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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:	580-78604-1, 580-78604-6, and 580-78604-7
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 10 surface sediment samples and one rinsate blank collected on July 2 and July 3, 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, zinc, and/or manganese), 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 580-78604-1, 580-78604-6, and 580-78604-7:

Sample ID	Laboratory ID
PDI-SG-B458	580-78604-1
PDI-SG-B470	580-78604-2
PDI-SG-B469	580-78604-3
PDI-SG-B456	580-78604-4
PDI-SG-B462	580-78604-5
PDI-SG-B463	580-78604-6
PDI-SG-B464	580-78604-7
PDI-SG-B466	580-78604-8
PDI-SG-B468	580-78604-9

Sample ID	Laboratory ID
PDI-SG-B429	580-78604-10
PDI-RB-VV-180703 (rinsate blank)	580-78604-11

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. AECOM added rush analysis of manganese for PDI-SG-B466 and metals including manganese, TOC, and total solids for PDI-SG-B468. The sample ID for the rinsate blank was changed from RB-VV-180703-1720 to PDI-RB-VV-180703. The rinsate blank results were reported in laboratory group 580-78604-1 on 7/31/18. The rush grain size, TOC, and metals results were reported in laboratory group 580-78604-6 on 8/1/18. Sample analyses authorized on August 16, 2018 were reported in laboratory group 580-78604-7. 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 – 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 blanks associated with prep batches 284043 (3.89 ug/kg) and 284408 (5.71 ug/kg) at concentrations between the method detection limits (MDLs) and reporting limits. bis(2-Ethylhexyl)phthalate was detected in PDI-SG-B456 and PDI-SG-B429 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 except as noted below:

   bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The surrogate recoveries for terphenyl-d14 in PDI-SG-B470 (148%) and the method blank associated with prep batch 284408 (129%) exceeded the control limits of 58-120%. Data were not qualified based on the surrogate recoveries in QC samples (method blank). bis(2-Ethylhexyl)phthalate was not

detected in PDI-SG-B470; therefore, data were not qualified based on the elevated surrogate recovery.

PAHs by EPA Method 8270D-SIM – The percent recoveries for the surrogate terphenyl-d14 in PDI-SG-B458 (56%) and PDI-SG-B466 (45%) were outside the control limits of 57-120%. PDI-SG-B458 was analyzed at a 25x dilution; therefore, data were not qualified based on the surrogate recovery. The PAHs in PDI-SG-B466 were qualified as estimated and flagged 'J' or 'UJ' based on this surrogate recovery.

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

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The percent recovery in the LCS (163%) associated with prep batch 278382 exceeded the control limits of 20-150%. The percent recovery in the LCSD and the relative percent difference (RPD) for the LCS/LCSD pair were acceptable; therefore, data were not qualified based on this LCS recovery.

PAHs by EPA Method 8270D-SIM – The percent recovery for acenaphthylene in the reanalysis of the LCS (66%) associated with prep batch 285535 was below the control limits of 68-120%. The result for acenaphthylene in the reanalysis of PDI-SG-B468 was qualified as estimated and flagged 'UJ' based on the LCS recovery. The result for acenaphthylene in the reanalysis of PDI-SG-B466 was qualified as estimated and flagged 'J' based on the surrogate recovery and no further qualification was necessary based on this LCS recovery.

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 analyzed using PDI-SG-B463. The percent recoveries for the MS and MSD and the RPD could not be calculated because the sample was diluted and the MSD was recovered below the elevated MDL. Results were not qualified based on MS/MSD results that could not be calculated.

PAHs by EPA Method 8270D-SIM – An MS/MSD was analyzed using PDI-SG-B463. The following analytes were outside of the control limits:

Analyte	MS	MSD	RPD	Control Limits (matrix spike / RPD)
Benzo[a]anthracene	60%	64%	ok	66-120% / 14%
Benzo[a]pyrene	39%	41%	ok	72-124% / 12%
Benzo[b]fluoranthene	35%	26%	15%	63-121% / 10%
Benzo[g,h,i]perylene	50%	57%	ok	63-120% / 14%
Benzo[k]fluoranthene	56%	ok	ok	63-123% / 15%
Chrysene	31%	33%	ok	69-120% / 10%
Fluoranthene	-31%	-31%	ok	74-125% / 13%
Naphthalene	58%	58%	ok	70-120% / 12%
Phenanthrene	-18%	-22%	ok	73-120% / 11%
Pyrene	-38%	-41%	ok	70-120% / 12%

ok - acceptable

The percent recovery in the MSD and the RPD for the MS/MSD pair were acceptable for benzo[k]fluoranthene; therefore, data were not qualified based on the MS result. The results

for benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[g,h,i]perylene, chrysene, fluoranthene, naphthalene, phenanthrene, and pyrene in PDI-SG-B463 were qualified as estimated and flagged 'J' based on the MS/MSD results.

TPHs by Method NWTPH-Dx – An MS/MSD was analyzed using PDI-SG-B463. Results were acceptable.

