

Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site Surface Sediment – Stratified Random	
Laboratory:	TestAmerica Laboratories, Incorporated, Seattle, WA	
Laboratory Group:	580-77608-1	
Analyses:	Petroleum Hydrocarbons, Metals, Total Organic Carbon (TOC), Total Solids, and Grain Size	
Validation Level:	Stage 2A	
AECOM Project Number:	60566335, Task #2.12	
Prepared by:	Chelsey Cook/AECOM	Completed on: September 4, 2018
Reviewed by:	Karen Mixon/AECOM	File Name: 580-77608-1 DVR

SUMMARY

The data quality review of three surface sediment samples collected between April 28 and April 30, 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, total solids by American Society for Testing and Materials (ASTM) Method D-2216, moisture content at 70 degrees centigrade (70°C), and 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, and Annual Book of ASTM Standards, ASTM, Philadelphia, Pennsylvania. The laboratory provided level 2 and level 4 data packages containing sample results, associated quality assurance (QA) and quality control (QC) data, preparation logs, and raw instrument outputs (where applicable). The following samples are associated with laboratory group 580-77608-1:

Sample ID	Laboratory ID
PDI-SG-B405-BL1	580-77608-12
PDI-SG-B409-BL1	580-77608-13
PDI-SG-B414-BL1	580-77608-14

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 temperatures were recorded. The coolers were received at temperatures within the EPA-recommended limits of greater than 0°C and less than or equal to 6°C. The samples in this laboratory group were frozen after sample collection until they were shipped to TA on May 25, 2018.



Data Validation Report

Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling

Surface Sediment – Stratified Random

TestAmerica Lab Group: 580-77608-1

AECOM instructed TA to hold the samples in the freezer until they were released for analysis on June 7, 2018. TA originally logged these samples with samples from a separate COC. AECOM instructed them to separate them into their own laboratory group.

ORGANIC ANALYSES

Samples were analyzed for TPHs by method NWTPH-Dx.

1. Holding Times – Acceptable
2. Blanks – Acceptable

Two rinsate blanks were collected on April 29 and May 2, 2018, were reported with laboratory groups 580-76932 (ID 580-76932-35) and 580-77073 (ID 580-77073-28), and are applicable to the samples reported in this laboratory group. TPHs were not detected in these rinsate blanks.

3. Surrogates – Acceptable except as noted below:

The percent recoveries for the surrogate o-terphenyl in PDI-SG-B405-BL1 (48%) and the laboratory duplicate of PDI-SG-B414-BL1 (46%) were below the control limits of 50-150%. The results for diesel-range hydrocarbons and motor oil-range hydrocarbons in PDI-SG-B405-BL1 were qualified as estimated and flagged 'J' based on this low surrogate recovery. Data were not qualified based on the surrogate recovery in the laboratory duplicate performed with PDI-SG-B414-BL1.

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

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

6. Laboratory Duplicate – Acceptable

A laboratory duplicate was performed using PDI-SG-B414-BL1. Results were comparable.

7. Reporting Limits – Acceptable

The reporting limits for PDI-SG-B405-BL1 and PDI-SG-B414-BL1 were raised due to dilutions that were required prior to analysis to bring the concentration of target analytes within the calibration range of the instrument.

Analyte concentrations detected between the method detection limit (MDL) and the reporting limit are reported by the laboratory with a 'J' flag. Laboratory 'J'-flagged results are considered estimated results. As the result is between the MDL and the reporting limit, there is a greater level of uncertainty associated with the numerical result.

8. Other Items of Note:

The laboratory indicated that the diesel-range hydrocarbon elution patterns were later than the typical diesel pattern in PDI-SG-B405-BL1 and PDI-SG-B414-BL1.

