6. For each transferred primary egg case estimate the number of eggs in the egg case using the ring method. When the integrity of the egg case precludes use of the ring method (i.e., the egg case is convoluted or distorted), the eggs should be enumerated using the direct count method (note that if the direct count method is used, hatchability data will not be obtained for that particular egg case).

11.6.1. **Ring Method**: (1) for each egg case, the mean number of eggs in five rings is determined; (2) these rings should be selected at about equal distances along the length of the egg case; (3) the number of eggs/ring multiplied by the number of number of rings in the egg case will provide an estimate of the total number of eggs. The ring method is best suited to the "C" shaped egg cases.

11.6.2. **Direct Method**: Each egg case is placed into a 5-cm glass culture tube containing 2 mL of 2 N sulfuric acid (H_2SO_4) and left overnight. After digestion, the eggs are collected with a Pasteur pipette and spread across a microscope slide or Petri plate with a grid for counting under a dissecting microscope. The direct count method does not permit determination of hatching success.

11.7. Record the number of dead adults, number of egg cases oviposited, and number of eggs produced daily. Determine successful hatch rate for each egg case. Although the time required to initiate hatching at this temperature is about 2 d, the period of time required to bring about complete hatch may be as long as 6 d. Therefore, hatching success is determined after 6 d of incubation. After 6 days of incubation, determine the number of eggs that remain unhatched. Unhatched eggs either remain in the gelatinous egg case or are distributed on the bottom of the Petri dish. Subtract the number of unhatched eggs from the total number of eggs originally estimated for that egg case. The "% Successful Hatch" is then calculated as that difference divided by the total number of eggs estimated for that particular egg case.

12. **Day 28**

12.1. Place emergence traps on the auxiliary male replicate beakers.

13. **Day 28 – Test End**

13.1. Transfer males emerging from the auxiliary male replicates to individual R/O chambers. The auxiliary males are used for mating with females from the same test treatment for which there are an insufficient number of males. For each R/O chamber lacking a live male or in which a female has not oviposited an egg case within three days, add an auxiliary male to the R/O chamber.

14. **Test Termination Day 56-Day 64**

14.1. After 7 days during which no emergence is observed in any of the 8 replicates for a given sediment treatment, the replicates at that treatment can be terminated.

14.2. Determine the temperature within one randomly-selected replicate beaker at the test treatment(s). Measure water quality (pH, D.O., and conductivity) on a composite sample for each treatment.

14.3. Process each of the 8 replicates as described for day 20 (Section 10, above).

15. **Test Acceptability Criteria**

15.1. Average size of larva in control sediment at day 20 must be at least 0.6 mg dry weight/surviving organisms or 0.48 AFDW per surviving organism.

15.2. Control emergence should be greater than or equal to 50%. Pupae survival is typically >83% and adult survival is >96%.

15.3. Control time to death after emergence is <6.5 d for males and <5.1 d for females.

15.4. Control mean number of eggs/ egg case should be greater than or equal to 800 and the percent hatch should be greater than or equal to 80%.

16. Recording Data

16.1. All specified data should be recorded in full on the appropriate data sheets (i.e., organism acclimation, water quality, survival, reproduction).

16.2. If any parameters are not within the specified range, record on a datasheet, make a note in the comments and contact study coordinator.

17. References

ASTM International. 2010. Standard test method for measuring the toxicity of sediment-associated contaminants with estuarine and marine invertebrates. ASTM, 1706-05. ASTM International, West Conshohocken, PA

US EPA. 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates. EPA-600-R99-064. US EPA. Washington, DC.

USEPA/USACE 1998. US Environmental Protection Agency / US Army Corps of Engineers. Evaluation of Material Proposed for Discharge to Waters of the U.S. - Testing Manual (Inland Testing Manual). EPA-823-B-98-004, Office of Water, Washington DC.

Table A.3-1 Summary of Conditions for Life Cycle sediment toxicity test using *Chironomus dilutus*

Description	Condition
Test Type	Static renewal
Test Duration	~64 days
Temperature	23.0 ±1.0°C
pH	6.5-9
Light Quality	Wide spectrum fluorescent lights
Light Intensity	About 100-1000 lux
Photoperiod	16:8 L:D
Test Chamber Size	300 mL (with screens)
Sediment Depth/volume	2 cm (100 mL)
Overlying Water Volume	Fill to bottom of screens
Sediment settling time	Overnight
Renewal of Overlying water	Two 100% water volume additions daily. Water is moderately hard reconstituted water created using methods specified in USEPA (2000)
Age of Test Organisms	4 d old
Organisms/chamber	12
Replicates/treatment	25 (16 replicates for biological analysis and 9 replicates for chemistry analysis)
Organisms/treatment	300 (192 in replicates for biological analysis and 108 in replicates for chemistry analysis)
Feeding Regime	1.5 ml of water/food slurry containing 6 mg of Tetramin® daily into each chamber
Test Chamber Cleaning	Clean screens as required with small brush
Test Solution Aeration	None unless DO drops below 2.5 mg/L; Trickle flow (<100 bubbles/min); >60% saturation
Dilution Water	Moderately hard reconstituted water created using methods specified in USEPA (2000)

Test Concentrations	Site sediment, reference, control
Endpoint(s)	% Survival, biomass, dry weight and ash-free dry weight after 20 days; female and male emergence, adult mortality (time to death), number of eggs produced/egg case, number of eggs hatched, % hatch.

Table A.3-2. Summary of Tasks for life cycle sediment toxicity test using *Chironomus dilutus*

Day	Task
-3	Receive egg masses and place in culture bowls/crystalizing dishes
-1	Eggs should begin hatching; provide food as described in protocol. Homogenize sediment in shipping bucket or other container appropriate for contaminant(s). Collect pore water samples for chemical analysis via centrifugation. Measure porewater water quality characteristics if not performed earlier. Fill test chambers with sediment and add overlying water.
0	Test initiation: Record temperature, pH, D.O. alkalinity, hardness, conductivity and total ammonia from a composite overlying water sample from all replicates of a sediment treatment. Transfer 12 larvae to each test chamber. Feed 6 mg of Teftrafin® in a water slurry per replicate. Place first set of peepers into chemistry replicates.
1-End	Record temperature and DO daily. Aerate if DO drops below 2.5 mg/L. Provide each chamber with 6 mg of Tetramin® in a water slurry. Perform two volume additions per day.
7	Collect peepers and sediment porewater from chemistry replicates
7-10	Setup auxiliary male beakers (4 replicates/treatment)
14-20	Place second set of peepers in chemistry replicates
20	Terminate 4 replicates from sediment each treatment for determination of larvae survival, dry weight and ash –free dry weight. Place surviving organisms on pans. Install emergence traps on remaining replicates. Measure overlying water quality prior to termination.
21	Weigh pans for dry weight determination and place in muffle furnace. Weigh pans for ash-free dry weight determination.
21-27	Collect second set of peepers and sediment porewater from chemistry replicates
21-Test End	On a daily basis, record emergence of males and females, pupal, and adult mortality, and time to death for previously collected adults. Each day, transfer adults from each replicate to a corresponding

	reproduction/oviposition (R/O) chamber. Transfer each primary egg case from the R/O chamber to a corresponding Petri dish to monitor incubation and hatch. Record each egg case oviposited, number of eggs produced (using either the ring or direct count methods), and number or hatched eggs.
28	Place emergence traps on auxiliary male beakers
33-Test End	Transfer emerging auxiliary males to (R/O chambers). Use for mating with females of corresponding treatment if enough males are not produced in the primary test beakers.
33-42	Place third set of peepers in chemistry beakers
42-49	Collect third set of peepers and sediment porewater from chemistry replicates
40- Test End	After 7 days with no recorded emergence in the control sediment, terminate the study by recovering larva, pupae or exuviae. Measure overlying water quality prior to termination.

Appendix A.4 Protocol for Conducting 42-d Sediment Toxicity Tests Using *Hyalella Azteca*

Testing Protocol Provided by:

US Army Engineer Research and Development Center (ERDC)

Environmental Laboratory, EP-R

3909 Halls Ferry Rd

Vicksburg, MS 39180

Ecotoxicology and Environmental Risk Team

Team Leader:

Dr. Jacob Stanley
Tel: 601-634-3544
Jacob.K.Stanley@usace.army.mil

Mr. J. Daniel Farrar
Tel: 601-634-2118
Daniel.Farrar@usace.army.mil

Mr. Alan J. Kennedy
Tel: 601-634-3344
Alan.J.Kennedy@usace.army.mil

1. Testing Procedure Overview

The protocol to follow provides guidance for conducting 28-d sediment toxicity tests using the freshwater amphipod *Hyalella azteca.* The protocol is in basic accordance with the guidance provided in "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates" (EPA-600-R99-064) and "Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates" (ASTM, 1706-05, (2010)).

2. Required Materials for Preparation and Testing

- Biological testing material
  - ✓ *Hyalella azteca* (7-8 days old)
  - ✓ Control, reference and site sediment
  - ✓ Reconstituted water prepared as described in Borgmann (1996) modified to contain 0.4 mg/L bromide.
  - ✓ Environmental chamber with temperature and photoperiod control
  - ✓ YCT (yeast, Cerophyl® and trout chow) prepared as described in Appendix B of EPA600/R/R-99/064 (US EPA 2000).
- Glassware and accessories
  - ✓ Testing chambers (Eight 300 mL beakers per treatment) prepared for use in a Zumwalt or modified Brunson-Zumwalt water exchange system (US EPA 2000)
  - ✓
  - ✓ Test initiation/maintenance/breakdown materials
    - Transfer pipettes
    - Turbulence reducers (e.g., nylon, Teflon or polyethylene disks attached to a rod)
    - 12 inch diameter #25 sieves (710 μm mesh size size) for collection of known age neonates
    - 12 or 8 inch diameter #25 sieves (425 μm mesh sizes) for test termination
    - Plastic wash bottles
    - Type I water for probe rinsing, etc
    - Talley counters (optional)
    - Pans or bowls for counting
    - Aluminum pans for initial and final weight determination
    - Nitex screen (3x3 cm squares)

- Instruments and equipment
  - ✓ Electric drill/mixer with stainless steel mixing rod
  - ✓ Zumwalt or modified Brunson-Zumwalt water exchange system (US EPA 2000)
  - ✓ Stainless steel or high density polyethylene (HDPE) spoon/mixing rods
  - ✓ Centrifuge to separate pore water from sediment
  - ✓ Centrifuge tubes
  - ✓ Photoperiod timers
  - ✓ Temperature controllers
  - ✓ Dissolved oxygen (D.O.) meter

- ✓ Thermometer (°C)
- ✓ pH meter
- ✓ Light boxes
- ✓ Dessicators for holding dried organisms
- ✓ Drying oven (capable of 60°C temperature)
- ✓ Balance (0.01 mg resolution)
- ✓ Aeration system (if required due to low dissolved oxygen)
- ✓ Salinity/conductivity meter or refractometer
- ✓ ISE meter (Orion 720A or similar)
    - Ammonia sensitive electrode (Orion 95-12 or similar)
    - 0.1 M or 1000 ppm NH_3 standard, ionic strength adjuster

- Chemicals
    - ✓ Liquid soap (e.g., Liqui-Nox)
    - ✓ 10% nitric acid
    - ✓ Acetone
    - ✓ Ammonium chloride (reference test)
- Miscellaneous
    - ✓ Data sheets: live animal acclimation, water quality, survival, growth and reproduction
    - ✓ Labels for beakers
    - ✓ Safety equipment (e.g., gloves, lab coats, safety glasses, safety showers and eye fountains)

7.0 **Health, Safety and Waste Management**

7.1 Personal safety equipment should be used by all participating technicians due to the unknown nature of test sediment contamination.

