

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: 580-76685-1

Analyses: Petroleum Hydrocarbons, Metals, Total Organic Carbon (TOC), Total Solids, and Grain Size

Validation Level: Stage 2A

AECOM Project
 Number: 60566335, Task #2.12

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SUMMARY

The data quality review of 19 surface sediment samples and two rinsate blanks collected on April 16 and April 17, 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 (surface sediments) and EPA Method 7470A (water), TOC by EPA Method 9060 and Standard Method (SM) 5310B, total solids by American Society for Testing and Materials (ASTM) Method D-2216, 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, *Annual Book of ASTM Standards*, American Society for Testing & Materials (ASTM), Philadelphia, Pennsylvania, and *Standard Methods for the Examination of Water and Wastewater*. 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 580-76685-1:

Sample ID	Laboratory ID
PDI-SG-B133-BL1	580-76685-1
PDI-SG-B135-BL1	580-76685-2
PDI-SG-B112-BL1	580-76685-3
PDI-SG-B115-BL1	580-76685-4
PDI-SG-B156-BL1	580-76685-5
PDI-SG-B159-BL1	580-76685-6
PDI-SG-B163-BL1	580-76685-7
PDI-SG-B164-BL1	580-76685-8
PDI-SG-B164-BL1-D (field duplicate of PDI-SG-B164-BL1)	580-76685-9
PDI-SG-B167-BL1	580-76685-10
PDI-SG-B169-BL1	580-76685-11
PDI-SG-B114-BL1	580-76685-12
PDI-SG-B171-BL1	580-76685-13
PDI-SG-B173-BL1	580-76685-14

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Sample ID	Laboratory ID
PDI-SG-B175-BL1	580-76685-15
PDI-SG-B108-BL1	580-76685-16
PDI-SG-B160-BL1	580-76685-17
PDI-SG-B168-BL1	580-76685-18
PDI-SG-B202-BL1	580-76685-19
PDI-RB-VVSS-180416-1735 (rinsate blank)	580-76685-20
PDI-RB-VVSS-180416-1800 (rinsate blank)	580-76685-21

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. No discrepancies with sample identification were noted by TA.

ORGANIC ANALYSES

Samples were analyzed for TPHs by NWTPH-Dx.

1. Holding Times – Acceptable
2. Initial and Continuing Calibration Verifications – Acceptable except as noted below:

The laboratory noted that the percent differences (%Ds) for the surrogate o-terphenyl in the continuing calibration verifications (CCVs) associated with analytical batches 272686 and 273247 were outside the method limits of $\pm 15\%$. The o-terphenyl recoveries were acceptable in all samples associated with these CCVs; therefore, data were not qualified based on these CCV surrogate %Ds.

The laboratory noted that the %Ds for diesel-range hydrocarbons (high) and motor oil-range hydrocarbons (high) in the CCV associated with batch 272683 exceeded the method limits of $\pm 15\%$. Diesel-range hydrocarbons and motor oil-range hydrocarbons were not detected in the samples associated with this CCV; therefore, data were not qualified based on these CCV %Ds.

3. Blanks – Acceptable

Four rinsate blanks were collected on April 16, 2018, April 19, 2018, and April 22, 2018, were reported with TA laboratory groups 580-76685 (laboratory IDs 580-76685-20 and 580-76685-21), 580-76755 (laboratory ID 580-76755-28), and 580-76777 (laboratory ID 580-76777-32), and are applicable to the samples reported in this laboratory group. TPHs were not detected in these rinsate blanks.



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

An MS/MSD was performed using PDI-SG-B202-BL1. Results were acceptable.

7. Field Duplicate – Acceptable

A field duplicate was submitted for PDI-SG-B164-BL1 and identified as PDI-SG-B164-BL1-D. The relative percent difference (RPD) for diesel-range hydrocarbons was not calculable. Diesel-range hydrocarbons were not detected in PDI-SG-B164-BL1 and the concentration for diesel-range hydrocarbons in PDI-SG-B164-BL1-D was less than five times the reporting limit; therefore, data were not qualified based on the field duplicate results.