Data Validation Report

Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling

Surface Sediment – Stratified Random

TestAmerica Lab Group: 580-77608-1

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 after collection and prior to shipment to TA, and again by TA upon receipt. Mercury does not have an extension to the holding time if the samples are frozen; therefore the holding time remains at 28 days to final analysis. The holding time for mercury was exceeded in PDI-SG-B405-BL1, PDI-SG-B409-BL1, and PDI-SG-B414-BL1 by 25 to 27 days. The results for mercury in these samples were qualified as estimated and flagged 'J' based on the holding time exceedance.

2. Blanks – Acceptable

General – Two rinsate blanks were collected on April 29 and May 2, 2018, were reported with laboratory groups 580-76932 (laboratory ID 580-76932-35) and 580-77073 (laboratory ID 580-77073-28), and are applicable to the samples reported in this laboratory group. Copper (0.00063 mg/L) was detected in 580-76932-35. Data were not qualified based on rinsate blank results.

Mercury by Method 7471A – Mercury was detected in the method blank associated with analytical batch 276893 (0.0101 mg/kg) at a concentration below the reporting limit but above the MDL. Mercury was detected in PDI-SG-B409-BL1 at a concentration above the reporting limit but less than ten times the method blank detection. Mercury in PDI-SG-B409-BL1 was previously qualified as estimate and flagged 'J' based on the holding time exceedance and no additional qualification was necessary based on the method blank result.

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-B405-BL1. The percent recoveries for copper in the MSD (138%) and lead in the MS (129%), MSD (194%), and relative percent different (RPD) for lead in the MS/MSD pair (26%), exceeded the control limits of 80-120% and 20%, respectively. The percent recovery for copper in the MS and the RPD for the MS/MSD pair was acceptable; therefore, data were not qualified based on the MS recovery for copper. The result for lead in PDI-SG-B405-BL1 was qualified as estimate and flagged 'J' based on the MS/MSD results.

Mercury by Method 7471A – An MS/MSD was not performed using a sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSD.

5. Laboratory Duplicate – Acceptable except as noted below:

Metals by Method 6020B – A laboratory duplicate was performed using PDI-SG-B405-BL1. The RPDs for arsenic (33%), cadmium (28%), and lead (37%) exceeded the control limit of 20%. The sample concentration of cadmium in PDI-SG-B405-BL1 was less than five times the reporting limit; therefore, cadmium was not qualified based on this elevated laboratory duplicate RPD. The result for lead in PDI-SG-B405-BL1 was previously qualified as

Data Validation Report

Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling

Surface Sediment – Stratified Random

TestAmerica Lab Group: 580-77608-1

estimated and flagged 'J' based on the MS/MSD results and no further qualification was necessary based on the elevated laboratory duplicate RPD. The result for arsenic in PDI-SG-B405-BL1 was qualified as estimate and flagged 'J' based on the elevated laboratory duplicate RPD.

Mercury by Method 7471A – A laboratory duplicate was not performed using a sample from this laboratory group. Precision was assessed using the LCS/LCSD.

6. Serial Dilution – Acceptable

Metals by Method 6020B – A serial dilution was performed using PDI-SG-B405-BL1. Results were comparable.

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 – The 7-day holding time indicated for total solids in the QAPP was exceeded for the samples in this laboratory group as the samples were temporarily held in freezer storage before shipping to TA. Total solids were analyzed within the time that other analyses were performed for samples in this laboratory group. No data qualifiers were assigned based on the holding time exceedance.

Moisture Content at 70°C – The 7-day holding time indicated for total solids in the QAPP was exceeded for the samples in this laboratory group as the samples were temporarily held in freezer storage before shipping to TA. Total solids were analyzed within the time that other analyses were performed for samples in this laboratory group. No data qualifiers were assigned based on the holding time exceedance.

2. Blanks – Acceptable where applicable, except as noted below:

General – Two rinsate blanks were collected on April 29 and May 2, 2018, were reported with laboratory groups 580-76932 (ID 580-76932-35) and 580-77073 (ID 580-77073-28), and are applicable to the samples reported in this laboratory group. TOC was detected in 580-76293-35 (0.38 mg/L) and 580-77073-28 (0.29 mg/L) but were qualified as not detected and flagged 'U' at the reporting limits based on the associated method blanks. Data were not qualified based on rinsate blank results.

