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VOLUME II: Laboratory Reports

**Sediment Sampling, Biological Analyses, and
Chemical Analyses for Eighteenmile Creek AOC,
Olcott, New York**

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CHEMISTRY

1.0 INTRODUCTION

The U.S. Army Corps of Engineers, Engineer Research and Development Center, Environmental Laboratory Environmental Processes Chemistry Branch (EPC) and Environmental Processes Risk Assessment Branch (EPR) provided under MIPR No. W81EU632333137 the personnel, labor, materials, equipment, and laboratory facilities to perform chemical and biological testing on samples collected from Eighteenmile Creek Area of Concern (AOC).

The Eighteenmile Creek AOC is located in the town of Olcott, Niagara county, in western New York state. The creek flows from the south and discharges into Lake Ontario, approximately 18 miles east of the mouth of the Niagara River, through Olcott Harbor. The AOC includes Olcott Harbor at the mouth of the creek and extends upstream to the farthest point at which backwater conditions exist during Lake Ontario's highest monthly average lake level.

The Buffalo District personnel were responsible for all sediment sampling. The latitude and longitude of the sampling sites are provided in Table 1. Sampling sites of the areas are located in Figures 1-3 of Appendix M. A global positioning system (GPS) was used to locate the sampling locations. If it was necessary to deviate from any location to obtain a sufficient or representative sample, coordinates of the new location(s) were recorded. The actual GPS location coordinates were recorded in the field notes along with a description of the sample and water depth. Appendix N contains the sampling field logs.

Sediment used as the control sediment was collected from Brown's Lake located on the property of the ERDC site in Vicksburg, MS. Sediment was collected using a hand shovel, collecting approximately the top 10 cm of the sediment. Analytical chemistry was conducted on the sediment in the spring of 2000. Brown's Lake sediment was mainly silty material with 1.8% sand, 98.2% fines (clay and silt), and 0.65% total organic carbon. Concentrations of PAHs, heavy metals, and pesticides were either below detection level or at concentrations not associated with adverse effects to aquatic invertebrates.

As directed by the USACE technical POC, David Melfi, Buffalo District personnel supplied the fifteen sediment samples and five composites (biological unit sediments) directly to the Environmental Processes Chemistry Branch (EPC). EPR performed sediment bioaccumulation testing on four sediments (EBU-1, EBU-2, EBU-3, and EBU-4) using *Lumbriculus variegatus* according to the test conditions specified in the Great Lakes Dredged Material Testing and Evaluation Manual. EPC performed bulk chemical analyses following EPA SW846 methodology for the fifteen sediment samples and five composites. EPC contracted with Severn Trent Laboratory (STL), Knoxville, TN, to analyze the fifteen sediment samples for Dioxin. The USACE ERDC Geotechnical and

Earthquake Engineering Branch Materials Testing Center performed particle sizing analyses according to ASTM Procedure D422 for the five composite samples.

TABLE 1
Eighteenmile Creek Sample Location Coordinates

Sampling Area	Latitude	Longitude
EMC-1	43° 20.302'	78° 43.107'
EMC-2	43° 20.235'	78° 42.999'
EMC-3	43° 20.147'	78° 42.931'
EMC-4	43° 20.042'	78° 42.976'
EMC-5	43° 19.937'	78° 42.950'
EMC-6	43° 19.829'	78° 42.928'
EMC-7	43° 19.723'	78° 42.976'
EMC-8	43° 19.621'	78° 43.027'
EMC-9	43° 19.517'	78° 43.080'
EMC-10	43° 19.442'	78° 43.017'
EMC-11	43° 19.351'	78° 42.973'
EMC-12	43° 19.282'	78° 42.872'
EMC-13	43° 19.191'	78° 42.853'
EMC-14	43° 19.089'	78° 43.005'
EMC-15	43° 19.012'	78° 42.996'

Chemical analysis summary tables for the sediments and *Lumbriculus variegatus* tissues are presented in Appendices A and B. Appendix C contains quality assurance/quality control (QA/QC) summary tables. The chemical analysis reports for the sediments and tissues are located in Appendices D and E. The bioaccumulation results are summarized in Appendix F and Appendix G contains the bioaccumulation results including statistics.

2.0 ANALYTICAL METHODS & CHAIN OF CUSTODY

Chemical analyses were performed using the Eighteenmile Creek sediments and *Lumbriculus variegatus* tissues. Summaries of the sediment and tissue data are located in Appendices A and B. The bioavailability and bioaccumulation can be determined from the chemical analyses of the sediments and *Lumbriculus variegatus* tissues. Review of the historical data from Eighteenmile Creek provided a list of contaminants of concern.

The USACE ERDC Environmental Processes Chemistry Branch performed all chemical analyses except for particle sizing, which was performed by USACE ERDC Geotechnical and Earthquake Engineering Branch, Materials Testing Center and dioxin, which was contracted to Severn Trent Laboratory (STL) of Knoxville, TN. All analyses followed EPA SW846 methodology except for

particle sizing, which followed ASTM Procedure D422. TOC analysis followed EPA SW846 method 9060 modified according to manufacturer suggestions. As found in the scope of work (Appendix I), a summary of the analyses performed including the required reporting limits is listed in Table 2.

Table 2. Required Parameters, Methodology, and Reporting Limits

Parameter	Method	Reporting Limits
	Sediments	mg/kg (dry wt)
Metals (TAL)	6010B	0.050
Mercury	7471	0.025
Total Organic Carbon (TOC)	9060 Modified	500
PCBs, Congeners	8082	0.010
Pesticides	8081A	0.010
Dioxin	9060	0.000002
	Tissue	mg/kg (wet wt)
Metals (TAL)	6020/6010B	0.025
Mercury	7470A	0.0040
PCBs, Summation of Ind. Congeners	8082	0.025
Pesticides	8081A	0.025
Lipid	Van Handel (1985)-IR	1 µg

As outlined in the scope of work (Appendix I), the required chemical and toxicological testing is listed in Table 3. Tables summarizing the chemical analytical results for each sediment and tissue sample are located in Appendices A and B. The laboratory reports, summaries of each analysis, and toxicological data are provided in Volume II.

The fifteen sediment samples and five composite sediments were received at ERDC EPC-Vicksburg on 29 August 2003. Upon arrival at EPC, the coolers were inspected and were found to have intact chain of custody seals. The temperature of the samples upon arrival ranged from 6-15°C. (Appendix J contains copies of the chain-of-custody and cooler receipt records.) Samples were stored at 4°C.