7.1.1 Use gloves, laboratory coats, safety glasses and respirators where applicable

7.1.2 Manipulate sediment (as needed) and other chemicals (e.g., acids, volatile organic solvents) under fume hood

7.2 Laboratory safety equipment such as first aid kits, emergency showers, eye fountains must be available to workers

7.3 All sediment wastes should be captured in a labeled five gallon bucket (or similar container) during the breakdown and sieving of the toxicity test.

7.4 All containers (chemical and waste) must be properly labeled.

7.5 Sediment and chemical wastes should be held and disposed of in compliance with the participating laboratory's applicable state and federal laws.

8.0 **Receipt of sediment**

8.1 Sediment will be shipped to the ERDC in plastic buckets or other container as deemed appropriate based on the chemical class of the contaminant of concern. Sediment will be shipped at 4.0 ± 2°C. The temperature and other pertinent information (COC, etc.) should be recorded on arrival, sediment logged into the sediment storage log book and placed into the cold room at 4.0 ± 2°C until required for testing.

9.0 **Collection of 7-8 day old amphipods**

9.1 *Hyalella azteca* can be ordered at the correct age from a commercial vendor or collected from in-house cultures. If ordered from a commercial vendor, an organism receipt and acclimation form should be completed upon receipt of the amphipods and any deviations in the water quality ranges noted.

9.2 If temperature on arrival is greater than 3° C from the holding/testing temperature then the amphipods should be slowly acclimated to the correct temperature. Amphipods should be evaluated 24 hours after receipt for dead organisms. If mortality exceeds 10% then the amphipods should not be used for testing. Amphipods should be held for 24-48 hours before use in testing to ensure that injured and unhealthy organisms are not used. Amphipods should be fed the normal culture or testing food ration during holding.

9.3 If amphipods are collected from in-house cultures, amphipods to be used in testing are collected by placing adult aphipods on a #25 (710 µm) sieve containing pre-conditioned leaves. Place sieves in a larger holding container filled with reconstituted water. Provide gentle aeration. The following day, collect the neonates passing through the sieve into the holding container by gently shaking amphipods off the leaves. Count the neonates produced into a bowl/container containing fresh reconstituted water. Based on the count determine if enough neonates have been collected to start testing. A goal of 125% of total required should be collected. If not enough are produced in a single day, the process can be repeated and the neonates produced over a two day period can be combined. Adults are returned to tehculture once enough neonates are collected. Starting weight of amphipods must be in the range of about 0.02 to 0.035 mg/organism. Amphipods should be fed the normal culture or testing food ration during holding. Water exchanges should be conducted every 3 days during holding.

10.0 **Test chamber and sediment preparation (day –1)**

10.1 Glass soap water/acetone/acid-washed 300 mL beakers containing mesh screen overflow holes will serve as test chambers. The number required is eighteen (18) replicate beakers: twelve (12) replicates/beakers per treatment for biological endpoints, 3 for pore water chemistry analysis between day 21-27, and 3 for porewater chemistry on day 28. All replicate beakers will contain *H. azteca*. Each beaker should be labeled with the treatment and replicate (e.g., A-R) designation.

10.2 Prior to test initiation, sediment should be homogenized thoroughly within the bucket or other suitable container using an electric drill or mixer equipped with a stainless steel shaft and prop/agitator. After ~5 minutes of thorough mixing, the sediment in the buckets should be manually mixed with a stainless steel spatula or HDPE scoop, involving complete turnover of the sediment (i.e., bringing the bottom to the top). Utilize the power mixer again and repeat until the sediment is thoroughly mixed, homogenous in texture/color and has an even distribution of water. Repeat this process for all sediments following decontamination of equipment (Section 6.3).

10.3 Decontamination: The homogenization equipment (e.g., mixer, spatula) should be thoroughly decontaminated between the mixing of each sediment. This includes a

(1) water rinse, (2) soap water scrubbing, (3) rinse with 10% nitric acid, (4) rinse with acetone[3] and (5) deionized water wash. Rinses must be thorough to ensure all acid/solvent is removed and soaking glassware overnight in deionized water is an option.

10.4 Beakers will be filled with 100 mL (~ 2 cm) of homogenized sediment (as described in section 6.2) from each site, control and reference using a decontaminated stainless steel or HDPE scoop/spatula. The sediment should be added carefully to minimize contact with the sides of the beakers and leveled by tapping the side of the beakers. Slowly add the overlaying reconstituted water using a turbulence reducer to minimize resuspension of the sediment. Beakers should be filled to the overflow holes on the beakers.

10.5 Arbitrarily place all treatment replicates in the specified Zumwalt or modified Brunson-Zumwalt water exchange system. The water exchange system should be in a controlled environmental chamber at 23 ± 1°C. No aeration is provided unless DO drops below minimum requirements (2.5 mg/L) during testing.

10.6 Porewater characterization is conducted on day -1 or shortly after sediment receipt. Place 50 mL of sediment into a 50 mL centrifuge tube. Centrifuge at 2500 g for 30 minutes. Decant supernatant and measure temperature, pH, and total ammonia on the supernatant. Porewater for chemistry analysis is collected in an identical manner at day-1 using centrifuge bottles of appropriate volume.

11.0 Test initiation (day 0)

11.1 A water exchange should be conducted on day 0 prior to measuring parameters and adding organisms.

11.2 Water quality measurements (temperature, pH, dissolved oxygen (DO), hardness, alkalinity, conductivity and total ammonia) will be taken in a composite sample collected from all replicates of a sediment treatment and recorded. If parameters are within the specified ranges (US EPA 2000), the test may be initiated.

11.3 *Hyalella azteca* (7-8 days old) will be placed in a culture bowl or counting tray. Ten organisms should be randomly counted into HDPE cups or glass 50 mL beakers so that initial and QA-QC counts are manageable. Organism transfer should be carried out using wide-bore pipettes, fully submerging the pipettes to minimize injury (forceps are not to be used). Only apparently healthy[4] individuals should be selected at random for testing. Any organisms that are dropped or contact a dry surface cannot be used in testing.

11.4 After the appropriate number of counting-containers is filled, a technician should check to ensure each container has 10 individuals.

11.5 Include an additional 8 counting chambers (80 organisms) for initial weight measurements.

---

[1]Attention should be given to whether all equipment is stainless steel or HDPE before using acetone rinses due to concern regarding chemical release (e.g., phthalates from plastics).

[2]Amphipods may swim or curl up.

11.6 A second technician should be present to ensure that only 10 organisms are added to each chamber and that all individuals are removed from the counting beakers. Squirt bottles containing fresh filtered/dechlorinated water can be used to dislodge amphipods that are stuck to the sides of the counting chambers. Special attention should be paid to floating amphipods, which should be submerged using a drop of test water from the test beaker using a pipette.

11.7 Load all A replicates first with the sediment treatments randomized. Repeat the process for the remaining replicates. Upon addition of test organisms, the time of test initiation must be recorded on the appropriate data sheet.

11.8 Organisms in each beaker are provided fed 1.0 mL of YCT (1800 mg/L stock) to each test chamber.

11.9 Approximately one hour following the addition of test organisms, chambers should be observed for injured or unhealthy amphipods. At this time, injured or unhealthy individuals or individuals may be replaced if the response does not appear to be specific to the particular sediment treatment. Floating amphipods should be submerged using a drop of test water from the test beaker using a pipette.

12.0 Test monitoring and maintenance (days 1 – 27)

12.1 Test chambers should be observed twice daily for floating amphipods during the first week. After the first week they should be observed once daily. Any amphipods observed floating should be submerged using water droplets dropped gently from a pipette.

12.2 Water exchanges (100%) will be conducted twice daily using the automated Brunson-Zumwalt system or manually using the Zumwalt delivery system.

12.3 Water quality parameters (temperature and DO) will be measured daily in a replicate of each treatment prior to water exchange. Dissolved oxygen will be measured more frequently if it decreases more than 1 mg/L in a treatment since the previous measurement. Aeration will be provided if DO drops below 2.5 mg/L. Measure pH on composite sample three times weekly (e.g., M,W,F) and conductivity once per week.

12.4 Organisms in each beaker are fed 1 ml of YCT/day (from a 1800 mg solids/L stock) on days 0-13, and 2 ml/day will be added daily to each test chamber during the remainder of the exposure.

12.5 Record environmental chamber temperature daily.

12.6 Check light cycle and ensure that each test chamber is adequately aerated (if required) daily.

13.0 Day 28 assessment of survival and growth

9.1 Water quality: temperature, pH, and DO, alkalinity, hardness, conductivity and ammonia are measured in a composite sample collected from all replicates of a sediment treatment and recorded.

9.2 Gently pour off all but 50 mL of overlying water through an 8 or 12-inch diameter 425 µM ASTM testing sieve to isolate larvae. Swirl and suspend sediment in the remaining overlying water for easier passing of sediment through the sieves. If using an 8 inch

sieve, a 12-inch diameter sieve (1 mm mesh) can be placed over the bucket receiving the waste to support the 8 inch sieve.

9.3 Transfer small amounts of the material retained on each sieve to a counting bowl or tray for examination. Count surviving amphipods. All missing individuals or amphipods that fail to move following close observation (e.g., under a dissection microscope) and gentle prodding with a blunt probe are considered dead. Individuals exhibiting any movement are counted as alive.

9.4 Surviving amphipods from each replicate beaker are placed on pre-dried/pre-weighed pans and placed in a drying oven at 60° C for 24 hours. After 24 hours, pans are placed in a desiccator and allowed to cool. Dry weights are recorded for each pan. Dry weight is then determined by the following formula:

(amphipod and pan dry weight – pan dry weight) = amphipod dry weight

9.5 Biomass will be reported as average final mass of each sediment replicate. Weight is reported as the average individual dry weight for a sediment by dividing total biomass of each replicate by the number of surviving organisms within the replicate then calculating the mean value.

10. **Day 28 initiation of water only exposures**

10.1 Prepare a "water only" replicate for the remaining 8 replicates using a 400 mL beaker. Label each replicate and fill with reconstituted water.