Tributyltin by Krone et al. – An MS/MSD was analyzed using PDI-SG-B463. The RPD for the MS/MSD pair (80%) exceeded the control limit of 20%. The percent recoveries in the MS and MSD were acceptable; therefore, data were no qualified based on this elevated RPD.

6. Laboratory Duplicate

TPHs by Method NWTPH-Dx – Laboratory duplicates were performed using PDI-SG-B429 and PDI-SG-B468. Results were comparable.

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 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 multiple analytes in multiple samples reported as not detected exceeded the cleanup level for carcinogenic PAHs 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 limits for the results reported as not detected in PDI-SG-B462, PDI-SG-B463, PDI-SG-B466, and PDI-SG-B468 exceeded the cleanup level (135 ug/kg), but the MDL did not. The reporting limit and MDL exceeded the cleanup level for bis(2-ethylhexyl)phthalate in PDI-SG-B458, PDI-SG-B470, PDI-SG-B469, and PDI-SG-B464.

8. Other Items of Note:

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The laboratory noted that the percent difference (%D) for bis(2-ethylhexyl)phthalate in the continuing calibration verification (CCV) associated with analytical batches 278847 and the surrogate terphenyl-d14 in the CCVs associated with analytical batches 284395 and 284567 were outside the control limits of ±20% (high). bis(2-Ethylhexyl)phthalate was not detected in the samples associated with analytical batch 278847; therefore, data were not qualified based on the elevated %D. As the surrogate recoveries in the associated samples were acceptable, data were not qualified based on these high surrogate %Ds.

PAHs by EPA Method 8270D-SIM – The laboratory noted that the %D for the surrogate terphenyl-d14 in the CCV associated with analytical batch 285645 was outside the control limits of ±20% (low). As the surrogate recoveries in the associated samples were acceptable, data were not qualified based on these high surrogate %Ds

The laboratory noted that PDI-RB-VV-180703 was analyzed for PAHs 24 minutes past the 12 hour tune window. A closing CCV was evaluated after the sample injection which passed the recovery criteria for all reporting targets and surrogates; therefore, data were not qualified based on this sample injection outside the tune window.

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-B458, PDI-SG-B470, PDI-SG-B469, PDI-SG-B456, PDI-SG-B462, PDI-SG-B464, PDI-SG-B466, and PDI-SG-B468.

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 ±15% (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 in all sediment samples was exceeded by 23-24 days due to delay in authorization of analysis. The results for mercury in all sediment samples were qualified as estimated and flagged 'J' based on the holding time exceedance.

2. Blanks – Acceptable except as noted below:

   General – One rinsate blank was submitted with this laboratory group. Arsenic (0.00021 mg/L) and manganese (0.0011 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) – Acceptable except as noted below:

   Metals by Method 6020B – An MS/MSD and PDS was performed using PDI-SG-B463. Results were acceptable.

   An MS/MSD and PDS was performed using PDI-SG-B468. The percent recovery for manganese in the PDS (-15%) was outside of the control limits of 80-120%. The sample concentration for manganese in PDI-SG-B468 was greater than four times the spike added; therefore, data were not qualified based on the PDS result.

   Mercury by Method 7471A – An MS/MSD was performed using PDI-SG-B463. Results were acceptable.

Mercury by Method 7470A – An MS/MSD was performed using PDI-RB-VV-180703. Results were acceptable.

5. Laboratory Duplicate – Acceptable

Metals by Method 6020B – A laboratory duplicate was performed using PDI-SG-B463. Results were comparable.

A laboratory duplicate was performed using PDI-SG-B468. The RPDs for cadmium (28%) and lead (30%) exceeded the control limit of 20%. The result for cadmium in PDI-SG-B468 was less than five times the reporting limit; therefore, data were not qualified based on the elevated laboratory duplicate RPD. The result for lead in PDI-SG-B468 was qualified as estimated and flagged 'J' based on the elevated laboratory duplicate RPD.

Mercury by Method 7471A – A laboratory duplicate was performed using PDI-SG-B463. Results were comparable.

Mercury by Method 7470A – A laboratory duplicate was performed using PDI-RB-VV-180703. Results were comparable.

6. Serial Dilution – Acceptable

Metals by Method 6020B – Serial dilutions were performed using PDI-SG-B468 and PDI-SG-B463. 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/Moisture Content at 70°C – The 7-day holding time indicated for total solids in the QAPP was exceeded for all samples by 7-67 days except total solids in PDI-SG-B466. 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 (0.37 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 was detected in the method blank associated with analytical batch 278904 (63.1 mg/kg) at a concentration between the MDL and the reporting limit. 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 – MS/MSDs were performed using PDI-SG-B466 and PDI-SG-B463. Results were acceptable.

6. Laboratory Replicate – Acceptable

   TOC by Method 9060 – Laboratory duplicates and triplicates were performed using PDI-SG-B466 and PDI-SG-B463. Results were comparable.