3.0 SEDIMENT CHEMICAL ANALYSIS

The fifteen sediment samples and five composite sediments were received at ERDC EPC-Vicksburg on 29 August 2003. All fifteen sediments (EMC1-15) were sub-sampled for dioxin analysis, mercury, and total organic carbon. The five composites (EBU1-5) were sub-sampled for mercury (Hg) and total organic carbon (TOC). The sub-samples of the sediments and composites were sent on 2

September 2003 to ERDC EPC-Omaha via Fed-Ex overnight express. USACE ERDC EPC-Omaha forwarded the Dioxin samples to Severn Trent Laboratory (STL) of Knoxville, TN on 6 September 2003 via Fed-Ex overnight express. The five composite samples (EBU1-5) were sub-sampled and delivered to USACE ERDC Geotechnical and Earthquake Engineering Branch, Materials Testing Center for particle sizing on 2 September 2003.

TABLE 3
EIGHTEENMILE CREEK
2003 TESTING

	Sediment					Biological					
	Metals	Dioxin	Pest	PCB	TOC	Particle	Lv	Metals	PCB	Pest	Lipid
EMC-1	X	X	X	X	X						
EMC-2	X	X	X	X	X						
EMC-3	X	X	X	X	X						
EBU-1	X		X	X	X	X	X	X	X	X	X
EMC-4	X	X	X	X	X						
EMC-4 QA				X							
EMC-5	X	X	X	X	X						
EMC-6	X	X	X	X	X						
EBU-2	X		X	X	X	X	X	X	X	X	X
EMC-7	X	X	X	X	X						
EMC-8	X	X	X	X	X						
EMC-9	X	X	X	X	X						
EBU-3	X		X	X	X	X	X	X	X	X	X
EMC-10	X	X	X	X	X						
EMC-11	X	X	X	X	X						
EMC-12	X	X	X	X	X						
EBU-4	X		X	X	X	X	X	X	X	X	X
EMC-13	X	X	X	X	X						
EMC-14	X	X	X	X	X						
EMC-15	X	X	X	X	X						
EBU-5	X		X	X	X	X	X	X	X	X	X
TOTALS	20	15	20	21	20	5	5	25	25	25	26

3.1 SEDIMENT QUALITY ASSURANCE /QUALITY CONTROL (QA/QC)

Quality Assurance and Quality Control (QA/QC) data is summarized in Tables 1-2 of Appendix C and included in the analytical reports found in Appendix D. All

QA/QC followed US EPA methodology as described in the scope of work (Appendix J).

All compounds for the analysis of pesticides were within acceptable limits for the method blank, laboratory control sample (LCS) recoveries, surrogates, and relative percent differences (RPDs). The matrix spike/matrix spike duplicate (MS/MSD) for sample 115078 was within limits for all compounds except for low recoveries of aldrin, d-BHC, and a-BHC. The replication of these low recoveries in the MS and MSD indicate a matrix effect. The recovery for DDE was 0% and 13%. The sample concentrations for DDE were significantly higher than spiked amounts resulting in erratic recoveries for these analytes.

For the PCB analysis, the method blank, LCS, LCSD, surrogates, and RPDs were within laboratory control limits. For sample 115018, the MS and MSD recovery values for PCB 1016 were 161% and 164%, which exceeded the laboratory QC limit of 140%. The MS/MSD recovery values for PCB 1260 were within the laboratories QC limits (102% and 104%). The high recoveries for PCB 1016 in the MS/MSD appear to be due to a matrix effect.

All method blanks, LCS, surrogates, and RPDs were within laboratory control limits for the PCB congener analysis. All MS/MSDs were within laboratory control limits except for congeners 18, 31, 44, 49, and 52, which were not reported because sample concentrations were significantly higher than spiked amounts resulting in erratic recoveries for these analytes. All MS/MSD RPDs were <40%. A sample duplicate was extracted and analyzed for site EMC-4 with RPDs <40% for all detectable analytes present in the sample. The calibration for congener 77 had a relative standard deviation (RSD) of 27.1% on the SPB-octyl column; however, the analyte had a linear coefficient of 0.994. Therefore, the reported values that were at or below the response of the low standard were manually calculated.

For the metals analysis, the continuing calibration blanks (CCBs) were below the detection limits except for copper, lead, chromium, cobalt, nickel, zinc, aluminum, iron, sodium, and magnesium. The analytes detected in the CCBs were low concentrations compared to the high sample concentrations, which resulted in minimal sample bias. All RPDs were within laboratory control limits. All analytes in the LCS and MS were within the method control limits (75-125%) except for antimony (Sb) and aluminum (Al) in the MS. The low recovery of antimony is due to the digestion methodology requiring the use of nitric acid. Appendix M contains an article published in Environmental Science & Technology that discusses low antimony recoveries in geological materials using nitric acid for digestions.

The following sections for the sediment analysis address specific analytical problems encountered during the analysis of the samples. Appendix D contains the laboratory reports and corrective action forms for the sediment analyses.

3.2 PESTICIDE ANALYSIS – SEDIMENT

Approximately 15 g-wet weight aliquots of 9 sediments (114792-800) plus method blank, laboratory control spike (LCS), matrix spike, and matrix spike duplicate were extracted by SW846 method 3545 (Accelerated solvent extraction) using hexane/acetone as the extraction solvent on 9 September 2003 and 11 sediments (114801-12) plus method blank, laboratory control sample (LCS), and laboratory control sample duplicate (LCSD) were extracted on 10 September 2003. The extracts were concentrated and cleaned up following a modification of method 3620B (florisil) and concentrated to 5ml for analysis. The extracts were further treated for sulfur following method 3660B using tetrabutylammonium (TBA) sulfite reagent. The samples were extracted and analyzed within holding times.

Pesticides were analyzed on 29 September 2003 and 30 September 2003 following SW846 method 8081 using a Hewlett-Packard 6890 and 5890 gas chromatographs. Each GC has dual capillary columns equipped with dual electron capture detectors. For the 6890 GC, the primary capillary column used for the analysis was a DB-1701 30 meter X 0.32mm ID X 0.52-micron film thickness column and the secondary column was a Restek CLP Pesticide 1 30 meter X 0.32mm ID X 0.25-micron film thickness column. For the 5890 GC, the primary column was a Restek CLP Pesticide 1 30meter X 0.32mm ID X 0.25-micron film thickness column and the secondary column was a Restek CLP Pesticide 2 30 meter X 0.32mm ID X 0.25-micron film thickness column.