10.2 Add a 3-cm x 3-cm piece of Nitex screen to each water only replicate. Nitex screen will serve as a substrate.

10.3 Water quality: temperature, pH, and DO, alkalinity, hardness, conductivity and ammonia are measured in a composite sample collected from all replicates of a sediment treatment and recorded.

10.4 Process each test replicate as described in section 9.2-9.3. Transfer surviving amphipods of each sediment treatment to its corresponding "water only" replicate.

10.5 Place the "water only" replicates back into the water exchange system.

11. **Test monitoring and maintenance (days 28 – 42)**

11.1 Perform twice daily water renewals.

11.2 Monitor each replicate and remove any dead organisms.

11.3 Water quality: Temperature is measured daily in a replicate of each treatment. Water pH and DO are measured three times weekly in a composite sample collected from all replicates of a sediment treatment and recorded. Conductivity is measured weekly.

11.4 Feed 1.0 mL of YCT per replicate

12. Day 35 reproduction assessment of water only exposure

12.1 Measure water quality as described in section 10.3.

12.2 Remove and count offspring in each replicate. Return "water only" replicates to water exchange system after counting neonates.

13. **Day 42 Test Termination of water only exposures**

13.1 Water quality: temperature, pH, and DO, alkalinity, hardness, conductivity and ammonia are measured in a composite sample collected from all replicates of a sediment treatment and recorded.
13.2 Remove and count adults and neonates in each replicate.
13.3 Determine and record the number of adult males and females in each replicate through the presence of a large 2nd gnathopod on male amphipods.
13.4 Determine the number of young produce per female by summing the Day 35 and Day 42 neonate counts and dividing by the total number of females present at day 42.
13.5 Measure dry weight as described in section 9.4.

14. **Acceptability**

14.1 Test acceptability requirements include:
14.1.1. Tests must be started with 7-8 day old amphipods.
14.1.2. 80% or greater average survival in the control on day 28
14.1.3. Reproduction ≥2 offspring/female.
14.1.4. Mean control weight on Day 28 should be greater than or equal to 0.4 mg dry/individual and greater than or equal to 0.5 mg dry/individual on Day 42.
14.1.5. Hardness, alkalinity, and ammonia in the overlying water typically should not vary by more than 50% during the test, and dissolved oxygen should be maintained above 2.5 mg/L in the overlying water.

15 **Recording data**

15.1 All specified data should be recorded in full on the appropriate data sheets (i.e., organism acclimation, water quality, survival, reproduction).
15.2 If any parameters are not within the specified range, record on a datasheet, make a note in the comments and contact study coordinator.

References

ASTM International. 2010. Standard test method for measuring the toxicity of sediment-associated contaminants with estuarine and marine invertebrates. ASTM, 1706-05. ASTM International, West Conshohocken, PA.

Borgmann 1996. Systematic analysis of aqueous ion requirements of *Hyalella azteca*: A standard artificial medium including the essential bromide ion. *Arch. Environ. Contam. Toxicol.* 30:356-363.

US EPA. 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates. EPA-600-R99-064. US EPA. Washington, DC.

USEPA/USACE 1998. US Environmental Protection Agency / US Army Corps of Engineers. Evaluation of Material Proposed for Discharge to Waters of the U.S. - Testing Manual (Inland Testing Manual). EPA-823-B-98-004, Office of Water, Washington DC.

Table A.4-1. Summary of Conditions for 42-d sediment toxicity test using *Hyalella azteca*

Description	Condition
Test Type	Static renewal
Test Duration	42 days
Temperature	23.0 ±1.0°C
pH	6.5-9
Light Quality	Wide spectrum fluorescent lights
Light Intensity	About 100-1000 lux
Photoperiod	16:8 L:D
Test Chamber Size	300 mL (with screens)
Sediment Depth/volume	2 cm (100 mL)
Overlying Water Volume	Fill to bottom of screens
Sediment settling time	Overnight
Renewal of Overlying water	100% water renewal twice daily. Water is reconstituted water created using methods specified in Borgmann (1996) but modified to contain 0.4 mg/L bromide
Age of Test Organisms	7-8 days old (starting weight must be in the range of about 0.02 to 0.035 mg/organism)
Organisms/chamber	10
Replicates/treatment	12 (plus 6 chemistry only replicates)
Organisms/treatment	180
Feeding Regime	1 ml of YCT/day (from a 1800 mg solids/L stock) on days 0-13, and 2 ml/day during the remainder of the exposure
Test Chamber Cleaning	Clean screens as required with small brush
Test Solution Aeration	None unless DO drops below 2.5 mg/L; Trickle flow (<100 bubbles/min); >60% saturation
Dilution Water	Reconstituted water created using methods specified in Borgmann (1996) but modified to contain 0.4 mg/L bromide
Test Concentrations	Site sediment, reference, control
Endpoint(s)	Survival, biomass and dry weight, reproduction

Table A.4-2. Summary of Tasks for 42-d sediment toxicity test using *Hyalella azteca*

Day	Task
-7	Sample pore water by centrifugation for porewater water quality analysis and chemistry analysis. Homogenize sediment in shipping bucket or other container appropriate for contaminant(s). Fill test chambers with sediment and add overlying water.
-1	Collect pore water samples for chemical analysis via centrifugation. Measure porewater water quality characteristics if not performed earlier. Fill test chambers with sediment and add overlying water.
0	Test initiation: Record temperature, pH, D.O. alkalinity, hardness, conductivity and total ammonia on a composite overlying water sample of all replicates from a sediment treatment. Add 10 amphipods per chamber. Collect 80 amphipods for initial weight determination. Provide each chamber with 1 mL of YCT (1800 mg/L stock solution). Place first set of peepers in sediment chemistry replicates.
1-27	Perform exposure chamber observations. Measure temperature and DO daily in a composite sample collected from replicates of a sediment treatment. Measure pH thre times weekly and conductivity weekly. Provide each chamber with 1 mL of YCT (1800 mg/L stock solution). Perform twice daily water renewals.
7	Collect day 7 peepers and sediment pore water from chemistry replicates.
14	Increase feeding rate to 2 mL YCT per replicate
14-20	Place peepers in second set of chemistry replicates.
21-27	Collect day 14-20 peepers and sediment pore water from chemistry replicates.
28	Record temperature, pH, D.O. alkalinity, hardness conductivity and total ammonia from a composite sample collected from all replicates of a sediment treatment. Sieve sediment for amphipods and record survival. Place surviving adults from 4 replicates on pans and place in drying oven at 60°C for 24 hours for determination of biomass and dry weight test endpoints. Setup eight "water only replicates" for reproduction. Add Nitex screen to each replicate. Place surviving organisms in there corresponding

	"water only" replicate. Provide each chamber with 1 mL of YCT (1800 mg/L stock solution).
29-35	Feed daily. Measure temperature and DO daily, pH three times per week and conductivity once per week. Perform twice daily water renewals.
35	Record number surviving adults and offspring in each replicate. Return adults to their original beaker and feed.
36-41	Feed daily. Measure temperature and DO daily, pH three times per week, and conductivity once per week. Perform twice daily water renewals.
42	Record temperature, pH, D.O. alkalinity, hardness, conductivity and total ammonia on a composite overlying water sample of all replicates from a sediment treatment. Record the number of surviving adults and number of neonates. Determine the number of males and females in each replicate. Weigh adult test organisms for biomass and dry weight endpoints.

Appendix B – ERDC Control Sediment Chemistry

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

ANALYTICAL REPORT

Job Number: 200-7098-1

SDG Number: 1091204

Job Description: ERDC-El-EP-C

For:
White Water Associates
429 River Lane
PO BOX 27
Amasa, MI 49903

Attention: Dr. Bette J Premo

Approved for release.
Kathryn A Kelly
Project Manager I
9/28/2011 2:28 PM

Kathryn A Kelly Project Manager I
kathryn.kelly@testamericainc.com
09/28/2011

TestAmerica Laboratories, Inc.
TestAmerica Burlington 30 Community Drive, Suite 11, South Burlington, VT 05403
Tel (802) 660-1990 Fax (802) 660-1919 www.testamericainc.com

ACCREDITED IN ACCORDANCE WITH nelac

CASE NARRATIVE

Client: White Water Associates

Project: ERDC-El-EP-C Report

Number: 200-7098-1

With the exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. In addition all laboratory quality control samples were within established control limits, with any exceptions noted below. Each sample was analyzed to achieve the lowest possible reporting limit within the constraints of the method. In some cases, due to interference or analytes present at high concentrations, samples were diluted. For diluted samples, the reporting limits are adjusted relative to the dilution required.

Calculations are performed before rounding to avoid round-off errors in calculated results.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

RECEIPT

The samples were received on 09/21/2011; the samples arrived in good condition, properly preserved and on ice. The temperature of the coolers at receipt was 5.1 C.

GRAIN SIZE

Sample UMFS was analyzed for grain size in accordance with D422 grain size. The samples were analyzed on 09/22/2011.

No difficulties were encountered during the grain size analysis.

All quality control parameters were within the acceptance limits.

TOTAL ORGANIC CARBON

Sample UMFS was analyzed for total organic carbon in accordance with Lloyd Kahn Method. The samples were analyzed on 09/23/2011.

The sample was prepared and analyzed outside the method defined holding time because the request for the test was made after the holding time for the sample expired.

No difficulties were encountered during the TOC analysis.

All quality control parameters were within the acceptance limits.