8. Laboratory Duplicate – Acceptable

Laboratory duplicates were performed using PDI-SG-B112-BL1 and PDI-SG-B173-BL1. Results were comparable.

9. Reporting Limits – Acceptable except as noted below:

The reporting limits for diesel-range hydrocarbons reported as not detected in multiple samples were elevated due to the moisture content of the samples. The reporting limits for diesel-range hydrocarbons reported as not detected in PDI-SG-B133-BL1 and PDI-SG-B108-BL1 exceeded the cleanup level. The method detection limits (MDLs) did not exceed the cleanup level in the samples noted above.

Analyte concentrations detected between the MDL and the reporting limit are reported by the laboratory with a 'J' flag. One or more results were flagged 'J' by the laboratory. 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.

10. Other Items of Note:

The laboratory indicated that the diesel-range hydrocarbon elution patterns were later than the typical diesel pattern in PDI-SG-B133-BL1, PDI-SG-B135-BL1, PDI-SG-B112-BL1, PDI-SG-B115-BL1, PDI-SG-B156-BL1, PDI-SG-B159-BL1, PDI-SG-B163-BL1, PDI-SG-B164-BL1, PDI-SG-B167-BL1, PDI-SG-B169-BL1, PDI-SG-B108-BL1, and PDI-SG-B160-BL1.

METALS ANALYSES

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

1. Holding Times – Acceptable
2. Blanks – Acceptable

General – Four rinsate blanks were collected on April 16, 2018, April 19, 2018, and April 22, 2018, were reported with TA laboratory groups 580-76685 (laboratory IDs 580-76685-20 and 580-76685-21), 580-76755 (laboratory ID 580-76755-28), and 580-76777 (laboratory ID 580-

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76777-32), and are applicable to the samples reported in this laboratory group. One or more metals were detected in these rinsate blanks at concentrations below the reporting limits but above the MDLs as noted in the table below.

Rinsate Blank ID	Metal	Concentration (mg/L)
580-76685-20	Copper	0.00064
580-76685-21	Arsenic	0.00027
	Copper	0.00065
	Lead	0.00051

Data were not qualified based on rinsate blank detections.

- Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) - Acceptable
- Matrix Spike/Matrix Spike Duplicate (MS/MSD) and Post Digestion Spike (PDS, where applicable) – Acceptable except as noted below:

Metals by Method 6020B – MS/MSDs and PDSs were performed using PDI-SG-B202-BL1 and PDI-SG-B168-BL1. The following analytes were outside the control limits in the MS/MSDs:

Sample	Analyte	MS	MSD	RPD (control limit = 20%)	Control Limits
PDI-SG-B202-BL1	Copper	122%	ok	ok	80-120%
PDI-SG-B168-BL1	Arsenic	ok	ok	24%	80-120%
	Cadmium	ok	ok	30%	80-120%
	Lead	ok	ok	25%	80-120%

ok – acceptable

As two of the quality control parameters (MS, MSD, and/or RPD) were acceptable, data were not qualified for copper, arsenic, cadmium, and lead based on these MS/MSD results.

Mercury by Method 7471A – An MS/MSD was performed using PDI-SG-B202-BL1. Results were acceptable.

An MS/MSD was performed using PDI-SG-B168-BL1. The percent recovery in the MSD (158%) and the RPD for the MS/MSD pair (47%) exceeded the control limits of 80-120% and 20%, respectively. The result for mercury in PDI-SG-B168-BL1 was qualified as estimated and flagged 'J' based on the MS/MSD results.

- Field Duplicate – Acceptable

General – A field duplicate was submitted for PDI-SG-B164-BL1 and identified as PDI-SG-B164-BL1-D. Results were comparable.