A six-point calibration curve for individual standard mix A and B (INDA and INDB) was analyzed on 29 September 2003 on the 6890 GC. The samples (115074-82) were analyzed according to SW846 method 8081 on 29 September 2003. The CCV-1 for INDB had an average difference (D) of 11.6% (first column) and 7.76% (second column) and INDA had an average D of 11.1% and <15%, respectively. CCV-2 for INDA had an average D of 9.30% and <15%, respectively and INDB had an average of 8.77% and <15%, respectively. Using a low standard of 2.5/5.0 ng/ml, wet sample weight of 15.0 g, and an extract volume of 5 ml yielded a detection limit of 0.83/1.67 µg/Kg. Correcting for % solids for the samples yielded detection limits of 1.11-2.44 µg/Kg. The samples were extracted and analyzed within holding times. The blank, LCS, surrogates, and RPDs were within laboratory control limits except as noted. The 115078 matrix spike/matrix spike duplicate (MS/MSD) had low recoveries for aldrin, d-bhc, and a-bhc. The replication of these low recoveries indicated a matrix effect. The MS/MSD recovery for DDE was 0% and 13%. The sample concentrations for DDE were significantly higher than spiked amounts resulting in erratic recoveries for these analytes.

A six-point calibration curve for individual standard mix A and B (INDA and INDB) was analyzed on 24 September 2003 on the 5890 GC. The samples (115083-93) were analyzed according to SW846 method 8081 on 30 September

2003. The CCV-1 for INDA had an average D of 9.11% (first column) and 8.69% (second column) and INDB had an average D of 8.92% and 10.8%, respectively. CCV-2 for INDA had an average D of 18.8% and 9.12% and INDB had an average of 18.9% and 10.4%, respectively. Using a low standard of 2.5/5.0 ng/ml, wet sample weight of 15.0 g, and extract volume of 5 ml yielded a detection limit of 0.83/1.67 µg/Kg. Correcting for % solids for the samples yielded detection limits of 1.21-2.44 µg/Kg. The samples were extracted and analyzed within holding times. The blank, LCS, LCSD, surrogates, and RPDs were within lab control limits.

The pesticide analytical reports are summarized in Appendix A (Table 1) and located in Table 1 of Appendix D.

3.3 POLYCHLORINATED BIPHENYL (PCB) ANALYSIS – SEDIMENT

Approximately 15 g-wet weight aliquots of 1 sediment (115018, EMC-4 QA) plus method blank, laboratory control spike (LCS), LCSD, matrix spike, and matrix spike duplicate were extracted by SW846 method 3545 (Accelerated solvent extraction) on 10 September 2003. The extracts were concentrated and cleaned up following a modification of method 3620B (florisil) and concentrated to 5ml for analysis. The extracts were further treated for sulfur following method 3660B using tetrabutylammonium (TBA) sulfite reagent.

A six-point calibration for PCB1016/1260, PCB1242, and PCB1248 was analyzed beginning on 7 October 2003. CCV-1 had a % difference (D) that was <15% on both the primary and secondary columns for PCB1016/1260. CCV-2 for PCB1016/1260 had a %D that was <15 on both columns. Using a low standard of 25 ng/mL, 5 mL extract volume, and 15.0g wet sample weights yielded approximately an 8.33 µg/Kg detection limit for these samples. Correcting for percent solids yielded sample detection limits of 20.4 µg/Kg.

The samples were extracted and analyzed within holding times. The blank, LCS, LCSD, surrogates, and RPDs were within laboratory control limits. The recovery values for PCB1016 in sample 115018-matrix spike and matrix spike duplicate (MS/MSD) were 161% and 164%, which exceeded the laboratory QC limit of 140%. The recovery values for PCB1260 were 102% and 104% in the MS/MSD. The laboratory control sample/ laboratory control sample duplicate (LCS/LCSD) values were within limits; therefore, the high recovery in the MS/MSD appears to have been due to a matrix effect in the sample.

The PCB analytical reports are summarized in Appendix A (Table 2) and located in Table 2 of Appendix D.

3.4 PCB CONGENER ANALYSIS – SEDIMENT

Approximately 15 g-wet weight aliquots of 9 sediments (114792-800) plus method blank, laboratory control spike (LCS), matrix spike, and matrix spike duplicate were extracted by SW846 method 3545 (Accelerated solvent extraction) using hexane/acetone as the extraction solvent on 9 September 2003 and 11 sediments (114801-12) plus method blank, laboratory control sample (LCS), and laboratory control sample duplicate (LCSD) were extracted on 10 September 2003. The extracts were concentrated and cleaned up following a modification of method 3620B (florisil) and concentrated to 5ml for analysis. The extracts were further treated for sulfur following method 3660B using tetrabutylammonium (TBA) sulfite reagent. The samples were extracted and analyzed within holding times.

The extracts were analyzed on 21 and 24 September 2003 for PCB congeners by SW846 method 8082 using a Hewlett-Packard 5890 gas chromatograph with dual capillary columns equipped with dual electron capture detectors. The primary capillary column used for the analysis was a J&W DB5 30 meter X 0.25mm ID X 0.25-micron film thickness column. The secondary column was a Supelco SPB-octyl 30meter X 0.25mm ID X 0.25-micron film thickness column. Using a low standard of 2 ng/mL, 5mL extract volume, and 15.0g wet sample weights yielded approximately a 0.67- $\mu\text{g}/\text{Kg}$ -detection limit for these samples. Correcting for sample dry weight yielded sample detection limits of 0.89 to 1.95 $\mu\text{g}/\text{Kg}$.

The samples were calculated against a 5 level calibration curve, which was analyzed on 28 August 2003. This curve had an average RSD for all analytes of <20% on both columns. For the analyses on 21 and 24 September 03, all CCVs had an average D of <15%. Pentachloronitrobenzene and 4,4'-dibromobiphenyl were used as internal standards. All tetrachloro-m-xylene (surrogate) recoveries were within laboratory limits. No MS/MSD recoveries were reported for congeners 18, 31, 44, 49, and 52 because sample concentrations were significantly higher than spiked amounts resulting in erratic recoveries for these analytes. All MS/MSD RPDs were <40%. A sample duplicate was extracted and analyzed for site EMC 4 with RPD <40% for all detectable analytes present in the sample. All LCS recoveries and RPDs were within laboratory limits. The calibration for congener 77 had a RSD of 27.1% on the SPB-octyl column; however, the analyte had a linear coefficient of 0.994. Therefore, the reported values that were at or below the response of the low standard were manually calculated.

No data was reported for congeners 15, 87, 153, 171, 159, and 86 due to co-elutions with other congeners on both columns. Congener 97 is reported as a total of 86 and 97. Congener 101 is reported as a total of 101 and 90. Congeners flagged with a C are estimated values because they co-elute with other congeners on one of the columns. The presence of the congener is confirmed but the concentration is not confirmed due to the co-elution.

The congeners present in the sample, EMC-4 QA, were totaled and compared to the total of the PCBs present in the same sample. For comparison purposes, the reporting limit was divided by three and used in the calculation of the total for all congeners and PCBs that were below the laboratory reporting limits. The value of three was chosen because the method detection limit is approximately one third of the reporting limit. The total congeners present in the sample were 269 µg/kg and the total PCBs present in the sample were 759 µg/kg. The RPD was 23.9. Despite the difficulties in reporting some congeners due to co-elution and matrix interferences, the RPD is within the limits established in EM 200-1-3, Appendix I, Shell for Analytical Chemistry Requirements (01 Feb 01). The total congeners reported for the sample represent 35.4% of the total PCBs reported.