EXECUTIVE SUMMARY - Detections

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

Lab Sample ID / Analyte	Client Sample ID	Result	Qualifier	Reporting Limit	Units	Method
200-7098-1	**UMFS**					
Total Organic Carbon		14700	H	1380	mg/Kg	Lloyd Kahn
Percent Moisture		27.4		0.25	%	Moisture
Percent Solids		72.6		0.25	%	Moisture
Sieve Size 3 inch - Percent Finer		100.0			% Passing	D422
Gravel		0.0			%	D422
Sieve Size 2 inch - Percent Finer		100.0			% Passing	D422
Sand		12.7			%	D422
Sieve Size 1.5 inch - Percent Finer		100.0			% Passing	D422
Coarse Sand		0.0			%	D422
Sieve Size 1 inch - Percent Finer		100.0			% Passing	D422
Medium Sand		0.5			%	D422
Sieve Size 0.75 inch - Percent Finer		100.0			% Passing	D422
Fine Sand		12.2			%	D422
Sieve Size 0.375 inch - Percent Finer		100.0			% Passing	D422
Silt		61.1			%	D422
Sieve Size #4 - Percent Finer		100.0			% Passing	D422
Clay		26.2			%	D422
Sieve Size #10 - Percent Finer		100.0			% Passing	D422
Sieve Size #20 - Percent Finer		99.9			% Passing	D422
Sieve Size #40 - Percent Finer		99.5			% Passing	D422
Sieve Size #60 - Percent Finer		95.2			% Passing	D422
Sieve Size #80 - Percent Finer		92.2			% Passing	D422
Sieve Size #100 - Percent Finer		90.7			% Passing	D422
Sieve Size #200 - Percent Finer		87.3			% Passing	D422
Hydrometer Reading 1 - Percent Finer		63.6			% Passing	D422
Hydrometer Reading 2 - Percent Finer		52.6			% Passing	D422
Hydrometer Reading 3 - Percent Finer		38.0			% Passing	D422
Hydrometer Reading 4 - Percent Finer		30.7			% Passing	D422
Hydrometer Reading 5 - Percent Finer		26.2			% Passing	D422
Hydrometer Reading 6 - Percent Finer		19.7			% Passing	D422
Hydrometer Reading 7 - Percent Finer		16.1			% Passing	D422

METHOD SUMMARY

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

Description	Lab Location	Method	Preparation Method
Matrix: Solid			
Organic Carbon, Total (TOC)	TAL BUR	EPA Lloyd Kahn	
Percent Moisture	TAL BUR	EPA Moisture	
Grain Size	TAL BUR	ASTM D422	

Lab References:

TAL BUR = TestAmerica Burlington

Method References:

ASTM = ASTM International

EPA = US Environmental Protection Agency

METHOD / ANALYST SUMMARY

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

Method	Analyst	Analyst ID
EPA Lloyd Kahn	Nelson, Andrea J	AJN
EPA Moisture	Nelson, Andrea J	AJN
ASTM D422	Peterson, Mark A	MAP

SAMPLE SUMMARY

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

Lab Sample ID	Client Sample ID	Client Matrix	Date/Time Sampled	Date/Time Received
200-7098-1	UMFS	Solid	06/03/2011 0000	09/21/2011 1020

SAMPLE RESULTS

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

General Chemistry

Client Sample ID: **UMFS**

Lab Sample ID: 200-7098-1
Client Matrix: Solid
% Moisture: 27.4

Date Sampled: 06/03/2011 0000
Date Received: 09/21/2011 1020

Analyte	Result	Qual	Units	RL	RL	Dil	Method
Total Organic Carbon	14700	H	mg/Kg	1380	1380	1.0	Lloyd Kahn
Analysis Batch: 200-25719	Analysis Date: 09/23/2011 1433						ryWt Corrected: Y
Percent Moisture	27.4		%	0.25	0.25	1.0	Moisture
Analysis Batch: 200-25651	Analysis Date: 09/22/2011 1229						ryWt Corrected: N
Percent Solids	72.6		%	0.25	0.25	1.0	Moisture
Analysis Batch: 200-25651	Analysis Date: 09/22/2011 1229						ryWt Corrected: N

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

Client Sample ID: **UMFS**

Lab Sample ID: 200-7098-1
Client Matrix: Solid

Date Sampled: 06/03/2011 0000
Date Received: 09/21/2011 1020

D422 Grain Size

Analysis Method:	D422	Analysis Batch:	200-25826	Instrument ID:	D422_import
	N/A	Prep Batch:	N/A	Lab File ID:	200-7098-C-1.txt
Dilution:	1.0			Initial Weight/Volume:	165.27 g
Analysis Date:	09/22/2011 2250			Final Weight/Volume:	
Prep Date:	N/A				

Analyte	DryWt Corrected: N	Result (% Passing)	Qualifier	NONE	NONE
Sieve Size 3 inch - Percent Finer		100.0			
Sieve Size 2 inch - Percent Finer		100.0			
Sieve Size 1.5 inch - Percent Finer		100.0			
Sieve Size 1 inch - Percent Finer		100.0			
Sieve Size 0.75 inch - Percent Finer		100.0			
Sieve Size 0.375 inch - Percent Finer		100.0			
Sieve Size #4 - Percent Finer		100.0			
Sieve Size #10 - Percent Finer		100.0			
Sieve Size #20 - Percent Finer		99.9			
Sieve Size #40 - Percent Finer		99.5			
Sieve Size #60 - Percent Finer		95.2			
Sieve Size #80 - Percent Finer		92.2			
Sieve Size #100 - Percent Finer		90.7			
Sieve Size #200 - Percent Finer		87.3			
Hydrometer Reading 1 - Percent Finer		63.6			
Hydrometer Reading 2 - Percent Finer		52.6			
Hydrometer Reading 3 - Percent Finer		38.0			
Hydrometer Reading 4 - Percent Finer		30.7			
Hydrometer Reading 5 - Percent Finer		26.2			
Hydrometer Reading 6 - Percent Finer		19.7			
Hydrometer Reading 7 - Percent Finer		16.1			

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

Client Sample ID: **UMFS**

Lab Sample ID: 200-7098-1
Client Matrix: Solid

Date Sampled: 06/03/2011 0000
Date Received: 09/21/2011 1020

D422 Grain Size

Analysis Method:	D422	Analysis Batch:	200-25826	Instrument ID:	D422_import
	N/A	Prep Batch:	N/A	Lab File ID:	200-7098-C-1.txt
Dilution:	1.0			Initial Weight/Volume:	165.27 g
Analysis Date:	09/22/2011 2250			Final Weight/Volume:	
Prep Date:	N/A				

Analyte	DryWt Corrected: N	Result (%)	Qualifier	NONE	NONE
Gravel		0.0			
Sand		12.7			
Coarse Sand		0.0			
Medium Sand		0.5			
Fine Sand		12.2			
Silt		61.1			
Clay		26.2			

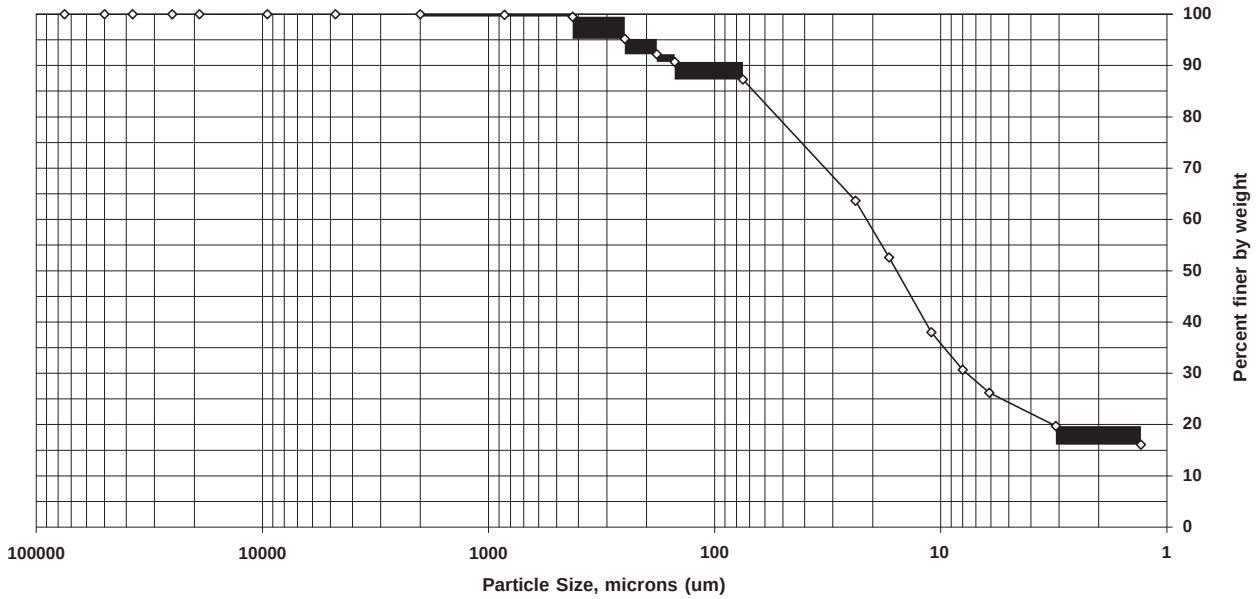
Particle Size of Soils by ASTM D422

				Date Received:	9/21/2011
Sample ID:	UMFS	**Percent Solids:**	53.3%	**Start Date:**	9/22/2011
Lab ID:	200-7098-C-1	**Specific Gravity:**	2.650	**End Date:**	9/26/2011

Shape (> #10): na

Non-soil material: na

Hardness (> #10): na



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	99.9	0.1
#40	425	99.5	0.4
#60	250	95.2	4.3
#80	180	92.2	3.0
#100	150	90.7	1.5
#200	75	87.3	3.4
Hyd1	23.8	63.6	23.7
Hyd2	16.9	52.6	11.0
Hyd3	11	38.0	14.6
Hyd4	8	30.7	7.3
Hyd5	6.1	26.2	4.5
Hyd6	3.1	19.7	6.5
Hyd7	1.3	16.1	3.6

Soil Classification	Percent of sample
Gravel	0.0
Sand	12.7
Coarse Sand	0.0
Medium Sand	0.5
Fine Sand	12.2
Silt	61.1
Clay	26.2

TestAmerica Burlington

Sediment Grain Size - D422

Client			Date Received	9/21/2011
Client Sample ID	UMFS		Start Date	09/22/2011 22:50
Lab Sample ID	200-7098-C-1		End Date	09/26/2011 19:55
Dry Weight Determination			Non-soil material:	na
Tin Weight	1.01	g	Shape (> #10):	na
Wet Sample + Tin	34.45	g	Hardness (> #10):	na
Dry Sample + Tin	18.82	g		
% Moisture	46.74	%	Date/Time in oven	09/22/2011 22:51
			Date/Time out of oven	09/23/2011 19:14

Sample Weights	Tare (g)	Pan+Samp (g)	Samp (g)
Sample Weight (Wet)		165.27	165.27
Sample Weight (Oven Dried)			88

Sample Split (oven dried)	Tare (g)	Pan+Samp (g)	Samp (g)
Sample >=#10			0
Sample <#10			88
% Passing #10			53.2

Hydrometer Data	
Serial Number	741402
Calib. Date (mm/dd/yyyy)	12/21/2010
Low Temp (C)	17.0
Reading at Low Temp	1.0035
High Temp (C)	23.0
Reading at High Temp	1.0030
Hydrometer Cal Slope	-8.33333E-05
Hydrometer Cal Intercept	1.004916667
Default Soil Gravity	2.6500

Gravel/Sand Fraction (Sieves)

Sample Fraction	Size (um)	Pan Tare (g)	Pan+Sample (g)	Sample		% Finer	Classification	Sub Class
3 inch	75000			0.00	g	100.0	Gravel	
2 inch	50000			0.00	g	100.0	Gravel	
1.5 inch	37500			0.00	g	100.0	Gravel	
1 inch	25000			0.00	g	100.0	Gravel	
3/4 inch	19000			0.00	g	100.0	Gravel	
3/8 inch	9500			0.00	g	100.0	Gravel	
#4	4750			0.00	g	100.0	Gravel	
#10	2000			0.00	g	100.0	Sand	Coarse
#20	850	384.07	384.14	0.07	g	99.9	Sand	Medium
#40	425	353.75	354.10	0.35	g	99.5	Sand	Medium
#60	250	341.79	345.58	3.79	g	95.2	Sand	Fine
#80	180	330.69	333.35	2.66	g	92.2	Sand	Fine
#100	150	326.97	328.33	1.36	g	90.7	Sand	Fine
#200	75	312.56	315.59	3.03	g	87.3	Sand	Fine
				0.00	g	87.3		