7. Reporting Limits – Acceptable

   TOC by Method 9060 – 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.

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 580-78604-1, 580-78604-6, and 580-78604-7 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Downtown/Upriver
TestAmerica Laboratory Groups: 580-78604-1, 580-78604-6, and 580-78604-7

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B458	580-78604-1	SW7471A	Mercury	0.10	mg/kg	0.10 J	h
PDI-SG-B470	580-78604-2	SW7471A	Mercury	0.034 J	mg/kg	0.034 J	h
PDI-SG-B469	580-78604-3	SW7471A	Mercury	0.036 J	mg/kg	0.036 J	h
PDI-SG-B456	580-78604-4	SW7471A	Mercury	0.036 J	mg/kg	0.036 J	h
PDI-SG-B456	580-78604-4	SW8270D	Bis(2-ethylhexyl)phthalate	130 J	ug/kg	130 J	bl
PDI-SG-B462	580-78604-5	SW7471A	Mercury	0.059	mg/kg	0.059 J	h
PDI-SG-B463	580-78604-6	SW7471A	Mercury	0.032 J	mg/kg	0.032 J	h
PDI-SG-B463	580-78604-6	SW8270DSIM	Benz(a)anthracene	80	ug/kg	80 J	m
PDI-SG-B463	580-78604-6	SW8270DSIM	Benzo(a)pyrene	110	ug/kg	110 J	m
PDI-SG-B463	580-78604-6	SW8270DSIM	Benzo(b)fluoranthene	150	ug/kg	150 J	m,md
PDI-SG-B463	580-78604-6	SW8270DSIM	Benzo(g,h,i)perylene	100	ug/kg	100 J	m
PDI-SG-B463	580-78604-6	SW8270DSIM	Chrysene	160	ug/kg	160 J	m
PDI-SG-B463	580-78604-6	SW8270DSIM	Fluoranthene	350	ug/kg	350 J	m
PDI-SG-B463	580-78604-6	SW8270DSIM	Naphthalene	25 J	ug/kg	25 J	m
PDI-SG-B463	580-78604-6	SW8270DSIM	Phenanthrene	310	ug/kg	310 J	m
PDI-SG-B463	580-78604-6	SW8270DSIM	Pyrene	380	ug/kg	380 J	m
PDI-SG-B464	580-78604-7	SW7471A	Mercury	0.049	mg/kg	0.049 J	h
PDI-SG-B466	580-78604-8	SW7471A	Mercury	0.038 J	mg/kg	0.038 J	h
PDI-SG-B466	580-78604-8	SW8270DSIM	2-Methylnaphthalene	8.7 U	ug/kg	8.7 UJ	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Acenaphthene	8.7 U	ug/kg	8.7 UJ	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Acenaphthylene	1.2 J	ug/kg	1.2 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Anthracene	2.2 J	ug/kg	2.2 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Benz(a)anthracene	2.7 J	ug/kg	2.7 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Benzo(a)pyrene	3.0 J	ug/kg	3.0 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Benzo(b)fluoranthene	4.4 J	ug/kg	4.4 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Benzo(g,h,i)perylene	2.2 J	ug/kg	2.2 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Benzo(k)fluoranthene	1.3 J	ug/kg	1.3 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Chrysene	3.8 J	ug/kg	3.8 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Dibenz(a,h)anthracene	8.7 U	ug/kg	8.7 UJ	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Fluoranthene	4.8 J	ug/kg	4.8 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Fluorene	1.1 J	ug/kg	1.1 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Indeno(1,2,3-cd)pyrene	1.6 J	ug/kg	1.6 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Naphthalene	2.1 J	ug/kg	2.1 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Phenanthrene	7.5 J	ug/kg	7.5 J	s
PDI-SG-B466	580-78604-8	SW8270DSIM	Pyrene	7.0 J	ug/kg	7.0 J	s
PDI-SG-B468	580-78604-9	SW7471A	Mercury	0.026 J	mg/kg	0.026 J	h
PDI-SG-B468	580-78604-9	SW8270DSIM	Acenaphthylene	8.0 U	ug/kg	8.0 UJ	l
PDI-SG-B468	580-78604-9	SW6020B	Lead	6.9	mg/kg	6.9 J	ld
PDI-SG-B429	580-78604-10	SW7471A	Mercury	0.040 J	mg/kg	0.040 J	h
PDI-SG-B429	580-78604-10	SW8270D	Bis(2-ethylhexyl)phthalate	150 J	ug/kg	150 J	bl

Notes:
bl - laboratory blank contamination
h - holding time
J - estimated value
l - laboratory control sample recovery
ld - laboratory duplicate RPD
m - matrix spike recovery
md - matrix spike/matrix spike duplicate RPD
mg/kg - milligram per kilogram
RPD - relative percent difference
s - surrogate recovery
U - Compound was analyzed for, but not detected above the value shown.
ug/kg - microgram per kilogram