The PCB congener analytical reports are summarized in Appendix A (Table 3) and located in Table 3 of Appendix D.

3.5 METALS ANALYSIS - SEDIMENT

The 20 sediment samples (114848-867) were digested on 22 September 2003 by SW846-3050B for Inductively Coupled Plasma Mass Spectrometer/Heated Graphite Furnace (ICPMS/HGA). The 20 sediment samples (114848-867) were digested on 25 September 2003 by SW846-3050B for analysis by Inductively Coupled Plasma (ICP) and antimony (Sb) for analysis by Heated Graphite Furnace (HGA). The digested samples were analyzed on 25-26 September 2003 and 6 October by SW846-6020A using a Perkin-Elmer Elan 6000 ICPMS. Samples, 114848 –114865, were analyzed for Zn, Al, Ca, Fe, Mg, Mn, Ba, and Cr by SW846-6010B on 1 October 2003 using a Perkin-Elmer Optima 3000DV ICP. Samples, 114866-114867, were analyzed by SW846-6010B on 8 October 2003 for Zn, Al, Ca, Fe, Mg, Mn, Ba, and Cr. Samples 114848-65 for antimony (Sb) were analyzed on 10 October 2003 by SW846-7041 using a Perkin-Elmer SIMAA 6000 HGA. All digestions and analyses were within method holding times.

The samples were analyzed against a three level calibration curve. The correlation coefficient for all of the curves was 0.999 or better. The samples were diluted 1/10 to to bring the sample concentrations within the calibration range. Any sample that exceeded the calibration was diluted 1/19 for the final reported value. All continuing calibration verifications (CCVs) were within the ± 10% limits. All continuing calibration blanks (CCBs) were below the detection limits except for copper, lead, chromium, cobalt, nickel, zinc, aluminum, iron, sodium, and magnesium. The analytes detected in the CCBs were low concentrations compared to the high sample concentrations, which resulted in minimal sample bias. All relative percent differences (RPDs) were <20%. All analytes in the LCS and MS were within the method range for analyte recovery of 75-125% except for antimony (Sb) and aluminum (Al) in the matrix spike. The low recovery of antimony is due to the digestion methodology requiring the use of nitric acid. Appendix M contains an article published in *Environmental Science &*

Technology that discusses low antimony recoveries in geological materials using nitric acid for digestions.

The metals analytical reports are summarized in Appendix A (Table 4) and located in Table 4 of Appendix D.

3.6 MERCURY ANALYSIS - SEDIMENT

The 20 sediment samples (114828-47) were received on 3 September 2003 by EPC-Omaha, digested on 18 September 2003, and analyzed on 19 September 2003 following SW846 method 7471A with atomic fluorescence detection. All QC were within method limits.

The mercury analytical reports are summarized in Appendix A (Table 5) and located in Table 5 of Appendix D.

3.7 TOTAL ORGANIC CARBON (TOC) ANALYSIS - SEDIMENT

The 20 sediment samples (114828-47) were received on 3 September 2003 by EPC-Omaha and analyzed on 10 October 2003. The samples were analyzed on a Tekmar/Dohrmann TOC analyzer following SW846 method 9060 with instrument manufacturer modifications. All QC were within method limits.

The TOC analytical reports are summarized in Appendix A (Table 5) and located in Table 5 of Appendix D.

3.8 DIOXIN ANALYSIS – SEDIMENT

The 15 sediment samples (114813-27) were received on 3 September 2003 by EPC-Omaha and were forwarded on 5 September 2003 to Severn Trent Laboratory (STL) of Knoxville, TN. STL received the samples on 6 September 2003 for dioxin analysis by SW846 Method 8290. The samples were extracted on 8-9 September 2003 and analyzed from 16-23 September 2003.

Sample EMC-15 exhibited internal standard recoveries that were outside QC limits (40-135%). The value for 13C-OCDD in sample EMC-15 was 38%. The 10:1 internal standard signal-to-noise ratio criterion was met in all cases. When properly applied, results from isotope dilution analyses are independent of internal standard percent recoveries. Therefore, since the internal standard signal-to-noise ratios were sufficient, the analysis results are not adversely affected.

The MS/MSD for sample EMC-2 was outside control limits (low) for OCDF. All positive 2378-TCDF results were confirmed on a DB-225 chromatography column. The analysis of the sample extract EMC-2, EMC-4, and EMC-10 on the DB-225 column exhibited co-eluting interferences which prevented accurate results. The 2378-TCDF results reported were obtained from the Rtx-5 analysis.

The Rtx-5 column is not isomer specific for 2378-TCDF; therefore, the reported value for 2378-TCDF is considered the highest amount of TCDF present.

The dioxin analytical reports are summarized in Appendix A (Table 6) and located in Table 6 of Appendix D. All Severn Trent narratives and laboratory reports are located in Appendix H.

3.9 PARTICLE SIZING - SEDIMENT

The five composite sediment samples (114787-91) were delivered to Geotechnical and Earthquake Engineering Branch, Materials Testing Center of ERDC-GSL, Vicksburg on 29 August 2003 for particle size analysis. The samples were analyzed on 18 September 2003. The particle size data is summarized in Table 7 (Appendix A) and located in Table 7 of Appendix D. The particle sizing data package is located in Appendix N.

4.0 TISSUE CHEMICAL ANALYSIS

Twenty-five tissue samples (RBU1-5) and three tissue controls were received at ERDC EPC-Vicksburg on 8 December 2003 from bioaccumulation studies performed by ERDC EPR using five Eighteenmile Creek sediments (RBU 1-5) in replicates of five. The samples were received into the lab at 0°C for processing.

The USACE ERDC Environmental Processes Chemistry Branch performed all chemical analyses on the tissues for pesticides, PCB congeners, and metals. All analyses followed EPA SW846 methodology. As found in the scope of work (Appendix H), a summary of the analyses performed is listed above in Table 3. Appendix B contains summary tables of the chemical analyses and Appendix E contains the analytical reports for the chemical analyses.

4.1 QUALITY ASSURANCE /QUALITY CONTROL – TISSUE

Quality Assurance and Quality Control (QA/QC) data is summarized in Tables 3 and 4 of Appendix C and is included in the analytical reports found in Appendix E. All QA/QC followed U.S. EPA methodology as described in the scope of work (Appendix I).