Adjusted Hydrometer Sample Mass

Hydrometer Sample Mass (g)	88

Silt/Clay Fraction (Hydrometer Test)

Hydrometer Test Time (min)	Actual	Spec. Gravity	Temp C	Particle Size (Micron)	% Finer	Classification	Sub Class
2	2	1.0380	21.0	23.8	63.6	Silt	
5	5	1.0320	21.0	16.9	52.6	Silt	
15	15	1.0240	21.0	11	38	Silt	
30	31	1.0200	21.0	8	30.7	Silt	
60	57	1.0175	21.0	6.1	26.2	Silt	
250	235	1.0140	20.5	3.1	19.7	Clay	
1440	1382	1.0120	21.0	1.3	16.1	Clay	

DATA REPORTING QUALIFIERS

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

Lab Section	Qualifier	Description
General Chemistry		
	U	Indicates the analyte was analyzed for but not detected.
	H	Sample was prepped or analyzed beyond the specified holding time

QUALITY CONTROL RESULTS

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

QC Association Summary

Lab Sample ID	Client Sample ID	Report Basis	Client Matrix	Method	Prep Batch
General Chemistry					
Analysis Batch:200-25651					
200-7098-1	UMFS	T	Solid	Moisture	
200-7098-1DU	Duplicate	T	Solid	Moisture	
Analysis Batch:200-25719					
LCS 200-25719/4	Lab Control Sample	T	Solid	Lloyd Kahn	
MB 200-25719/3	Method Blank	T	Solid	Lloyd Kahn	
200-7098-1	UMFS	T	Solid	Lloyd Kahn	

Report Basis
T = Total

Geotechnical					
Analysis Batch:200-25826					
200-7098-1	UMFS	T	Solid	D422	

Report Basis
T = Total

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

Method Blank - Batch: 200-25719

Method: Lloyd Kahn
Preparation: N/A

Lab Sample ID:	MB 200-25719/3	Analysis Batch:	200-25719	Instrument ID:	WCCH1
Client Matrix:	Solid	Prep Batch:	N/A	Lab File ID:	092311B003
Dilution:	1.0	Leach Batch:	N/A	Initial Weight/Volume:	1.0 g
Analysis Date:	09/23/2011 1201	Units:	mg/Kg	Final Weight/Volume:	1.0 g
Prep Date:	N/A				
Leach Date:	N/A				

Analyte	Result	Qual	RL	RL
Total Organic Carbon	1000	U	1000	1000

Lab Control Sample - Batch: 200-25719

Method: Lloyd Kahn
Preparation: N/A

Lab Sample ID:	LCS 200-25719/4	Analysis Batch:	200-25719	Instrument ID:	WCCH1
Client Matrix:	Solid	Prep Batch:	N/A	Lab File ID:	092311B005
Dilution:	1.0	Leach Batch:	N/A	Initial Weight/Volume:	1.0 g
Analysis Date:	09/23/2011 1213	Units:	mg/Kg	Final Weight/Volume:	1.0 g
Prep Date:	N/A				
Leach Date:	N/A				

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Total Organic Carbon	12600	11990	95	75-125	

Client: White Water Associates

Job Number: 200-7098-1
Sdg Number: 1091204

Duplicate - Batch: 200-25651

Method: Moisture
Preparation: N/A

Lab Sample ID:	200-7098-1	Analysis Batch:	200-25651	Instrument ID:	No Equipment
Client Matrix:	Solid	Prep Batch:	N/A	Lab File ID:	N/A
Dilution:	1.0	Leach Batch:	N/A	Initial Weight/Volume:	
Analysis Date:	09/22/2011 1229	Units:	%	Final Weight/Volume:	
Prep Date:	N/A				
Leach Date:	N/A				

Analyte	Sample Result/Qual	Result	RPD	Limit	Qual
Percent Moisture	27.4	26.6	3	20	
Percent Solids	72.6	73.4	1	20	

SUBCONTRACT ORDER

ERDC- EL-EP-C (Environmental Chemistry Braneb)

1091204

§ENDING LABQRATORy:

ERDCEL-EP-C {Environmental Chemistry Branch)
3909 Halls Ferry Road , Building 3299
Vicksburg. MS 39180
Phone:601-634-4826
Fax: 601-634-2742
Project Manager: Patty Tuminello

RECEIVING LABQRATORY:

White Water Associates
429 River Lane
Amasa, Ml49903
Phone :(906) 822-7889
Fax:-

BPA Call No: lC. (ECG V ＼

BPA Call Date:

Analysis	Due	Expires	Laboratory ID	Comments
ID:UMFS	Soii/Sedi1Sampled:Ol-Jun-1011 00:00			
TOC	11-0ct-2011 00:00	03-Jui·20Il 00:00		
Particle Size • Sieve	13-Sep-2011 00:00	17-Jun-2011 00:00		
Particle Size • Hydrometer	13-Sep-2011 00:00	17-Jun-20ll 00:00		
Containttrs Supplied:				

RLBy (7

q-2.0·1\
Date

0.J A\1 I \ o 'J..V
Da

Released By

Date

Page Rfifiv8 8_0

Date

Login Sample Receipt Checklist

Client: White Water Associates

Job Number: 200-7098-1
SDG Number: 1091204

Login Number: 7098
List Number: 1
Creator: Keeton, Jamie

List Source: TestAmerica Burlington

Question	Answer	Comment
Radioactivity either was not measured or, if measured, is at or below background	N/A	Lab does not accept radioactive samples.
The cooler's custody seal, if present, is intact.	N/A	Not present
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	5.1°C, IR GUN ID 9b, CF 0
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	N/A	Received project as a subcontract.
There are no discrepancies between the sample IDs on the containers and the COC.	True	
Samples are received within Holding Time.	False	Refer to Job Narrative for details.
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	N/A	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.	N/A	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

FROM: U.S. ARMY ERDC CE-WES-LM-MS (601) 634-2743
U.S. ARMY EROC CE-WES-LM-MS
3909 Halls Ferry Road
ANDREW BRAY
Vicksburg, MS 39180

11111111 1111111111
FedEl< Revenue Barcode

FedEx Express

TO: ·rESTAMERICA (802) 660·1990

30 COMMUNITY DRIVE SUITE 11

SOUTH BURLINGTON, VT 05403

CAD: 2207818
SHIP DATE: 20SEP11
WEIGHT: 37.0 LB

DIMMED: 25 X 15 X 151N

RELEASE#

Ref: 00820280W81EWFIU

DELIVERY ADDRESS (FedEx-EDR)

PRIORITY OVERNIGHT

WED
A4
Deliver by:
21SEP11

TRK *fl* 797§ 37§9 0391 FORM 0201

BTV

05403 -VT-US

XH BTVA

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL) ERDC, 3909 Halls Ferry Road Vicksburg MS, 39180	Project: Farrar DOER-TIE Project Manager: Daniel Farrar	**Reported:** 24-Oct-2011

UMFS

1091204-04 (Soil/Sediment)

ERDC- EL-EP-C (Environmental Chemistry Branch)

Metals by EPA 6000/7000 Series Methods

Analyte	Result	Reporting Limit	Units	Dilution	Prepared	Analyzed	Method	Notes
Mercury	**0.0551**	0.00400	mg/kg	1	14-Sep-2011	15-Sep-2011	EPA 7471A	
Aluminum	**10300**	10.0	mg/kg	2	16-Sep-2011	17-Sep-2011	SW 846/6010	
Calcium	**336**	10.0	mg/kg	2	16-Sep-2011	17-Sep-2011	SW 846/6010	B
Iron	**10100**	10.0	mg/kg	2	16-Sep-2011	17-Sep-2011	SW 846/6010	
Magnesium	**921**	10.0	mg/kg	2	16-Sep-2011	17-Sep-2011	SW 846/6010	
Potassium	**471**	10.0	mg/kg	2	16-Sep-2011	17-Sep-2011	SW 846/6010	
Sodium	**51.0**	10.0	mg/kg	2	16-Sep-2011	17-Sep-2011	SW 846/6010	B
Antimony	ND	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	U
Arsenic	**2.76**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Barium	**150**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Beryllium	**0.824**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Cadmium	ND	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	U
Chromium	**16.9**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Cobalt	**6.17**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Copper	**13.7**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Lead	**12.3**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Manganese	**450**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Molybdenum	**0.339**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	J
Nickel	**11.4**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Selenium	**0.760**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Silver	**0.283**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	J
Thallium	**0.182**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	J
Tin	**0.256**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	J
Vanadium	**23.4**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	
Zinc	**39.4**	0.500	mg/kg	10	16-Sep-2011	19-Sep-2011	SW 846/6020	

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL) ERDC, 3909 Halls Ferry Road Vicksburg MS, 39180	Project: Farrar DOER-TIE Project Manager: Daniel Farrar	**Reported:** 24-Oct-2011

UMFS

1091204-04 (Soil/Sediment)

ERDC- EL-EP-C (Environmental Chemistry Branch)

Organochlorine Pesticides by EPA Method 8081A

Analyte	Result	Reporting Limit	Units	Dilution	Prepared	Analyzed	Method	Notes
4,4´-DDD	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
4,4´-DDE	**0.57**	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	
4,4´-DDT	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Aldrin	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
alpha-BHC	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
alpha-Chlordane	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
beta-BHC	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
delta-BHC	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Dieldrin	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Endosulfan I	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Endosulfan II	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Endosulfan sulfate	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Endrin	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Endrin aldehyde	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Endrin ketone	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
gamma-BHC (Lindane)	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
gamma-Chlordane	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Heptachlor	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Heptachlor epoxide	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Methoxychlor	ND	0.35	ug/kg dry	1	12-Oct-2011	05-Oct-2011	EPA 8081A	U
Surrogate: 2,4,5,6 Tetrachloro-m-xylene		*58.0 %*	*40-125*		*12-Oct-2011*	*05-Oct-2011*	*EPA 8081A*	

The results in this report apply to the samples analyzed in accordance with the chain of custody document. This analytical report must be reproduced in its entirety.

