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# Data Validation Report

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| Project: | Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling<br>Portland Harbor Superfund Site<br>Surface Sediment – Downtown/Upriver |
| Laboratory: | TestAmerica Laboratories, Incorporated, Seattle, WA |
| Laboratory Groups: | 580-79669-1 and 580-79669-5 |
| 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 Number: | 60566335, Task #2.12 |
| Prepared by: | Chelsey Cook/AECOM — Completed on: October 30, 2018 |
| Reviewed by: | Amy Dahl/AECOM — File Name: 580-79669-1_5 DVR |

**SUMMARY**

The data quality review of three surface sediment samples collected on August 16, 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) and EPA Method 7471A (mercury), 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 580-79669-1 and 580-79669-5:

| Sample ID | Laboratory ID | Analytes |
|---|---|---|
| PDI-SG-B437 | 580-79669-1 | TPH, Metals, Tributyltin, bis(2-Ethylhexyl) phthalate, PAHs, TOC, Total Solids, Grain Size |
| PDI-SG-B437-D (field duplicate of PDI-SG-B437) | 580-79669-2 | TPH, Metals, Tributyltin, bis(2-Ethylhexyl) phthalate, PAHs, TOC, Total Solids |
| PDI-SG-B438 | 580-79669-3 | TPH, Metals, Tributyltin, bis(2-Ethylhexyl) phthalate, PAHs, TOC, 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.

## 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 TA on 8/17/18. PDI-SG-B437-D was marked incorrectly on the COC for grain size analysis and AECOM instructed TA to cancel grain size analysis for this sample and revise the COC. Rush grain size for PDI-SG-B437 and PDI-SG-B438 was reported in laboratory report 580-79669-5 on 9/5/18. All three samples were authorized for the other analyses on 9/7/18, 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. The analyses authorized on 9/7/18 were reported in laboratory group 580-79669-1.

## 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-B437-D at a concentration between the MDL and the reporting limit with an elevated reporting limit due to the dilution prior to analysis; therefore, the result for bis(2-ethylhexyl)phthalate was qualified as estimated and flagged 'J' based on the method blank result.

   bis(2-Ethylhexyl)phthalate was detected in the method blank associated with prep batch 284057 (8.73 ug/kg) at a concentration between the MDL and reporting limit. bis(2-Ethylhexyl)phthalate was not detected in the associated sample; therefore, data were not qualified based on this method blank result.

   PAHs by EPA Method 8270D-SIM – Fluoranthene (0.373 ug/kg), phenanthrene (0.734 ug/kg), and pyrene (0.314 ug/kg) were detected in the method blank associated with prep batch 284059 at concentrations between the MDLs and reporting limits. These analytes were detected in the associated samples at concentrations greater than the reporting limits; therefore, data were not qualified based on these method blank results.

3. Surrogates – Acceptable

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

5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Acceptable except as noted below:

   bis(2-Ethylhexyl)phthalate by EPA Method 8270D – An MS/MSD was performed using PDI-SG-B438. The percent recovery in the MS (161%) and the MSD (194%) exceeded the control limits of 59-123%. bis(2-Ethylhexyl)phthalate was not detected in PDI-SG-B438; therefore, data were not qualified based on the elevated MS/MSD results.

   PAHs by EPA Method 8270D-SIM – An MS/MSD was performed using PDI-SG-B438. Results were acceptable.

   TPHs by Method NWTPH-Dx – An MS/MSD was performed using PDI-SG-B438. The relative percent differences (RPDs) for the MS/MSD pair for diesel-range hydrocarbons (22%) and motor oil-range hydrocarbons (23%) exceeded the control limit of 16%. The percent recoveries in the MS and the MSD were acceptable for both diesel-range hydrocarbons and motor oil-range hydrocarbons; therefore, data were not qualified based on the elevated RPDs.

   Tributyltin by Krone et al. – An MS/MSD was performed using PDI-SG-B438. Results were acceptable.

6. Field Duplicate – Acceptable except as noted below:

   General – A field duplicate was submitted for PDI-SG-B437 and identified as PDI-SG-B437-D. The results were comparable with the following exceptions.

   PAHs by EPA Method 8270D-SIM – The RPDs for the following analytes exceeded 50%:

| Analyte | RPD |
|---|---|
| Benzo[a]anthracene | 74% |
| Benzo[a]pyrene | 81% |
| Benzo[b]fluoranthene | 97% |
| Benzo[g,h,i]perylene | 102% |
| Benzo[k]fluoranthene | 60% |
| Chrysene | 74% |
| Indeno[1,2,3-cd]pyrene | 96% |
| Pyrene | 56% |

   The sample concentrations for the analytes noted above were less than five times the reporting limits; therefore, data were not qualified based on the elevated field duplicate RPDs.

   bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The RPD for bis(2-ethylhexyl)phthalate (168%) in the field duplicate pair exceeded 50%. The sample concentrations were less than five times the reporting limits; therefore, data were not qualified based on the elevated field duplicate RPD.