For the pesticide analysis, all method blanks, LCS, MS/MSD, surrogate recoveries, and RPDs were within laboratory control limits except for D-BHC in 116475 MS which had a recovery of 27.2% and for Heptachlor Epoxide in sample 116475 MS/MSD which had a recovery of 33.7% and 34.7%, respectively. The laboratory control limits range from 40-140%.

In the PCB congener analysis, the lowest standard was not used for some of the analytes, which is reflected in the reported detection limits. The calibration curve analyzed on 12 December 2003 had an average RSD of <20% or a $r^2 > 0.99$ for all

analytes on both columns except for congener 70 which had a RSD of 21.2% on the SPB column. All surrogate recoveries were within laboratory control limits. All MS/MSD RPDs were <40% except for congener 138 which had a RPD of 67.3%. All LCS recoveries and RPDs were within laboratory control limits except for no spike recovery due to matrix interferences for congener 187 in LCS 2. Linear calibration was used for congeners 77 and 101 and the reported values that were at or below the response of the low standard were manually calculated.

For the metals analysis, all CCBs were within the $\pm 10\%$ limits. Because the tissue sample replicates were three separate samples versus a split composite, many of the RPDs were not within the 20% limits. The RPDs for 116527 were < 20% for Al, Ag, Be, Cd, Cu, Na, and Se and all other elements were > 20%. The MS for 116527 for Be, Cd, Co, Mn, Ni, and Zn was within the 75 – 125% method range and all other elements were outside of the range. The RPDs for 116538 were < 20% for As, Ag, Cu, Mg, and Tl and all other elements were > 20%. The MS for As, Cu, Mn, Pb, Sb, and Zn were within the 75 – 125% method range and all other elements were outside of the range.

In the metals analysis, the post digest duplicates and spikes were a better indicator of instrument performance for the tissue samples and were analyzed for samples 116527 and 116538. For 116527, the RPD for all elements were <20% and the post digest spike for all elements was within the 75-125% method range. For 116538, the RPD for all elements was <20% except for Al and Zn and the post digest spike for all elements was within the 75-125% method ranges except for Zn.

Low concentrations of Zn, Al, Ba, and Fe were detected in the method blank for samples 116517-116531. Due to the high concentrations detected in the samples, the results are not biased. For samples 116532-116544, Zn, Ba, Mn, and Fe were detected in the method blank, which did not bias the results due to the high concentrations detected in the samples. Low concentrations reported for Cr, Ni, and Co in samples 116517-116531 may be biased high due to the detection of low concentrations of the element in the method blank.

The following sections for tissue analyses address specific analytical problems encountered during the analysis of the *L. variegatus* samples. Appendix E contains the laboratory reports and corrective action forms that provide details concerning various problems found during the tissue analyses.

4.2 PESTICIDE ANALYSIS - TISSUE

Approximately, 1.005-1.703 grams-wet weight aliquots of fifteen tissues (116461-475) plus method blank, laboratory control spike (LCS), 116475 matrix spike and matrix spike duplicate (MS/MSD) were extracted on 17 December 2003 and thirteen tissues (116476-88) plus method blank and LCS were extracted on 19 December 2003. All tissue extractions followed SW846 method 3550 (Sonication Extraction Method) modified for small sample amounts. The extracts were

concentrated and cleaned up following method 3620B (florisil). The samples were extracted and analyzed within holding times.

The extracts were analyzed beginning on 29 December 2003 by SW846 method 8081 using a Hewlett-Packard 5890 gas chromatograph with dual capillary columns equipped with dual electron capture detectors. The primary capillary column used for the analysis was a Restek CLP Pesticide 1 30 meter X 0.32 mm ID X 0.52-micron film thickness column. The secondary column was a Restek CLP Pesticide 2 30 meter X 0.32 mm ID X 0.25-micron film thickness column. Using a low standard of 2.5/5.0 ng/mL, 1 mL extract volume, and 1.0g wet sample weight yielded approximately a 2.5/5.0- $\mu\text{g}/\text{Kg}$ -detection limit for these samples. Sample detection limits based on actual wet tissue weights that were extracted were 1.47/2.94 to 2.5/5.0 $\mu\text{g}/\text{Kg}$ for the tissues.

A six-point calibration curve for individual standard mix A and B (INDA and INDB) was analyzed on 23 December 2003. The first continuing calibration verification (CCV-1) for INDA had an average difference (D) of 10.1% on the primary column and 5.01% on the confirmation column and the %D for INDB was 9.52% for the primary column and 6.47% for the confirmation column. CCV-2 for INDA had an average D < 15% on both columns and INDB had an average D of 8.55% and 6.04%. The CCV-3 for INDA had an average D of 12.8% and 10.3%, and INDB had an average D of 11.0% and 8.82%. For INDA, CCV-4 had an average D of 12.0% and 9.30% and 12.8% and 10.2% for INDB. CCV-5 had an average D of 14.2% and 11.1% for INDA and INDB had an average D of 11.2% and 8.16%.

All method blanks, LCS, MS/MSD, surrogate recoveries, and relative percent differences (RPDs) were within laboratory control limits except for 116475 MS D-BHC which had a recovery of 27.2% and for 116475 MS/MSD Heptachlor Epoxide which had a recovery of 33.7% and 34.7%, respectively. The laboratory reporting limits range from 40-140%.

The pesticide analytical reports are summarized in Appendix B (Table 1) and located in Table 1 of Appendix E.

4.3 PCB CONGENER ANALYSIS – TISSUE

Approximately, 1.00-1.79 grams-wet weight aliquots of fifteen tissues (116489-503) plus method blank, laboratory control spike (LCS), 116497 matrix spike and matrix spike duplicate (MS/MSD) were extracted on 10 December 2003 and 13 tissues (116504-16) plus method blank and LCS were extracted on 15 December 2003. All tissue extractions followed SW846 method 3550 (Sonication Extraction Method) modified for small sample amounts. The extracts were concentrated and cleaned up following method 3630C (silica gel). The samples were extracted and analyzed within holding times.

The extracts were analyzed on 12 and 17 December 2003 for PCB congeners by SW846 method 8082 using a Hewlett-Packard 5890 gas chromatograph with dual capillary columns equipped with dual electron capture detectors. The primary capillary column used for the analysis was a J&W DB 5 30 meter X 0.25 mm ID X 0.25-micron film thickness column. The secondary column was a Supelco SPB-octyl 30 meter X 0.25 mm ID X 0.25-micron film thickness column. Using low standards of 1 or 2 ng/mL, 1 mL extract volume, and 1.0g wet sample weights yielded approximately a 1 or 2- μ g/Kg-detection limit for these samples.