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL) ERDC, 3909 Halls Ferry Road Vicksburg MS, 39180	Project: Farrar DOER-TIE Project Manager: Daniel Farrar	**Reported:** 24-Oct-2011

UMFS

1091204-04 (Soil/Sediment)

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Dilution	Prepared	Analyzed	Method	Notes
Organochlorine Pesticides by EPA Method 8081A								
Surrogate: Decachorobiphenyl [2C]		*84.5 %*	*55-130*		*12-Oct-2011*	*05-Oct-2011*	*EPA 8081A*	
Organophosphorus Pesticides by EPA Method 8141A								
Tokuthion (Prothiofos)	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Trichloronate	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Sulfotep	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Fensulfothion	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Dimethoate	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Disulfoton	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
EPN	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Ethion	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Ethoprop	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Malathion	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Fenthion	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Stirophos	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Merphos	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Mevinphos	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Parathion-ethyl	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Parathion-methyl	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Phorate	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Ronnel	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Dichlorvos	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL) ERDC, 3909 Halls Ferry Road Vicksburg MS, 39180	Project: Farrar DOER-TIE Project Manager: Daniel Farrar	**Reported:** 24-Oct-2011

UMFS

1091204-04 (Soil/Sediment)

Analyte	Result	Reporting Limit	Units	Dilution	Prepared	Analyzed	Method	Notes
ERDC- EL-EP-C (Environmental Chemistry Branch)								
Organophosphorus Pesticides by EPA Method 8141A								
Chlorpyrifos	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Coumaphos	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Diazinon	ND	9.5	ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Demeton	0.0		ug/kg dry	1	12-Oct-2011	20-Oct-2011	EPA 8141A	U
Surrogate: Triphenyl phosphate		*113 %*	*0-200*		*12-Oct-2011*	*20-Oct-2011*	*EPA 8141A*	
Polynuclear Aromatic Compounds by GC/MS with Selected Ion Monitoring								
2-Methylnaphthalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1-Methylnaphthalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2-ethylnaphthalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1-ethylnaphthalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2,6/2,7-dimethylnaphthalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1,3-dimethylnaphthalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1,6-dimethylnaphthtalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1,4/2,3-dimethylnaphthalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2-isopropylnaphthalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1,8-dimethylnaphthalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2,3,5-Trimethylnaphthalene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1-methylfluoene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2-ethylfluorene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1,8-dimethylfluorene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
Dibenzothiophene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL) ERDC, 3909 Halls Ferry Road Vicksburg MS, 39180	Project: Farrar DOER-TIE Project Manager: Daniel Farrar	**Reported:** 24-Oct-2011

UMFS

1091204-04 (Soil/Sediment)

ERDC- EL-EP-C (Environmental Chemistry Branch)

Polynuclear Aromatic Compounds by GC/MS with Selected Ion Monitoring

Analyte	Result	Reporting Limit	Units	Dilution	Prepared	Analyzed	Method	Notes
1-Methylphenanthrene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2-methylphenanthrene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2-methylanthracene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
9-methylanthracene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2,3-dimethylanthracene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
9,10-dimethylanthracene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2-tetrabutylanthracene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
9-ethyl-10-methylphenanthrene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1-methylpyrene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1,4,5,8-tetramethylanthracene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1-methylfluoranthene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2-methylfluoranthene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1-methylbenz(a)anthracene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
1-methylchrysene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
2-methylchrysene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
3-methylchrysene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
6-ethylchrysene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
6,8-dimethylbenz(a)anthracene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
3,9-dimethylbenz(a)anthracene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
8,9,11-trimethylbenz(a)anthracene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL) ERDC, 3909 Halls Ferry Road Vicksburg MS, 39180	Project: Farrar DOER-TIE Project Manager: Daniel Farrar	**Reported:** 24-Oct-2011

UMFS

1091204-04 (Soil/Sediment)

ERDC- EL-EP-C (Environmental Chemistry Branch)

Polynuclear Aromatic Compounds by GC/MS with Selected Ion Monitoring

Analyte	Result	Reporting Limit	Units	Dilution	Prepared	Analyzed	Method	Notes
8-methylbenzo(a)pyrene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
7,10-dimethylbenzo(a)pyrene	ND	16.8	ug/kg dry	1	12-Oct-2011	24-Oct-2011	EPA 8270	U
Acenaphthene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Acenaphthylene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Anthracene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Benzo (a) anthracene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Benzo (a) pyrene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Benzo (b) fluoranthene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Benzo (g,h,i) perylene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Benzo (k) fluoranthene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Chrysene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Dibenz (a,h) anthracene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Fluoranthene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Fluorene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Indeno (1,2,3-cd) pyrene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Naphthalene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Phenanthrene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Pyrene	ND	7.14	ug/kg dry	1	12-Oct-2011	18-Oct-2011	EPA 8270C	U
Surrogate: 2-Fluorobiphenyl		*72.0 %*	*45-105*		*12-Oct-2011*	*18-Oct-2011*	*EPA 8270C*	
Surrogate: Terphenyl-dl4		*64.5 %*	*30-125*		*12-Oct-2011*	*18-Oct-2011*	*EPA 8270C*	

Classical Chemistry Parameters

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)	Project: Farrar DOER-TIE	
ERDC, 3909 Halls Ferry Road		**Reported:**
Vicksburg MS, 39180	Project Manager: Daniel Farrar	24-Oct-2011

UMFS

1091204-04 (Soil/Sediment)

Analyte	Result	Reporting Limit	Units	Dilution	Prepared	Analyzed	Method	Notes
ERDC- EL-EP-C (Environmental Chemistry Branch)								
Classical Chemistry Parameters								
% Solids	**70.6**	0.100	g	1	16-Sep-2011	16-Sep-2011	% Calculation	

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL) ERDC, 3909 Halls Ferry Road Vicksburg MS, 39180	Project: Farrar DOER-TIE Project Manager: Daniel Farrar	**Reported:** 24-Oct-2011

Metals by EPA 6000/7000 Series Methods - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B109062 - EPA 3050B										
Blank (B109062-BLK1)				Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	ND	0.0001	mg/kg							U
Blank (B109062-BLK2)				Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	ND	0.0001	mg/kg							U
LCS (B109062-BS1)				Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.0852	0.0001	mg/kg	0.08000		107	75-125			
LCS (B109062-BS2)				Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.0903	0.0001	mg/kg	0.08000		113	75-125			
Duplicate (B109062-DUP1)		**Source: 1090104-37**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.0438	1.00E-4	mg/kg		0.0432			1.43	25	
Duplicate (B109062-DUP2)		**Source: 1090901-41**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.0535	0.0001	mg/kg		0.0545			1.95	25	
Duplicate (B109062-DUP3)		**Source: 1091201-02**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.0223	0.000101	mg/kg		0.0235			5.03	25	
Duplicate (B109062-DUP4)		**Source: 1091204-04**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.0518	0.0001	mg/kg		0.0551			6.21	25	
Matrix Spike (B109062-MS1)		**Source: 1090104-37**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.227	0.0002	mg/kg	0.2001	0.0432	91.6	75-125			
Matrix Spike (B109062-MS2)		**Source: 1090901-41**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.235	0.0002	mg/kg	0.2001	0.0545	90.2	75-125			

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)
ERDC, 3909 Halls Ferry Road
Vicksburg MS, 39180

Project: Farrar DOER-TIE

Project Manager: Daniel Farrar

Reported:
24-Oct-2011

Metals by EPA 6000/7000 Series Methods - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B109062 - EPA 3050B										
Matrix Spike (B109062-MS3)		**Source: 1091201-02**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.204	0.0002	mg/kg	0.1996	0.0235	90.7	75-125			
Matrix Spike (B109062-MS4)		**Source: 1091204-04**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.231	0.0002	mg/kg	0.2000	0.0551	88.0	75-125			
Matrix Spike Dup (B109062-MSD1)		**Source: 1090104-37**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.238	0.0002	mg/kg	0.2000	0.0432	97.2	75-125	5.89	25	
Matrix Spike Dup (B109062-MSD2)		**Source: 1090901-41**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.237	0.0002	mg/kg	0.2004	0.0545	91.0	75-125	0.927	25	
Matrix Spike Dup (B109062-MSD3)		**Source: 1091201-02**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.202	0.0002	mg/kg	0.2001	0.0235	89.0	75-125	1.83	25	
Matrix Spike Dup (B109062-MSD4)		**Source: 1091204-04**		Prepared: 14-Sep-2011 Analyzed: 15-Sep-2011						
Mercury	0.233	0.0002	mg/kg	0.2005	0.0551	88.7	75-125	0.818	25	
Batch B109084 - EPA 3050B										
Blank (B109084-BLK1)				Prepared: 16-Sep-2011 Analyzed: 17-Sep-2011						
Aluminum	ND	10.0	mg/kg							
Calcium	17.8	10.0	mg/kg							
Iron	4.23	10.0	mg/kg							J
Magnesium	6.29	10.0	mg/kg							J
Potassium	8.26	10.0	mg/kg							J
Sodium	13.9	10.0	mg/kg							

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)	Project: Farrar DOER-TIE	
ERDC, 3909 Halls Ferry Road		**Reported:**
Vicksburg MS, 39180	Project Manager: Daniel Farrar	24-Oct-2011

Metals by EPA 6000/7000 Series Methods - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B109084 - EPA 3050B										
Blank (B109084-BLK2)				Prepared: 16-Sep-2011 Analyzed: 19-Sep-2011						
Antimony	ND	0.500	mg/kg							U
Arsenic	ND	0.500	mg/kg							U
Barium	0.100	0.500	mg/kg							J
Beryllium	ND	0.500	mg/kg							U
Cadmium	ND	0.500	mg/kg							U
Chromium	0.252	0.500	mg/kg							J
Cobalt	ND	0.500	mg/kg							U
Copper	0.120	0.500	mg/kg							J
Lead	ND	0.500	mg/kg							U
Manganese	ND	0.500	mg/kg							U
Molybdenum	ND	0.500	mg/kg							U
Nickel	0.144	0.500	mg/kg							J
Selenium	ND	0.500	mg/kg							U
Silver	ND	0.500	mg/kg							U
Thallium	ND	0.500	mg/kg							U
Tin	ND	0.500	mg/kg							U
Vanadium	ND	0.500	mg/kg							U
Zinc	0.283	0.500	mg/kg							J
LCS (B109084-BS1)				Prepared: 16-Sep-2011 Analyzed: 17-Sep-2011						
Aluminum	899	10.0	mg/kg	1000		89.9	80-120			
Calcium	970	10.0	mg/kg	1000		97.0	80-120			B
Iron	972	10.0	mg/kg	1000		97.2	80-120			
Magnesium	964	10.0	mg/kg	1000		96.4	80-120			
Potassium	919	10.0	mg/kg	1000		91.9	80-120			
Sodium	912	10.0	mg/kg	1000		91.2	80-120			B
LCS (B109084-BS2)				Prepared: 16-Sep-2011 Analyzed: 19-Sep-2011						
Antimony	90.0	0.500	mg/kg	100.0		90.0	80-120			
Arsenic	47.9	0.500	mg/kg	50.00		95.9	80-120			
Barium	178	0.500	mg/kg	200.0		88.8	80-120			
Beryllium	50.3	0.500	mg/kg	50.00		101	80-120			
Cadmium	48.2	0.500	mg/kg	50.00		96.5	80-120			
Chromium	101	0.500	mg/kg	100.0		101	80-120			
Cobalt	101	0.500	mg/kg	100.0		101	80-120			
Copper	100	0.500	mg/kg	100.0		100	80-120			
Lead	90.5	0.500	mg/kg	100.0		90.5	80-120			
Manganese	251	0.500	mg/kg	250.0		101	80-120			
Molybdenum	48.6	0.500	mg/kg	50.00		97.1	80-120			
Nickel	100	0.500	mg/kg	100.0		100	80-120			
Selenium	48.7	0.500	mg/kg	50.00		97.4	80-120			