7. Laboratory Duplicate

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

8. 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 all 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 PAHs 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 all 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 limit for the result reported as not detected in PDI-SG-B438 exceeded the cleanup level (135 ug/kg) but the MDL did not.

   TPHs by Method NWTPH-Dx – The laboratory indicated that the diesel-range hydrocarbon elution pattern was later than the typical diesel pattern in PDI-SG-B437 and PDI-SG-B437-D.

9. Other Items of Note:

   bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The laboratory noted that the percent differences (%Ds) for the surrogate terphenyl-d14 in the continuing calibration verifications (CCVs) associated with analytical batches 284395 and 284567 were outside the control limits of ±20% (high). As the surrogate recoveries in the associated samples were acceptable, data were not qualified based on these high surrogate %Ds.

   TPHs by Method NWTPH-Dx – The laboratory noted that the %Ds for the surrogate o-terphenyl in the CCVs associated with analytical batches 284139 and 284335 were outside the control limits of ±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

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) – Acceptable except as noted below:

Metals by Method 6020B – An MS/MSD and PDS were performed using PDI-SG-B438. The percent recoveries for copper in the MS (133%) and the MSD (121%) exceeded the control limits of 80-120%. The result for copper in PDI-SG-B438 was qualified as estimated and flagged 'J' based on the MS/MSD results.

Mercury by Method 7471A – An MS/MSD was performed using PDI-SG-B438. The percent recovery for mercury in the MS (121%) exceeded the control limits of 80-120%. The percent recovery in the MSD and the RPD for the MS/MSD pair were acceptable; therefore, data were not qualified based on the MS result.

5. Field Duplicate – Acceptable

General – A field duplicate was submitted for PDI-SG-B437 and identified as PDI-SG-B437-D. The results were comparable.

6. Laboratory Duplicate – Acceptable

Metals by Method 6020B – A laboratory duplicate was performed using PDI-SG-B438. Results were comparable.

Mercury by Method 7471A – A laboratory duplicate was performed using PDI-SG-B438. Results were comparable.

7. Serial Dilution – Acceptable

Metals by Method 6020B – A serial dilution was performed using PDI-SG-B438. Results were comparable.

8. 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 – The 7-day holding time indicated for total solids in the QAPP was exceeded for the samples in this laboratory group by 21-29 days. 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.

TOC by Method 9060 – TOC was detected in the method blank associated with analytical batch 284391 (119 mg/kg) at a concentration between the MDL and the RL. TOC was detected in the associated samples at concentrations significantly greater than the method blank detection; therefore, data were not qualified 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) – Acceptable

   TOC by Method 9060 – An MS/MSD was performed using PDI-SG-B438. Results were acceptable.

5. Field Duplicate – Acceptable

   General – A field duplicate was submitted for PDI-SG-B437 and identified as PDI-SG-B437-D. The results were comparable.

6. Laboratory Replicate

   TOC by Method 9060 – A laboratory duplicate and triplicate were performed using PDI-SG-B438. Results were comparable.

   Total Solids by ASTM Method D-2216 – A laboratory duplicate was performed using PDI-SG-B438. Results were comparable.

   Moisture Content at 70°C – A laboratory duplicate was performed using PDI-SG-B437-D. Results were comparable.

7. 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 – Acceptable except as noted below:

   The laboratory performed duplicate analysis at a rate of 1 per 20 samples per their internal requirements. A laboratory duplicate was performed using PDI-SG-B437. The result for the clay fraction for sample PDI-SG-B437 was assigned an 'L' qualifier to indicate that the grain size fraction was greater than 5 percent of the total combined fractions and the RPD for duplicate analysis on the sample fraction was greater than 20%.

## 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 580-79669-1 and 580-79669-5 is 100%.

**Table 1**
**QA/QC Data Summary Review**
**Portland Harbor**
**Surface Sediment - Downtown/Upriver**
**TestAmerica Laboratory Groups: 580-79669-1 and 580-79669-5**

| Sample ID | Laboratory ID | Method | Analyte | Laboratory Result | Units | Final Result | Reason Code |
|---|---|---|---|---|---|---|---|
| PDI-SG-B437 | 580-79669-1 | D7928/D6913 | Clay | 5.4 | % | 5.4 L | ld |
| PDI-SG-B437-D | 580-79669-2 | SW8270D | Bis(2-ethylhexyl)phthalate | 150 J | ug/kg | 150 J | bl |
| PDI-SG-B438 | 580-79669-3 | SW6020B | Copper | 31 | mg/kg | 31 J | m |

Notes:
% - percent
bl - laboratory blank contamination
J - estimated value
L - the grain size fractions were greater than 5 percent of the total combined fractions and the RPD for duplicate analysis on the sample fractions were greater than 20%
ld - laboratory duplicate RPDs
m - matrix spike recovery
mg/kg - milligram per kilogram
RPD - relative percent difference
ug/kg - microgram per kilogram