

Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
 Portland Harbor Superfund Site
 Surface Sediment – Downtown/Upriver

Laboratory: TestAmerica Laboratories, Incorporated, Seattle, WA

Laboratory Groups: 580-80213-5, 580-80213-6, and 580-80213-9

Analyses: Petroleum Hydrocarbons, Metals, Total Organic Carbon (TOC), Tributyltin, Polycyclic Aromatic Hydrocarbons (PAHs), bis(2-Ethylhexyl)phthalate, Total Solids, and Grain Size

Validation Level: Stage 2A

AECOM Project

Number: 60566335, Task #2.12

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SUMMARY

The data quality review of 2 surface sediment samples and one rinsate blank collected on September 7, 2018, has been completed. Samples were analyzed for total petroleum hydrocarbons (TPHs, diesel-range and motor oil-range) by Washington State Department of Ecology (Ecology) Method NWTPh-Dx; metals by United States Environmental Protection Agency (EPA) Method 6020B (arsenic, cadmium, copper, lead, and zinc), EPA Method 7471A (mercury in sediments), and EPA Method 7470A (mercury in waters); TOC by EPA Method 9060 (sediments) and Standard Method (SM) 5310B (waters); tributyltin by Krone et al.; PAHs by EPA Method 8270D modified by selected ion monitoring (SIM); bis(2-ethylhexyl) phthalate by EPA Method 8270D; total solids by American Society for Testing and Materials (ASTM) Method D-2216; moisture content at 70 degrees Celsius (°C); and/or grain size by ASTM Method D7928/D6913 by TestAmerica Laboratories, Incorporated (TA) located in Tacoma, Washington. The analyses were performed in general accordance with the methods specified in EPA's *Test Methods for Evaluating Solid Waste (SW-846)*, Ecology's *Analytical Methods for Petroleum Hydrocarbons*, June 1997, Standard Methods for the Examination of Water and Wastewater, *Annual Book of ASTM Standards*, ASTM, Philadelphia, Pennsylvania, and Krone CA et al., *A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound*, Marine Environmental Research, 1989. The laboratory provided level 2 and level 4 data packages containing sample results, and associated quality assurance (QA) and quality control (QC) data, preparation logs, and raw instrument outputs (where applicable). The following samples are associated with laboratory groups 580-80213-5, 580-80213-6, and 580-80213-9:

Sample ID	Laboratory ID
PDI-SG-B431	580-80213-1
PDI-SG-B479	580-80213-2
PDI-RB-VV-090718 (rinsate blank)	580-80213-3

Data validation is based on method performance criteria and QC criteria documented in the *Quality Assurance Project Plan (QAPP)*, dated March 23, 2018, as amended. If data qualification was required, data were qualified based on the definitions and use of qualifying flags outlined in the EPA documents *USEPA National Functional Guidelines for Organic Superfund Methods Data Review*, January 2017, and *USEPA National Functional Guidelines for Inorganic Superfund Methods Data Review*, January 2017. Data qualifiers assigned to this sample set are included in Table 1.



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SAMPLE RECEIPT

Upon receipt by TA, the sample jar information was compared to the chain-of-custody (COC) and the cooler temperature was recorded. The cooler was received at a temperature within the EPA-recommended limits of greater than 0°C and less than or equal to 6°C. The rush grain size was reported in laboratory group 580-80213-5 on 9/20/18. The rinsate blank was reported in laboratory group 580-80213-6 on 9/27/18. Samples PDI-SG-B431 and PDI-SG-B479 were authorized for the remaining analyses on 9/26/18 and 10/3/18, respectively, and reported in laboratory group 580-80213-9 on 10/22/18. TA froze samples PDI-SG-B431 and PDI-SG-B479 on 9/11/18 to extend the holding time.

ORGANIC ANALYSES

Samples were analyzed for TPHs, tributyltin, PAHs, and bis(2-ethylhexyl)phthalate by the methods identified in the introduction to this report.