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)	Project: Farrar DOER-TIE	
ERDC, 3909 Halls Ferry Road		**Reported:**
Vicksburg MS, 39180	Project Manager: Daniel Farrar	24-Oct-2011

Metals by EPA 6000/7000 Series Methods - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B109084 - EPA 3050B										
LCS (B109084-BS2)				Prepared: 16-Sep-2011 Analyzed: 19-Sep-2011						
Silver	46.5	0.500	mg/kg	50.00		93.0	80-120			
Thallium	48.7	0.500	mg/kg	50.00		97.4	80-120			
Tin	47.3	0.500	mg/kg	50.00		94.6	80-120			
Vanadium	100	0.500	mg/kg	100.0		100	80-120			
Zinc	187	0.500	mg/kg	200.0		93.5	80-120			
Duplicate (B109084-DUP1)	**Source: 1091204-04**			Prepared: 16-Sep-2011 Analyzed: 17-Sep-2011						
Aluminum	9990	10.0	mg/kg		10300			3.10	20	
Calcium	315	10.0	mg/kg		336			6.46	20	B
Iron	9750	10.0	mg/kg		10100			3.79	20	
Magnesium	866	10.0	mg/kg		921			6.20	20	
Potassium	445	10.0	mg/kg		471			5.52	20	
Sodium	50.0	10.0	mg/kg		51.0			2.03	20	B
Duplicate (B109084-DUP2)	**Source: 1091204-04**			Prepared: 16-Sep-2011 Analyzed: 19-Sep-2011						
Antimony	ND	0.500	mg/kg		ND				20	U
Arsenic	2.64	0.500	mg/kg		2.76			4.38	20	
Barium	136	0.500	mg/kg		150			10.1	20	
Beryllium	0.755	0.500	mg/kg		0.824			8.82	20	
Cadmium	ND	0.500	mg/kg		ND				20	U
Chromium	16.3	0.500	mg/kg		16.9			3.81	20	
Cobalt	5.81	0.500	mg/kg		6.17			5.97	20	
Copper	12.9	0.500	mg/kg		13.7			5.95	20	
Lead	11.1	0.500	mg/kg		12.3			10.9	20	
Manganese	421	0.500	mg/kg		450			6.77	20	
Molybdenum	0.269	0.500	mg/kg		0.339			23.1	20	J
Nickel	10.6	0.500	mg/kg		11.4			7.25	20	
Selenium	0.652	0.500	mg/kg		0.760			15.2	20	
Silver	0.150	0.500	mg/kg		0.283			61.3	20	J
Thallium	0.160	0.500	mg/kg		0.182			13.3	20	J
Tin	0.140	0.500	mg/kg		0.256			58.7	20	J
Vanadium	23.3	0.500	mg/kg		23.4			0.316	20	
Zinc	37.8	0.500	mg/kg		39.4			4.01	20	

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)
ERDC, 3909 Halls Ferry Road
Vicksburg MS, 39180

Project: Farrar DOER-TIE

Project Manager: Daniel Farrar

Reported:
24-Oct-2011

Metals by EPA 6000/7000 Series Methods - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B109084 - EPA 3050B										
Matrix Spike (B109084-MS1)	**Source: 1091204-04**			Prepared: 16-Sep-2011 Analyzed: 17-Sep-2011						
Aluminum	14200	10.0	mg/kg	993.4	10300	388	80-120			
Calcium	1280	10.0	mg/kg	993.4	336	94.7	80-120			B
Iron	11100	10.0	mg/kg	993.4	10100	102	80-120			
Magnesium	1970	10.0	mg/kg	993.4	921	106	80-120			
Potassium	1420	10.0	mg/kg	993.4	471	95.9	80-120			
Sodium	929	10.0	mg/kg	993.4	51.0	88.4	80-120			B
Matrix Spike (B109084-MS2)	**Source: 1091204-04**			Prepared: 16-Sep-2011 Analyzed: 19-Sep-2011						
Antimony	1.75	0.500	mg/kg	99.34	ND	1.76	80-120			
Arsenic	45.7	0.500	mg/kg	49.67	2.76	86.5	80-120			
Barium	331	0.500	mg/kg	198.7	150	90.9	80-120			
Beryllium	54.0	0.500	mg/kg	49.67	0.824	107	80-120			
Cadmium	48.5	0.500	mg/kg	49.67	ND	97.7	80-120			
Chromium	126	0.500	mg/kg	99.34	16.9	110	80-120			
Cobalt	110	0.500	mg/kg	99.34	6.17	104	80-120			
Copper	113	0.500	mg/kg	99.34	13.7	100	80-120			
Lead	102	0.500	mg/kg	99.34	12.3	90.6	80-120			
Manganese	719	0.500	mg/kg	248.4	450	108	80-120			
Molybdenum	33.8	0.500	mg/kg	49.67	0.339	67.4	80-120			
Nickel	114	0.500	mg/kg	99.34	11.4	104	80-120			
Selenium	47.2	0.500	mg/kg	49.67	0.760	93.4	80-120			
Silver	45.9	0.500	mg/kg	49.67	0.283	91.8	80-120			
Thallium	48.9	0.500	mg/kg	49.67	0.182	98.1	80-120			
Tin	3.50	0.500	mg/kg	49.67	0.256	6.52	80-120			
Vanadium	123	0.500	mg/kg	99.34	23.4	100	80-120			
Zinc	234	0.500	mg/kg	198.7	39.4	97.7	80-120			

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)	Project: Farrar DOER-TIE	
ERDC, 3909 Halls Ferry Road		**Reported:**
Vicksburg MS, 39180	Project Manager: Daniel Farrar	24-Oct-2011

Organochlorine Pesticides by EPA Method 8081A - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B110067 - EPA 3545										
Blank (B110067-BLK1)				Prepared: 12-Oct-2011 Analyzed: 05-Oct-2011						
4,4´-DDD	ND	0.25	ug/kg wet							U
4,4´-DDE	ND	0.25	ug/kg wet							U
4,4´-DDT	ND	0.25	ug/kg wet							U
Aldrin	ND	0.25	ug/kg wet							U
alpha-BHC	ND	0.25	ug/kg wet							U
alpha-Chlordane	ND	0.25	ug/kg wet							U
beta-BHC	ND	0.25	ug/kg wet							U
delta-BHC	ND	0.25	ug/kg wet							U
Dieldrin	ND	0.25	ug/kg wet							U
Endosulfan I	ND	0.25	ug/kg wet							U
Endosulfan II	ND	0.25	ug/kg wet							U
Endosulfan sulfate	ND	0.25	ug/kg wet							U
Endrin	ND	0.25	ug/kg wet							U
Endrin aldehyde	ND	0.25	ug/kg wet							U
Endrin ketone	ND	0.25	ug/kg wet							U
gamma-BHC (Lindane)	ND	0.25	ug/kg wet							U
gamma-Chlordane	ND	0.25	ug/kg wet							U
Heptachlor	ND	0.25	ug/kg wet							U
Heptachlor epoxide	ND	0.25	ug/kg wet							U
Methoxychlor	ND	0.25	ug/kg wet							U
Surrogate: 2,4,5,6 Tetrachloro-m-xylene	*2.64*		*ug/kg wet*	*4.000*		*66.0*	*40-125*			
Surrogate: Decachorobiphenyl [2C]	*3.62*		*ug/kg wet*	*4.000*		*90.5*	*55-130*			
LCS (B110067-BS1)				Prepared: 12-Oct-2011 Analyzed: 05-Oct-2011						
4,4´-DDD	4.1	0.25	ug/kg wet	4.000		102	30-135			
4,4´-DDE	4.6	0.25	ug/kg wet	4.000		116	70-125			
4,4'-DDT [2C]	4.7	0.25	ug/kg wet	4.000		116	45-140			
Aldrin [2C]	3.2	0.25	ug/kg wet	4.000		80.0	45-140			
alpha-BHC [2C]	3.8	0.25	ug/kg wet	4.000		94.5	60-125			
alpha-Chlordane	4.9	0.25	ug/kg wet	4.000		124	65-120			
beta-BHC	3.6	0.25	ug/kg wet	4.000		91.0	60-125			
delta-BHC	3.1	0.25	ug/kg wet	4.000		77.0	55-130			
Dieldrin	4.1	0.25	ug/kg wet	4.000		103	65-125			
Endrin	4.2	0.25	ug/kg wet	4.000		106	60-135			
Endrin aldehyde	3.6	0.25	ug/kg wet	4.000		89.0	35-145			
Endrin ketone	4.2	0.25	ug/kg wet	4.000		106	65-135			
gamma-BHC (Lindane)	4.1	0.25	ug/kg wet	4.000		104	60-125			
gamma-Chlordane	3.5	0.25	ug/kg wet	4.000		87.0	65-125			
Heptachlor	3.8	0.25	ug/kg wet	4.000		96.0	50-140			
Heptachlor epoxide	4.0	0.25	ug/kg wet	4.000		99.0	65-130			
Methoxychlor	5.1	0.25	ug/kg wet	4.000		126	55-145			

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)	Project: Farrar DOER-TIE	
ERDC, 3909 Halls Ferry Road		**Reported:**
Vicksburg MS, 39180	Project Manager: Daniel Farrar	24-Oct-2011

Organochlorine Pesticides by EPA Method 8081A - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B110067 - EPA 3545										
LCS (B110067-BS1)				Prepared: 12-Oct-2011 Analyzed: 05-Oct-2011						
Surrogate: 2,4,5,6 Tetrachloro-m-xylene	*2.60*		*ug/kg wet*	*4.000*		*65.0*	*40-125*			
Surrogate: Decachorobiphenyl [2C]	*3.84*		*ug/kg wet*	*4.000*		*96.0*	*55-130*			

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)	Project: Farrar DOER-TIE	
ERDC, 3909 Halls Ferry Road		**Reported:**
Vicksburg MS, 39180	Project Manager: Daniel Farrar	24-Oct-2011