The samples were analyzed against a six level calibration curve, which was analyzed on 12 December 2003. For some analytes, the lowest standard was not used which is reflected in the reported detection limits. This curve had an average RSD of <20% or an r^2 >0.99 for all analytes on both columns except for congener 70 which had a RSD of 21.2% on the SPB column. For the analyses on 12 and 17 December 03, all CCVs had an average D of <15%. Pentachloronitrobenzene and 4,4'-dibromobiphenyl were used as internal standards. All tetrachloro-m-xylene (surrogate) recoveries were within laboratory limits. All MS/MSD RPDs were <40% except for congener 138 which had a RPD of 67.3%. All LCS recoveries and RPDs were within laboratory limits. No spike recovery for congener 187 was reported for LCS 2 due to interferences. Linear calibration was used for congeners 77 and 101 and the reported values that were at or below the response of the low standard were manually calculated.

No data was reported for congeners 15, 141, 153, 156, 159, 171, and 187 due to coelutions with other congeners on both columns except for samples that were at or below the laboratory reporting limit. Congener 101 is reported as a total of 101 and 90, and congener 97 is reported as a total of 97 and 86. Congeners flagged with a C are estimated values because they coelute with other congeners on one of the columns. The presence of the congener is confirmed but the concentration is not due to the coelution.

The PCB congener analytical reports are summarized in Appendix B (Table 2) and located in Table 2 of Appendix E.

4.4 METALS ANALYSIS - TISSUE

The twenty-eight tissue samples (EPC sample id: 116517-44) ranging in wet weight from 0.50 - 1.0 g each were digested by SW846-3050B on 10 December 2003 for HGA/ICPMS. The digested samples were analyzed on 29 December 2003 by SW846-6020A using a Perkin-Elmer Elan 6000 ICPMS. Al and Zn were analyzed on 6 January 2004 by SW846-6010A using a Perkin-Elmer Optima 3000DV ICP.

The samples were analyzed against a three level calibration curve. The correlation coefficient for all of the curves was 0.999 or better. The samples were diluted 1:9 - 1:99 to obtain concentrations within the calibration curve. All CCVs were

within the $\pm 10\%$ limits. Because the tissue sample replicates were three separate samples versus a split composite, many of the RPDs were not within the 20% limits. The RPDs for 116527 were $< 20\%$ for Al, Ag, Be, Cd, Cu, Na, and Se and all other elements were $> 20\%$. The MS for 116527 for Be, Cd, Co, Mn, Ni, and Zn was within the 75 – 125% method range and all other elements were outside of the range. The RPDs for 116538 were $< 20\%$ for As, Ag, Cu, Mg, and Tl and all other elements were $> 20\%$. The MS for As, Cu, Mn, Pb, Sb, and Zn were within the 75 – 125% method range and all other elements were outside of the range.

Post digest duplicates and spikes were a better indicator of instrument performance for the tissue samples and were analyzed for samples 116527 and 116538. For 116527, the RPD for all elements was $< 20\%$ and the post digest spike for all elements was within the 75 – 125% method range. For 116538, the RPD for all elements was $< 20\%$ except for Al and Zn and the post digest spike for all elements was within the 75 – 125% method range except for Zn.

Due to the high concentrations detected in the samples (116517-531) for Zinc, Aluminum, Barium, and Iron, the results are not biased although low concentrations of these metals were detected in the method blank. Due to the high concentrations detected in the samples (116532-544) for zinc, barium, manganese, and iron, the results are not biased although low concentrations of these metals were detected in the method blank. Low concentrations reported for chromium, nickel, and cobalt in samples 116517-531 may be biased high due to the detection of low concentrations of the element in the method blank.

The metals analytical reports are summarized in Appendix B (Table 3) and located in Table 3 of Appendix E along with laboratory corrective action forms.

4.5 MERCURY ANALYSIS – TISSUE

The twenty-eight tissue samples (116545-72) were received on 8 December 2003 by ERDC EPC – Vicksburg, digested on 17 December 2003, and analyzed on 23 December 2003 following SW846 method 7471A with atomic fluorescence detection using a seven level calibration curve, which had correlation coefficients of 0.999. All CCVs were within the $\pm 10\%$ limits. All CCBs were below the detection limits. All quality control was within method limits as well as digestion and analysis holding times

The Mercury analytical reports are summarized in Appendix B (Table 4) and located in Table 4 of Appendix E.

BIOACCUMULATION TESTS

Materials and Methods

5.1 INTRODUCTION

At the request of the United States Army Corps of Engineers Buffalo District, ERDC Vicksburg Environmental Laboratory Environmental Processes Risk Assessment Branch (EPR) performed bioaccumulation and toxicity tests with composite sediment samples (EBU-1, EBU-2, EBU-3, EBU-4, and EBU-5) collected from the Eighteenmile Creek. The 28-day tests were performed to measure the bioaccumulation of sediment contaminants in the benthic oligochaete *Lumbriculus variegatus*.

5.2 GENERAL TEST METHODS

Lumbriculus variegatus 28-d bioaccumulation test for sediments was conducted according to guidelines provided in the USEPA/U.S. Army Corps of Engineers 1998 *Great Lakes Dredged Material Testing and Evaluation Manual* (USEPA/USACE 1998). Contaminant whole-tissue residues in *Lumbriculus variegatus* were determined following twenty-eight-day sediment exposures. Adequate exposure conditions were maintained using an intermittent flow system for overlying water renewal.

5.3 TEST ORGANISMS

The freshwater oligochaete *Lumbriculus variegatus* was used in the 28-day bioaccumulation experiment. Organisms were obtained from a commercial vendor (Aquatic Bio Systems Inc., Fort Collins, Colorado). Flow-through culture conditions were maintained according to standard procedures (USEPA 2000).

5.4 OVERLYING WATER

De-chlorinated tap water filtered through paper and carbon filters was used for culturing the organisms and in bioaccumulation tests.

5.5 TEST SEDIMENTS

The USACE-Buffalo District personnel collected sediment samples from Eighteenmile Creek on 22 October 2003. The site sediment samples were received at ERDC EPC-Vicksburg on 29 August 2003. Upon arrival at EPC, the coolers were inspected and were found to have intact chain of custody seals. The temperature of the samples upon arrival ranged from 6-15°C. The samples labeled as EBU-1, EBU-2, EBU-3, EBU-4, and EBU-5 and were submitted to EPR for the initiation of bioaccumulation testing. Sediment samples were stored at 4°C.

5.6 LABORATORY CONTROL SEDIMENTS

Laboratory control sediment was collected from Brown's Lake located at the Waterways Experiment Station, ERDC-Vicksburg. Surface sediment was collected using a hand shovel, placed in 5 gallon plastic buckets, and stored in a cold room at 4°C until use. Brown's Lake sediment is mainly a silty material with 1.8% sand, 98.2% fines (clay and silt), and 0.65% total organic carbon. Concentrations of PAHs, heavy metals, and pesticides were either below detection level or at concentrations not associated with adverse effects to aquatic invertebrates.