1. Holding Times – Acceptable
2. Blanks – Acceptable except as noted below:

General – One rinsate blank was submitted with this laboratory group. TPHs, tributyltin, PAHs, and bis(2-ethylhexyl)phthalate were not detected in this rinsate blank.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – bis(2-Ethylhexyl)phthalate was detected in the method blank associated with prep batch 286343 (4.07 ug/kg) at a concentration between the method detection limit (MDL) and reporting limit. bis(2-Ethylhexyl)phthalate was detected in PDI-SG-B431 and PDI-SG-B479 at concentrations between the MDLs and the reporting limits in samples with elevated reporting limits due dilutions required prior to analysis; therefore, the results were qualified as estimated and flagged 'J' based on these method blank results.

3. Surrogates – Acceptable
4. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Acceptable except as noted below:

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The relative percent difference (RPD) for the LCS/LCSD pair (46%) associated with prep batch 283783 exceeded the control limit of 35%. The percent recoveries in the LCS and LCSD were acceptable; therefore, data were not qualified based on the elevated RPD.

PAHs by EPA Method 8270D-SIM – The percent recovery for anthracene in the LCS (71%) associated with prep batch 286335 was below the control limits of 73-125%. The results for anthracene in PDI-SG-B431 and PDI-SG-B479 were qualified as estimated and flagged 'UJ' based on the LCS recovery.

5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Acceptable

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – An MS/MSD was not performed using a sample from this laboratory group. Accuracy was assessed using the LCS. Precision was assessed using the LCS/LCSD where applicable.

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PAHs by EPA Method 8270D-SIM – An MS/MSD was not performed using a sample from this laboratory group. Accuracy was assessed using the LCS. Precision was assessed using the LCS/LCSD where applicable.

TPHs by Method NWTPH-Dx – An MS/MSD was not performed using a sample from this laboratory group. Accuracy was assessed using the LCS. Precision was assessed using the LCS/LCSD where applicable.

Tributyltin by Krone et al. – An MS/MSD was analyzed using PDI-SG-B479. The RPD for the MS/MSD pair (31%) exceeded the control limit of 20%. The percent recoveries in the MS and MSD were acceptable; therefore, data were not qualified based on the elevated RPD.

6. Laboratory Duplicate

TPHs by Method NWTPH-Dx – A laboratory duplicate was not performed using a sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSD.

7. Reporting Limits – Acceptable except as noted below:

General – Analyte concentrations detected between the MDLs and the reporting limits are reported by the laboratory with 'J' flags. Laboratory 'J'-flagged results are considered estimated results. As the results are between the MDLs and the reporting limits, there is a greater level of uncertainty associated with the numerical results.

PAHs by EPA Method 8270D-SIM – The reporting limits for both sediment samples in this laboratory group were raised because of the dilutions that were required prior to analysis due to the nature of the sample matrix. The reporting limits for multiple analytes in both samples reported as not detected exceeded the cleanup level for carcinogenic PAHs (12 ug/kg) but the MDLs did not.

8. Other Items of Note:

PAHs by EPA Method 8270D-SIM – The laboratory noted that the percent difference (%D) for benzo[k]fluoranthene in the continuing calibration verification (CCV) associated with analytical batch 284016 was outside the control limits of $\pm 20\%$ (high). Benzo[k]fluoranthene was not detected in PDI-RB-VV-090718; therefore, data were not qualified based on the elevated %D.

TPHs by Method NWTPH-Dx – The laboratory indicated that the diesel-range hydrocarbon elution patterns were later than the typical diesel pattern in PDI-SG-B431 and PDI-SG-B479.

The laboratory noted that the %D for the surrogate o-terphenyl in the CCV associated with analytical batch 284335 was outside the control limits of $\pm 15\%$ (high). As the surrogate recovery in PDI-RB-VV-090718 was acceptable, data were not qualified based on this elevated surrogate %D.

METALS ANALYSES

Samples were analyzed for metals by the methods identified in the introduction to this report.

1. Holding Times – Acceptable

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2. Blanks – Acceptable except as noted below:

General – One rinsate blank was submitted with this laboratory group. Arsenic (0.00022 mg/L) and zinc (0.0026 mg/L) were detected in this rinsate blank at concentrations between the MDLs and reporting limits. Sediment data were not qualified based on rinsate blank results.

3. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Acceptable

4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) and Post-Digestion Spike (PDS, where applicable)

General – MS/MSDs were not performed using a sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSDs.

5. Laboratory Duplicate

General – Laboratory duplicates were not performed using a sample from this laboratory group. Precision was assessed using the LCS/LCSDs.

6. Serial Dilution

Metals by Method 6020B – A serial dilution was not performed using a sample from this laboratory group. Precision was assessed using the LCS/LCSDs.

7. Reporting Limits – Acceptable

General – One or more results in multiple samples were reported at concentrations between the reporting limits and the MDLs and were flagged 'J' by the laboratory. As described above, laboratory 'J'-flagged results are considered estimated results.

CONVENTIONAL ANALYSES

Samples were analyzed for TOC and total solids by the methods identified in the introduction to this report.

1. Holding Times – Acceptable except as noted below:

Total Solids by ASTM Method D-2216/Moisture Content at 70°C – The 7-day holding time indicated for total solids in the QAPP was exceeded for both sediment samples by 27-28 days. No data qualifiers were assigned based on this holding time exceedance.

2. Blanks – Acceptable except as noted below:

TOC by Method 9060 – One rinsate blank was submitted with this laboratory group. TOC (0.75 mg/L) was detected in this rinsate blank at a concentration between the MDL and the reporting limit. Sediment data were not qualified based on rinsate blank results.

TOC by SM 5310B – TOC was detected in the method blank associated with analytical batch 284442 (0.423 mg/L) at a concentration between the MDL and the reporting limit. TOC was detected in PDI-RB-VV-090718 at a concentration between the MDL and the reporting limit;



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therefore, TOC was qualified as not detected and flagged 'U' at the reporting limit based on this method blank result.

3. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Acceptable
4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

TOC by Method 9060 – An MS/MSD was not performed using a sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSD.

5. Laboratory Replicate

General – Laboratory duplicates were not performed using a sample from this laboratory group. Precision were assessed using the LCS/LCSDs where applicable.

6. Reporting Limits – Acceptable except as noted below:

TOC by SM 5310B – TOC was detected at a concentration between the reporting limit and the MDL and flagged 'J' by the laboratory in PDI-RB-VV-090718, but this result was qualified as not detected based on the associated method blank.

GRAIN SIZE ANALYSES

Samples were analyzed for grain size by the methods identified in the introduction to this report. The data were reviewed to confirm that the required grain size fractions identified in the QAPP were reported for each sample.

1. Laboratory Duplicate

The laboratory performed duplicate analysis at a rate of 1 per 20 samples per their internal requirements. A laboratory duplicate was not performed using a sample from this laboratory group.

OVERALL ASSESSMENT OF DATA

The data reported in this laboratory group, as qualified, is considered usable for meeting project objectives. The completeness for laboratory groups 580-80213-5, 580-80213-6, and 580-80213-9 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Downtown/Upriver
TestAmerica Laboratory Groups: 580-80213-5, 580-80213-6, and 580-80213-9

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B431	580-80213-1	SW8270D	Bis(2-ethylhexyl)phthalate	63 J	ug/kg	63 J	bl
PDI-SG-B431	580-80213-1	SW8270DSIM	Anthracene	15 U	ug/kg	15 UJ	I
PDI-SG-B479	580-80213-2	SW8270D	Bis(2-ethylhexyl)phthalate	92 J	ug/kg	92 J	bl
PDI-SG-B479	580-80213-2	SW8270DSIM	Anthracene	15 U	ug/kg	15 UJ	I
PDI-RB-VV-090718	580-80213-3	SM5310B	Total Organic Carbon	0.75 J	mg/L	1.0 U	bl

Notes:

bl - laboratory blank contamination

J - estimated value

I - laboratory control sample

mg/L - milligram per liter

U - Compound was analyzed for, but not detected above the value shown.

ug/kg - microgram per kilogram

Note: Line items where the laboratory result contains a "J" and the final result contains a "U" with a data validation reason code "bl" indicate that the final result is reported as not detected ("U" flag) at the reporting limit.