Organophosphorus Pesticides by EPA Method 8141A - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B110067 - EPA 3545										
Blank (B110067-BLK1)				Prepared: 12-Oct-2011 Analyzed: 20-Oct-2011						
Phorate	ND	6.7	ug/kg wet							U
Ethoprop	ND	6.7	ug/kg wet							U
Fensulfothion	ND	6.7	ug/kg wet							U
Fenthion	ND	6.7	ug/kg wet							U
Malathion	ND	6.7	ug/kg wet							U
Merphos	ND	6.7	ug/kg wet							U
Mevinphos	ND	6.7	ug/kg wet							U
Ethion	ND	6.7	ug/kg wet							U
Parathion-methyl	ND	6.7	ug/kg wet							U
Dichlorvos	ND	6.7	ug/kg wet							U
Ronnel	ND	6.7	ug/kg wet							U
Stirophos	ND	6.7	ug/kg wet							U
Sulfotep	ND	6.7	ug/kg wet							U
Tokuthion (Prothiofos)	ND	6.7	ug/kg wet							U
Trichloronate	ND	6.7	ug/kg wet							U
Parathion-ethyl	ND	6.7	ug/kg wet							U
Dimethoate	ND	6.7	ug/kg wet							U
Diazinon	ND	6.7	ug/kg wet							U
Coumaphos	ND	6.7	ug/kg wet							U
Chlorpyrifos	ND	6.7	ug/kg wet							U
EPN	ND	6.7	ug/kg wet							U
Disulfoton	ND	6.7	ug/kg wet							U
Demeton	0.0		ug/kg wet							U
Surrogate: Triphenyl phosphate	*7.42*		*ug/kg wet*	*10.00*		*74.2*	*0-200*			
LCS (B110067-BS2)				Prepared: 12-Oct-2011 Analyzed: 20-Oct-2011						
EPN	8	6.7	ug/kg wet	8.000		96.2	40-150			
Parathion-ethyl	7	6.7	ug/kg wet	8.000		85.2	40-150			
Parathion-methyl	7	6.7	ug/kg wet	8.000		88.2	40-150			
Ronnel	8	6.7	ug/kg wet	8.000		96.5	40-150			
Tokuthion (Prothiofos)	7	6.7	ug/kg wet	8.000		92.5	40-150			
Sulfotep	6	6.7	ug/kg wet	8.000		78.2	40-150			J
Surrogate: Triphenyl phosphate	*ND*		*ug/kg wet*	*10.00*			*40-150*			*S-07, U*

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)	Project: Farrar DOER-TIE	
ERDC, 3909 Halls Ferry Road		**Reported:**
Vicksburg MS, 39180	Project Manager: Daniel Farrar	24-Oct-2011

Polynuclear Aromatic Compounds by GC/MS with Selected Ion Monitoring - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B110066 - EPA 3545										
Blank (B110066-BLK1)				Prepared: 12-Oct-2011 Analyzed: 18-Oct-2011						
Acenaphthene	ND	5.10	ug/kg wet							U
Acenaphthylene	ND	5.10	ug/kg wet							U
Anthracene	ND	5.10	ug/kg wet							U
Benzo (a) anthracene	ND	5.10	ug/kg wet							U
Benzo (a) pyrene	ND	5.10	ug/kg wet							U
Benzo (b) fluoranthene	ND	5.10	ug/kg wet							U
2-Methylnaphthalene	ND	12.0	ug/kg wet							U
Benzo (g,h,i) perylene	ND	5.10	ug/kg wet							U
1-Methylnaphthalene	ND	12.0	ug/kg wet							U
2-ethylnaphthalene	ND	12.0	ug/kg wet							U
Benzo (k) fluoranthene	ND	5.10	ug/kg wet							U
Chrysene	ND	5.10	ug/kg wet							U
1-ethylnaphthalene	ND	12.0	ug/kg wet							U
2,6/2,7-dimethylnaphthalene	ND	12.0	ug/kg wet							U
1,3-dimethylnaphthalene	ND	12.0	ug/kg wet							U
1,6-dimethylnaphthtalene	ND	12.0	ug/kg wet							U
1,4/2,3-dimethylnaphthalene	ND	12.0	ug/kg wet							U
Dibenz (a,h) anthracene	ND	5.10	ug/kg wet							U
2-isopropylnaphthalene	ND	12.0	ug/kg wet							U
Fluoranthene	ND	5.10	ug/kg wet							U
1,8-dimethylnaphthalene	ND	12.0	ug/kg wet							U
2,3,5-Trimethylnaphthalene	ND	12.0	ug/kg wet							U
Fluorene	ND	5.10	ug/kg wet							U
1-methylfluoene	ND	12.0	ug/kg wet							U
2-ethylfluorene	ND	12.0	ug/kg wet							U
1,8-dimethylfluorene	ND	12.0	ug/kg wet							U
Indeno (1,2,3-cd) pyrene	ND	5.10	ug/kg wet							U
Naphthalene	ND	5.10	ug/kg wet							U
Dibenzothiophene	ND	12.0	ug/kg wet							U
1-Methylphenanthrene	ND	12.0	ug/kg wet							U
Phenanthrene	ND	5.10	ug/kg wet							U
2-methylphenanthrene	ND	12.0	ug/kg wet							U
2-methylanthracene	ND	12.0	ug/kg wet							U
9-methylanthracene	ND	12.0	ug/kg wet							U
Pyrene	ND	5.10	ug/kg wet							U
2,3-dimethylanthracene	ND	12.0	ug/kg wet							U
9,10-dimethylanthracene	ND	12.0	ug/kg wet							U
2-tetrabutylanthracene	ND	12.0	ug/kg wet							U
9-ethyl-10-methylphenanthrene	ND	12.0	ug/kg wet							U
1-methylpyrene	ND	12.0	ug/kg wet							U
1,4,5,8-tetramethylanthracene	ND	12.0	ug/kg wet							U

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)	Project: Farrar DOER-TIE	
ERDC, 3909 Halls Ferry Road		**Reported:**
Vicksburg MS, 39180	Project Manager: Daniel Farrar	24-Oct-2011

Polynuclear Aromatic Compounds by GC/MS with Selected Ion Monitoring - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B110066 - EPA 3545										
Blank (B110066-BLK1)				Prepared: 12-Oct-2011 Analyzed: 24-Oct-2011						
1-methylfluoranthene	ND	12.0	ug/kg wet							U
2-methylfluoranthene	ND	12.0	ug/kg wet							U
1-methylbenz(a)anthracene	ND	12.0	ug/kg wet							U
1-methylchrysene	ND	12.0	ug/kg wet							U
2-methylchrysene	ND	12.0	ug/kg wet							U
3-methylchrysene	ND	12.0	ug/kg wet							U
6-ethylchrysene	ND	12.0	ug/kg wet							U
6,8-dimethylbenz(a)anthracene	ND	12.0	ug/kg wet							U
3,9-dimethylbenz(a)anthracene	ND	12.0	ug/kg wet							U
8,9,11-trimethylbenz(a)anthracene	ND	12.0	ug/kg wet							U
8-methylbenzo(a)pyrene	ND	12.0	ug/kg wet							U
7,10-dimethylbenzo(a)pyrene	ND	12.0	ug/kg wet							U
Surrogate: 2-Fluorobiphenyl	*98*		*ug/kg wet*	*133.3*		*73.5*	*45-105*			
Surrogate: Terphenyl-dl4	*74*		*ug/kg wet*	*133.3*		*55.5*	*30-125*			
LCS (B110066-BS1)				Prepared: 12-Oct-2011 Analyzed: 18-Oct-2011						
Acenaphthene	92.0	5.10	ug/kg wet	133.3		69.0	45-110			
Acenaphthylene	76.0	5.10	ug/kg wet	133.3		57.0	45-105			
Anthracene	118	5.10	ug/kg wet	133.3		88.5	55-105			
Benzo (a) anthracene	126	5.10	ug/kg wet	133.3		94.5	50-110			
Benzo (a) pyrene	136	5.10	ug/kg wet	133.3		102	50-110			
Benzo (b) fluoranthene	154	5.10	ug/kg wet	133.3		116	45-115			
Benzo (g,h,i) perylene	132	5.10	ug/kg wet	133.3		99.0	40-125			
1-Methylnaphthalene	92.0	12.0	ug/kg wet	133.3		69.0	50-110			
Benzo (k) fluoranthene	154	5.10	ug/kg wet	133.3		116	45-125			
Chrysene	96.0	5.10	ug/kg wet	133.3		72.0	55-110			
1,6-dimethylnaphthtalene	76.0	12.0	ug/kg wet	133.2		57.1	50-110			
Dibenz (a,h) anthracene	148	5.10	ug/kg wet	133.3		111	40-125			
Fluoranthene	110	5.10	ug/kg wet	133.3		82.5	55-115			
1,8-dimethylnaphthalene	76.0	12.0	ug/kg wet	131.2		57.9	50-110			
2,3,5-Trimethylnaphthalene	78.0	12.0	ug/kg wet	133.1		58.6	50-110			
Fluorene	88.0	5.10	ug/kg wet	133.3		66.0	50-110			
1-methylfluoene	88.0	12.0	ug/kg wet	131.2		67.1	50-110			
Indeno (1,2,3-cd) pyrene	158	5.10	ug/kg wet	133.3		118	40-120			
Naphthalene	80.0	5.10	ug/kg wet	133.3		60.0	40-105			
1-Methylphenanthrene	74.0	12.0	ug/kg wet	132.8		55.7	50-110			
Phenanthrene	70.0	5.10	ug/kg wet	133.3		52.5	50-110			
Pyrene	110	5.10	ug/kg wet	133.3		82.5	45-125			
9-methylanthracene	86.0	12.0	ug/kg wet	131.7		65.3	50-110			
Surrogate: 2-Fluorobiphenyl	*90*		*ug/kg wet*	*133.3*		*67.5*	*45-105*			
Surrogate: Terphenyl-dl4	*70*		*ug/kg wet*	*133.3*		*52.5*	*30-125*			

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL)	Project: Farrar DOER-TIE	
ERDC, 3909 Halls Ferry Road		**Reported:**
Vicksburg MS, 39180	Project Manager: Daniel Farrar	24-Oct-2011

Polynuclear Aromatic Compounds by GC/MS with Selected Ion Monitoring - Quality Control

ERDC- EL-EP-C (Environmental Chemistry Branch)

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B110066 - EPA 3545										
LCS (B110066-BS1)	Prepared: 12-Oct-2011 Analyzed: 18-Oct-2011									

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

ERDC -- Vicksburg (EL) ERDC, 3909 Halls Ferry Road Vicksburg MS, 39180	Project: Farrar DOER-TIE Project Manager: Daniel Farrar	**Reported:** 24-Oct-2011

Notes and Definitions

U	Analyte included in the analysis, but not detected
S-07	Surrogate inadvertently left out of sample.
J	Detected but below the Reporting Limit; therefore, result is an estimated concentration (CLP J-Flag).
B	Analyte is found in the associated blank as well as in the sample (CLP B-flag).
DET	Analyte DETECTED
ND	Analyte NOT DETECTED at or above the reporting limit
NR	Not Reported
Dry	Sample results reported on a dry weight basis
RPD	Relative percent difference

USACE ERDC-EP-C
3909 Halls Ferry Road
Vicksburg, MS 39180-6199