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

Data Validation Report

Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling

Surface Sediment – Stratified Random

TestAmerica Lab Group: 580-77608-1

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

TOC by Method 9060 – An MS/MSD was performed using PDI-SG-B405-BL1. Results were acceptable.

5. Laboratory Replicate – Acceptable

TOC by Method 9060 – A laboratory duplicate and triplicate were performed on PDI-SG-B405-BL1. Results were comparable.

Total Solids by ASTM Method D-2216 – A laboratory duplicate was performed using PDI-SG-B414-BL1. The RPD (35%) exceeded the control limit of 20%. The result for total solids in PDI-SG-B414-BL1 was qualified as estimated and flagged 'J' based on the elevated laboratory duplicate RPD. The laboratory duplicate results imply that a representative total solids result could not be obtained for the sample; therefore, the results of the other analyses were qualified as estimated and flagged 'J' based on the total solids. Mercury was already flagged 'J' based on the holding time exceedance and no further qualification was necessary based on the total solids.

Moisture Content at 70°C – A laboratory duplicate was not performed using a sample from this laboratory group. Precision was not assessed.

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. As indicated under sample receipt, the sample volume used for grain size analysis was frozen until shipped to TA. No data qualifiers were assigned to grain size results based on sample condition.

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 group 580-77608-1 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Stratified Random
TestAmerica Laboratory Group: 580-77608-1

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B405-BL1	580-77608-12	NWTPH-Dx	TPH-Diesel Range Organics	210 J	mg/kg	210 J	s
PDI-SG-B405-BL1	580-77608-12	NWTPH-Dx	TPH-Motor Oil Range Organics	1500	mg/kg	1500 J	s
PDI-SG-B405-BL1	580-77608-12	SW6020B	Lead	42	mg/kg	42 J	m,md
PDI-SG-B405-BL1	580-77608-12	SW6020B	Arsenic	4.7	mg/kg	4.7 J	ld
PDI-SG-B405-BL1	580-77608-12	SW7471A	Mercury	0.11	mg/kg	0.11 J	h
PDI-SG-B409-BL1	580-77608-13	SW7471A	Mercury	0.041	mg/kg	0.041J	h
PDI-SG-B414-BL1	580-77608-14	NWTPH-Dx	TPH-Diesel Range Organics	300 J	mg/kg	300 J	x
PDI-SG-B414-BL1	580-77608-14	NWTPH-Dx	TPH-Motor Oil Range Organics	1200	mg/kg	1200 J	x
PDI-SG-B414-BL1	580-77608-14	SW6020B	Lead	21	mg/kg	21 J	x
PDI-SG-B414-BL1	580-77608-14	SW6020B	Arsenic	3.2	mg/kg	3.2 J	x
PDI-SG-B414-BL1	580-77608-14	SW6020B	Cadmium	0.17 J	mg/kg	0.17 J	x
PDI-SG-B414-BL1	580-77608-14	SW6020B	Copper	23	mg/kg	23 J	x
PDI-SG-B414-BL1	580-77608-14	SW6020B	Zinc	66	mg/kg	66 J	x
PDI-SG-B414-BL1	580-77608-14	SW7471A	Mercury	0.16	mg/kg	0.16 J	h
PDI-SG-B414-BL1	580-77608-14	SW9060	Total Organic Carbon	47000	mg/kg	47000J	x
PDI-SG-B414-BL1	580-77608-14	D2216	Total Solids	54.9	%	54.9 J	ld

% - percent

J - estimated value

ld - laboratory duplicate relative percent difference

m - matrix spike recovery

md - matrix spike/matrix spike duplicate RPD

mg/kg - milligram per kilogram

RPD - relative percent difference

s - surrogate recovery

x - percent solids