5.7 SEDIMENT EXPOSURE

Sediment exposures were conducted under flow-through conditions in box aquaria (31.5 x 18 x 10.5 cm). Five replicates of test and control sediments were used. The day prior to test initiation, treatment sediments were removed from cold storage and mixed for 15 minutes with a laboratory impeller mixer. Thoroughly homogenized sediments were added to each aquarium to a final thickness of 3 cm (1.7 L). A water splitter chamber delivered test water provided by an automated water delivery system to the test chambers every 12 hours (1600 ml/cycle) providing two volume exchanges per day. The sediment was allowed to settle for 24 h prior to addition of test animals under the overlying exchange regime described above. Light aeration was provided to maintain adequate dissolved oxygen concentrations.

Animals retrieved from the mass culture were transferred to cultured bowls. Clusters of test animals were removed, blotted, weighed carefully and rapidly to minimize injury and desiccation, and transferred to beakers, approximately 5-g per beaker. To initiate the bioaccumulation test, worms from each beaker (5-g) were added to each chamber. Temperature was maintained at 23±1°C. The photoperiod was maintained at 16 hours of light and 8 hours of darkness per day under cool-white fluorescent light. Light trickle aeration was maintained for the duration of the exposure period. Overlying water quality parameters (conductivity, hardness, pH, alkalinity, temperature, ammonia concentration, and dissolved oxygen concentration) were measured at test initiation and termination. Temperature and dissolved oxygen concentration were monitored daily. Total ammonia was measured twice weekly.

All water quality parameters were measured from one replicate per sediment sample for all sampling periods. At the end of the 28-day test period, test sediments were sieved through a 0.5-mm mesh screen. The material retained on the screen was transferred to a white shallow pan to facilitate separation of worms from debris. Surviving worms were transferred to clean water in glass bowls for 12 hours to deplete the contents of their guts following current recommendation (USEPA 2000). From each replicate, five worms were blotted to remove excess water, transferred to pre-weighed bead-beating vials, weighed and frozen at -20°C

for total lipids analysis. Remaining animals were collected, blotted to remove excess water, weighed to determine wet biomass, transferred to suitable glass containers, and frozen at -20°C for chemical analysis. Tissue samples were split according to the required chemical analysis (Table 2). For each replicate, target tissue wet weights for chemical analysis were 1-g for pesticides, 1-g for PCB congeners, 0.5g for metals, and 0.5g for mercury.

5.8 LIPID ANALYSIS

Pre-exposure total lipid content was determined in four replicates using culture worms archived at test initiation. Total lipid content at exposure termination was determined from a subset of worms removed from test and control sediments. Lipid analysis was conducted using the Van Handel (1985) colorimetric method developed for small invertebrates (Lotufo et al. 2000; Landrum et al. 2002). Tissue samples (five to ten whole individual worms) were homogenized in 1.5 ml of chloroform/methanol (1:1. v/v). Homogenates were transferred to 13 x 100 mm tubes and centrifuged for 10 min at 1000 g. After recording the total volume, 0.5 ml of the supernatant was transferred to a new 13 x 100 mm tube and placed in a heating block at 100°C until all the solvent had evaporated. Concentrated sulfuric acid (0.2 ml) was then added and the tubes were re-heated at 100°C for 10 min. After cooling, 4.8 ml of vanillin reagent was added. Vanillin reagent was prepared by dissolving 600 mg of vanillin in 100 mL of hot water and adding 400 ml of 85% phosphoric acid. After 5 min, samples were read in a spectrophotometer at 490 nm against a reagent blank. Lipid content was derived from a calibration line obtained using samples of 50, 100, 200, 300 and 400 µg of soybean oil and the procedure described above.

Bioaccumulation Results and Discussion

6.1 OVERLYING WATER CHARACTERISTICS

Water quality parameters measured at initiation, termination and throughout the sediment exposure are reported in Table 4. The overlying water temperature in the test chambers ranged to between 20.1 and 23.6 °C. The temperature was maintained within the required range of 23±1°C during most of the experiment. Dissolved oxygen remained above 40% saturation throughout the exposure, with concentrations ranging from 5.0 to 9.0 mg/L. The pH ranged from 7.3 to 8.3 at experiment initiation and from 7.0 to 8.5 at experiment termination. Ammonia concentrations were high at experiment initiation (1 – 2 mg/L) for the EBU-4 and EBU-5 sediments but dropped to less than 1 mg/L on the second day of exposure and remained low thereafter for all sediments. The overall range of alkalinity values was from 105 to 235 mg/L as CaCO₃. The overall range of hardness values was from 100 to 250 mg/L as CaCO₃.

Table 4. Water quality parameters in the *Lumbriculus variegatus* 28-d test.

	Temperature (°C)		D.O. (mg/L)		pH		Ammonia (mg/L)	Alkalinity (mg/L CaCO ₃)	Hardness (mg/L CaCO ₃)
	Min	Max	Min	Max	Min	Max			
Day 0									
Control	20.1	20.5	6.0	8.6	8.1	8.2	<1	180	175
EBU-1	20.3	20.6	7.2	8.6	7.9	8.2	<1	110	160
EBU-2	20.0	20.6	7.4	8.7	7.8	8.1	<1	110	165
EBU-3	20.3	20.6	5.0	8.3	7.3	8.2	<1	105	145
EBU-4	20.1	20.6	7.1	8.7	7.7	8.3	1	130	170
EBU-5	20.3	20.5	6.0	8.0	7.7	8.0	2	235	250
Day 28									
Control	23.0	23.3	7.5	8.1	7.6	8.1	1	160	100
EBU-1	23.2	23.6	8.0	8.3	7.1	8.1	1	170	145
EBU-2	23.2	23.5	8.2	8.7	7.1	8.1	<1	110	170
EBU-3	23.0	23.5	7.2	8.6	7.0	7.7	<1	120	180
EBU-4	23.0	23.5	8.0	8.6	7.2	8.3	<1	130	140
EBU-5	23.0	23.4	8.1	8.7	7.2	8.5	1	150	140
Daily									
Control	19.7	22.6	7.0	9.1			1		
EBU-1	19.6	22.3	5.2	8.9			1		
EBU-2	19.7	22.2	6.1	8.9			<1		
EBU-3	19.8	22.1	6.2	8.9			<1		
EBU-4	19.8	21.2	5.9	9.0			<1		
EBU-5	19.8	21.6	5.1	9.0			1		

6.2 FINAL BIOMASS

Total worm biomass at exposure termination ranged from 3.0 to 9.1 g for the control and test sediments (Appendix G, Table 1). Target tissue masses for chemical analysis were obtained from all test chambers.

