

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: 580-78459-1 and 580-78459-5

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

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File Name: 580-78459-1_5 DVR

SUMMARY

The data quality review of three surface sediment samples collected June 28, 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), mercury by EPA Method 7471A, TOC by EPA Method 9060, 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, 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 580-78459-1 and 580-78459-5:

Sample ID	Laboratory ID	Analytes
PDI-SG-B430	580-78459-1	TPH, Metals, Mercury, Tributyltin, bis(2-Ethylhexyl) phthalate, Total Solids, Grain Size
PDI-SG-B431	580-78459-2	Grain size
PDI-SG-B432	580-78459-3	TPH, Metals, Mercury, Tributyltin, bis(2-Ethylhexyl) phthalate, Total Solids, Grain Size

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. All three samples were received by the lab on 6/29/18 for rush grain size analyses and the other analyses were on hold. Rush grain size was reported in laboratory report 580-78459-5 on 7/20/18. Samples PDI-SG-B430 and PDI-SG-B432 were authorized for the other analyses on 8/16/18, but due to laboratory oversight the samples were not frozen upon receipt at TA Tacoma. Frozen samples were shipped from TA Sacramento, where samples were properly frozen upon receipt, to TA Tacoma on 9/10/18. These frozen samples were used for analysis.

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 – A rinsate blank was not submitted with this laboratory group. Associated rinsate blanks are reported under separate cover. Target compounds may have been detected in the rinsate blanks associated with these samples. Sediment data were not qualified based on rinsate blank results.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – bis(2-Ethylhexyl)phthalate was detected in the method blank associated with prep batch 284043 (3.89 ug/kg) at a concentration between the method detection limit (MDL) and reporting limit. bis(2-Ethylhexyl)phthalate was detected in PDI-SG-B432 at a concentration between the MDL and the reporting limit with an elevated reporting limit due to the dilution that was required prior to analysis; therefore, the result was qualified as estimated and flagged 'J' based on this method blank result.

3. Surrogates – Acceptable except as noted below:

PAHs by EPA Method 8270D-SIM – The percent recovery for surrogate terphenyl-d14 in PDI-SG-B432 (33%) was below the control limits of 57-120%. PDI-SG-B432 was analyzed at a dilution greater than 10x; therefore, data were not qualified based on this surrogate recovery.

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

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

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 not assessed using a sample from this laboratory group.

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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 not assessed using a sample from this laboratory group.

Tributyltin by Krone et al. – An MS/MSD was not performed using a sample from this laboratory group. Accuracy was assessed using the LCS. Precision was not assessed using a sample from this laboratory group.

6. Laboratory Duplicate

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

7. Reporting Limits – Acceptable except as noted below:

General – Analyte concentrations detected between the method detection limits (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 PDI-SG-B430 and PDI-SG-B432 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.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The reporting limits for PDI-SG-B430 were raised because of the dilution that was required prior to analysis due to the nature of the sample matrix. The reporting limit for the result that was reported as not detected in PDI-SG-B430 (1,500 ug/kg) and the MDL (340 ug/kg) exceeded the cleanup level (135 ug/kg).

8. Other Items of Note:

PAHs by EPA Method 8270D-SIM – The laboratory noted that the internal standard responses for chrysene-d12 and perylene-d12 were outside of acceptance limits (high). The results for benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[g,h,i]perylene, and chrysene in PDI-SG-B432 were qualified as estimated and flagged 'J' based on these elevated internal standard responses.

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-B430 and PDI-SG-B432.

The laboratory noted that the percent difference (%D) for the surrogate o-terphenyl in the continuing calibration verification (CCV) associated with analytical batch 284139 was outside the control limits of $\pm 20\%$ (high). As the surrogate recovery in the associated sample was acceptable, data were not qualified based on this high surrogate %D.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The laboratory noted that the internal standard response in the method blank and LCS associated with analytical batch 284043 were outside of the acceptance limits. Data were not qualified based on internal standard responses in QC samples.

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The laboratory noted that the %Ds for the surrogate terphenyl-d14 in the CCVs associated with analytical batches 284395 and 284567 were outside the control limits of $\pm 20\%$ (high). As the surrogate recoveries in the associated samples were acceptable, data were not qualified based on these high surrogate %Ds.

METALS ANALYSES

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

1. Holding Times – Acceptable except as noted below:

Mercury by Method 7471A – As noted under sample receipt, all samples in this laboratory group were frozen by TA upon receipt. The holding time for mercury is not extended by freezing; therefore the holding time remains 28 days to final analysis. The holding time for mercury was exceeded in PDI-SG-B430 and PDI-SG-B432 by 27 days due to the delay to authorize sample analysis. The results for mercury in these samples were qualified as estimated and flagged 'J' based on the holding time exceedance.

2. Blanks – Acceptable except as noted below:

General – A rinsate blank was not submitted with this laboratory group. Associated rinsate blanks are reported under separate cover. Target compounds may have been detected in the rinsate blanks associated with these samples. 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 on samples 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/LCSD.

6. Serial Dilution

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

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.



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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 – The 7-day holding time indicated for total solids in the QAPP was exceeded for the samples in this laboratory group. No data qualifiers were assigned based on this holding time exceedance.

2. Blanks – Acceptable except as noted below:

General – A rinsate blank was not submitted with this laboratory group. Associated rinsate blanks are reported under separate cover. Target compounds may have been detected in the rinsate blanks associated with these samples. 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)

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 was assessed using the LCS/LCSD.

6. Reporting Limits – Acceptable

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 580-78459-1 and 580-78459-5 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Downtown/Upriver
TestAmerica Laboratory Groups: 580-78459-1 and 580-78459-5

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B430	580-78459-1	SW7471A	Mercury	0.038 J	mg/kg	0.038 J	h
PDI-SG-B432	580-78459-3	SW7471A	Mercury	0.047	mg/kg	0.047 J	h
PDI-SG-B432	580-78459-3	SW8270DSIM	Benzo(a)anthracene	11 J	ug/kg	11 J	i
PDI-SG-B432	580-78459-3	SW8270DSIM	Benzo(a)pyrene	20 J	ug/kg	20 J	i
PDI-SG-B432	580-78459-3	SW8270DSIM	Benzo(b)fluoranthene	19 J	ug/kg	19 J	i
PDI-SG-B432	580-78459-3	SW8270DSIM	Benzo(g,h,i)perylene	34 J	ug/kg	34 J	i
PDI-SG-B432	580-78459-3	SW8270DSIM	Chrysene	27 J	ug/kg	27 J	i
PDI-SG-B432	580-78459-3	SW8270D	Bis(2-ethylhexyl)phthalate	970 J	ug/kg	970 J	bl

Notes:

- bl - laboratory blank contamination
- h - holding time
- i - internal standard issues
- J - estimated value
- mg/kg - milligram per kilogram
- ug/kg - microgram per kilogram