- Laboratory Duplicate – Acceptable except as noted below:

Metals by Method 6020B – Laboratory duplicates were performed using PDI-SG-B202-BL1 and PDI-SG-B168-BL1. The RPD for cadmium in the laboratory duplicate performed using PDI-SG-B168-BL1 (87%) exceeded the control limit of 20%. The concentration for cadmium

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in this sample was less than five times the reporting limit; therefore, data were not qualified for cadmium based on the elevated laboratory duplicate RPD.

Mercury by Method 7471A – Laboratory duplicates were performed using PDI-SG-B202-BL1 and PDI-SG-B168-BL1. The RPDs for mercury in the laboratory duplicates performed using PDI-SG-B202-BL1 (63%) and PDI-SG-B168-BL1 (43%) exceeded the control limit of 20%. The concentrations for mercury in these samples were less than five times the reporting limits; therefore, data were not qualified for mercury based on the elevated laboratory duplicate RPDs.

7. Serial Dilutions – Acceptable

Metals by Method 6020B – Serial dilutions were performed using PDI-SG-B202-BL1 and PDI-SG-B168-BL1. Results were comparable.

8. Reporting Limits – Acceptable

One or more results in multiple samples were detected below the reporting limit but above the MDL and 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

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

TOC by Method 9060 – TOC (75.1 mg/kg) was detected in the method blank associated with analytical batch 272586 at a concentration below the reporting limit but above the MDL. TOC was detected in PDI-SG-B156-BL1 at a concentration below the reporting limit and above the MDL; therefore, the result for TOC in PDI-SG-B156-BL1 was qualified as not detected and flagged 'U' at the reporting limit.

Four rinsate blanks were collected on April 16, 2018, April 19, 2018, and April 22, 2018, were reported with TA laboratory groups 580-76685 (laboratory IDs 580-76685-20 and 580-76685-21), 580-76755 (laboratory ID 580-76755-28), and 580-76777 (laboratory ID 580-76777-32), and are applicable to the samples reported in this laboratory group. TOC was detected in 580-76685-20 (0.22 mg/L), 580-76685-21 (0.53 mg/L), 580-76755-28 (0.24 mg/L), and 580-76777-32 (0.28 mg/L) at concentrations below the reporting limits and above the MDLs. Data were not qualified based on rinsate blank detections.

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

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

TOC by Method 9060 – An MS/MSD was performed using PDI-SG-B202-BL1. Results were acceptable.

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5. Field Duplicate – Acceptable

A field duplicate was submitted for PDI-SG-B164-BL1 and identified as PDI-SG-B164-BL1-D. Results were comparable.

6. Laboratory Replicate – Acceptable where applicable

TOC by Method 9060 – A laboratory duplicate and triplicate was performed using PDI-SG-B202-BL1. Results were comparable.

Total Solids by ASTM Method D-2216 – A laboratory duplicate was performed using PDI-SG-B202-BL1. Results were comparable.

7. Reporting Limits – Acceptable

TOC by Method 9060 – One or more results in multiple samples were detected below the reporting limit but above the MDL and flagged 'J' by the laboratory. As described above, laboratory 'J'-flagged results are considered estimated results.

The result for TOC in PDI-SG-B156-BL1 was flagged 'J' by the laboratory; however, 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 – 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 on PDI-SG-B175-BL1. The result for the gravel fraction for sample PDI-SG-B175-BL1 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 group 580-76685-1 is 100%.

Table 1
QA/QC Data Summary Review
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Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B156-BL1	580-76685-5	SW9060	Total Organic Carbon	750 J	mg/kg	2,000 U	bl
PDI-SG-B175-BL1	580-76685-15	D7928/D6913	Gravel	11.4	%	11.4 L	ld
PDI-SG-B168-BL1	580-76685-18	SW7471A	Mercury	0.027 J	mg/kg	0.027 J	m,md

% - percent

bl - laboratory blank contamination

J - estimated value

L - 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%

ld - laboratory duplicate RPD

m - matrix spike recovery

md - matrix spike/matrix spike duplicate RPD

mg/kg - milligram per kilogram

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

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.