6.3 TOTAL LIPID CONTENT

The mean dry-to-wet-weight ratio for pre-exposure worms, measured in three replicates, was 0.164 (standard deviation = 0.03) (Appendix G, Table 3). Dry-to-wet-weight ratio of exposed worms was not determined. The lipid content (percent of wet-weight) of pre-exposed worms and worms exposed to laboratory control, EBU-1, EBU-2, EBU-3, EBU-4, and EBU-5 sediments is reported in Appendix G, Table 2. Mean pre-exposure lipid content was 0.71 %. Mean total lipid content was similar for pre-exposed worms and control worms (0.78 %) than for those exposed to test sediments (0.96 – 1.69 %). Overall, lipid values derived

in this study were lower than values previously derived for *L. variegatus* (Table 5).

Table 5. Lipids Determined in Various Studies

Average % lipids	Wet or dry wt.	Estimated % of wet wt.*	Reference
0.6 – 0.7%	Wet		This study
0.6	Wet		Brunson et al. 1998
1.2	Wet		Pickard et al. 2001
7.7	Dry	1.3	Leppanen and Kukkonen 2000
15.0	Dry	2.6	Loonen et al. 1997
8.0	Dry	1.4	Kukkonen and Landrum 1994
12.2	Dry	2.1	Landrum et. al. 2002

Estimated using dry-to-wet wt. ratio of 0.17.

6.4 CHLORINATED PESTICIDES BIOACCUMULATION

Tissue samples from worms exposed to laboratory control sediments and test sediments EBU-1, EBU-2, EBU-3, EBU-4, and EBU-5 were submitted to EPC for chlorinated pesticides analysis. The concentration of chlorinated pesticides was below the laboratory reporting limit (BRL) in most samples (Appendix G, Table 4). Concentrations were labeled as BRL when the concentration was lower than one-third of the reporting limit (RL). Concentrations lower than the RL but higher than the method detection limits were labeled as “J” values. For calculation of means and standard deviations of chlorinated pesticides concentrations, “J” concentrations were used unchanged and RL values (< values) were divided by three to represent the upper end of the range of the actual analyte concentration based upon method detection limits. Therefore, concentrations assigned to BRL samples are likely overestimates of the actual concentrations.

Tissue concentrations were below detection limit in all control and test sediment samples for Aldrin, A-BHC, B-BHC, D-BHC, pp-DDT, Dieldrin, B-Endosulfan, Endrin Aldehyde, Heptachlor Epoxide, Chlordane, Toxaphene, alpha Chlordane, and gamma Chlordane. For control samples, all compounds analyzed were below reporting limits except for B-Endosulfan in one replicate, Aldrin and Endosulfan Sulfate in a different replicate, and A-BHC and Endosulfan Sulfate in a third replicate. The compound pp-DDE was detected in worms exposed to all test sediments. Detectable concentrations of pp-DDT and Heptachlor were observed in some worm samples for EBU-1, EBU-2, EBU-4, and EBU-5 at concentrations typically lower than twice the RL. Detectable concentrations of A-BHC, B-BHC and Endrin were observed in worms exposed to the EBU-3 sediment only. Detectable concentrations of Endosulfan Sulfate were observed in worms exposed to the EBU-3 and EBU-4 sediments only.

6.5 PCB CONGENER BIOACCUMULATION

Tissue samples from worms exposed to laboratory control sediments and test sediments EBU-1, EBU-2, EBU-3, EBU-4, and EBU-5 were submitted to EPC for PCB congener analysis. The concentration of PCB congeners was below the laboratory reporting limit (BRL) in several samples (Appendix G, Table 5). Concentrations were labeled as BRL when the concentration was lower than one-third of the reporting limit (RL). Concentrations lower than the RL but higher than the method detection limits were labeled as “J” values. For calculation of means and standard deviations of PCB congener concentrations, “J” concentrations were used unchanged and RL values (< values) were divided by three to represent the upper end of the range of the actual analyte concentration based upon method detection limits. Therefore, concentrations assigned to BRL samples are likely overestimates of the actual concentrations.

Tissue concentrations of all congeners except for PCB 49, PCB 52, and PCB 187 were below laboratory reporting limits in control samples. Tissue concentrations of PCB 15, PCB 18, PCB 40, PCB 44, PCB 49, PCB 52, PCB 60, PCB 77, PCB 87, PCB 97, PCB 101, PCB 103, PCB 105, PCB 118, PCB 128, PCB 138, PCB 151, and PCB 180 were above method detection limits in every sample for all test sediments. Tissue concentrations of PCBs 201, 203, and 206 were above method detection limits in most samples for all test sediments. Tissue concentrations of PCB 141, PCB 167, PCB 182, PCB 194, PCB 195, and PCB 196 were above method detection limits in only a few of the samples analyzed for all test sediments. Tissue concentrations of PCB 54, PCB 103, PCB 121, PCB 143, PCB 154, PCB 155, PCB 173, PCB 189, PCB 191, and PCB 196 were below laboratory-reporting limits in every sample for all test sediments. Tissue concentrations were not obtained for the congeners PCB 15, PCB 141, PCB 153, PCB 156, PCB 159, PCB 171 and PCB 187 for all test sediment samples.

Overall, mean tissue concentrations of PCB congeners were highest for test sediments EBU-3 and EBU-4 and lowest for EBU-5 (Appendix F, Table 5). The variation in PCB congener concentrations among replicates was typically low.

6.6 HEAVY METAL BIOACCUMULATION

Tissue samples from worms exposed to laboratory control sediments and test sediments EBU-1, EBU-2, EBU-3, EBU-4, and EBU-5 were submitted to EPC for Target Analyte List (TAL) Metals and mercury (Hg) analysis. The concentration of some metals was below the laboratory reporting limit (BRL) in several samples (Appendix G, Table 6). Concentrations were labeled as BRL when the concentration was lower than one-third of the reporting limit (RL). For calculation of means and standard deviations of metal concentrations, RL values (< values) were divided by three to represent the upper end of the range of the actual analyte concentration based upon method detection limits. Therefore,

concentrations assigned to BRL samples are likely overestimates of the actual concentrations.

Overall, mean tissue concentrations of the metals As, Se, Ba, Mg, K, and Na, were similar for site and control sediments (Appendix F, Table 6). The mean tissue concentration of the metals Sb, Be, Cr, Cu, Pb, Ni, Ag, Tl, Zn, Al, Ba, Ca, Co, Fe, Mg, Mn, K, Na, and V were higher for test sediments than for the control sediment and were typically highest for the EBU-3 and EBU-4 sites (Appendix F, Table 6). The tissue concentration of Hg in control and test sediments was low and similar among samples except for one sample for the EBU-1 site (Appendix F, Table 6). The variation in heavy metal concentrations among replicates was typically low.

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