

Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
 Portland Harbor Superfund Site
 Fish Tissue

Laboratory: ALS Environmental, Kelso, WA

Laboratory Group: K1810425

Analyses/Method: bis(2-Ethylhexyl)phthalate, Pentachlorophenol, Total Metals, and Total Solids

Validation Level: Stage 2

AECOM Project

Number: 60566335 Task #2.12

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Completed on: January 25, 2019

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File Name: K1810425 DVR

SUMMARY

The data quality review of 20 fish tissue samples collected between August 20 and September 6, 2018, has been completed. Samples were analyzed for low-level bis(2-ethylhexyl)phthalate and pentachlorophenol (PCP) by EPA Method 8270D modified by selected ion monitoring (SIM), total metals by EPA Methods 6020A (arsenic) and 7471B (mercury), and total solids by ALS standard operating procedure (SOP) *Tissue Sample Preparation*, MET-TISP, Revision 11, dated February 23, 2017, at ALS Environmental (ALS) located in Kelso, Washington. All fish tissue sample aliquots for metals analysis were freeze dried prior to sample preparation in accordance with ALS SOP MET-TISP. Sample aliquots for organic analysis were not freeze dried.

The method revisions referenced by ALS for the arsenic and mercury analyses did not match the method revisions identified in the project Quality Assurance Project Plan (QAPP). The QAPP method revisions for arsenic and mercury in fish tissue samples identified in the QAPP were in error. AECOM approved the method revisions used by ALS, and ALS is accredited for fish tissue analysis using the method revisions noted in the previous paragraph. There are no substantive differences between the method revisions and data quality was not adversely impacted.

The analyses were performed in general accordance with the methods specified in EPA's *Test Methods for Evaluating Solid Waste (SW-846)* and the laboratory's fish tissue preparation SOP. 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 group K1810425:

Sample ID	Laboratory ID
PDI-TF-SMB028	K1810425-001
PDI-TF-SMB039	K1810425-002
PDI-TF-SMB040	K1810425-003
PDI-TF-SMB020	K1810425-004
PDI-TF-SMB010	K1810425-005
PDI-TF-SMB022	K1810425-006
PDI-TF-SMB024	K1810425-007
PDI-TF-SMB021	K1810425-008
PDI-TF-SMB023	K1810425-009
PDI-TF-SMB015	K1810425-010



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Sample ID	Laboratory ID
PDI-TF-SMB037	K1810425-011
PDI-TF-SMB011	K1810425-012
PDI-TF-SMB128	K1810425-013
PDI-TF-SMB053	K1810425-014
PDI-TF-SMB009	K1810425-015
PDI-TF-SMB058	K1810425-016
PDI-TF-SMB066	K1810425-017
PDI-TF-SMB109	K1810425-018
PDI-TF-SMB069	K1810425-019
PDI-TF-SMB056	K1810425-020

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

The samples were frozen in the field and submitted to SGS-AXYS, located in Sidney, British Columbia, Canada, for homogenization. SGS-AXYS submitted aliquots of the homogenized fish tissue samples to ALS. The aliquots were received by ALS on October 24, 2018. Upon receipt by ALS, the sample jar information was compared to the chain-of-custody (COC) and the cooler temperatures were recorded. No discrepancies related to sample identification were noted by ALS. Five of the six coolers were received at temperatures above the QAPP limits of less than -10°C at temperatures between -9.4°C and 4°C. ALS noted that one or more samples were received partially thawed. Per ALS protocol, samples were immediately placed in frozen storage upon receipt. There were no adverse impacts to the data based on partially thawed samples. Data were not qualified based on the elevated cooler temperatures.

ORGANIC ANALYSIS

Samples were analyzed for bis(2-ethylhexyl)phthalate and PCP by EPA Method 8270D-SIM.

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

bis(2-Ethylhexyl)phthalate (17 ug/kg) was detected at a concentration between the method detection limit (MDL) and the reporting limit in the method blank extracted on November 14, 2018. The result for bis(2-ethylhexyl)phthalate in PDI-TF-SMB128 was reported at a concentration less than the reporting limit but above the MDL in a sample that was diluted; therefore, the result for bis(2 ethylhexyl)phthalate in this sample was qualified as estimated and flagged 'J' based on this method blank result.



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4. Surrogates – Acceptable except as noted below:

The percent recovery for terphenyl-d14 (52%) was below the control limits of 55-137% in the laboratory control sample extracted on November 14, 2018. Data were not qualified based on the surrogate recoveries in QC samples.

5. Internal Standards – Acceptable
6. Laboratory Control Sample (LCS) – Acceptable
7. Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Acceptable

An MS/MSD was performed using PDI-TF-SMB037. Results were acceptable.

8. Reporting Limits – Acceptable except as noted below:

The result for bis(2-ethylhexyl)phthalate in PDI-TF-SMB128 was flagged 'J' by the laboratory to indicate the reported concentration was between the MDL and the reporting limit. Laboratory 'J'-flagged results are considered estimated. As the result is between the MDL and the reporting limit, there is a greater level of uncertainty associated with the numerical result.

The reporting limits for bis(2-ethylhexyl)phthalate and PCP in these samples were elevated due to dilutions for matrix interference. The reporting limits and MDLs for these analytes exceeded the cleanup levels in all samples reported in laboratory group K1810425.

METALS ANALYSES

Samples were analyzed for arsenic and mercury by the methods identified in the introduction to this report.

1. Holding Times – Acceptable
2. ICP-MS Instrument Performance Check & Tuning (where applicable) – Acceptable
3. Initial and Continuing Calibrations – Acceptable
4. Blanks – Acceptable except as noted below:

Total Mercury by Method 7471B – Mercury was detected in multiple continuing calibration blanks analyzed on November 20, 2018, at concentrations with absolute values between the MDLs and the reporting limits. The results for mercury in all samples reported in laboratory group K1810425 were qualified as estimated and flagged 'J' based on these continuing calibration blanks.

5. Internal Standards – Acceptable (where applicable)
6. Laboratory Control Sample (LCS) - Acceptable



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7. Matrix Spike/Matrix Spike Duplicate (MS/MSD) and Post Digestion Spike (PDS) – Acceptable

General – An MS/MSD and PDS for arsenic and mercury were performed using PDI-TF-SMB037. Results were acceptable.

8. Standard Reference Material (SRM) – Acceptable

General – Two SRMs from the National Research Council of Canada (NRCC) for each metal were analyzed. The SRMs were identified as Dorm-4 and Tort-3. Results were acceptable as shown below.

Analyte	True Value	Lab Result	Percent Recovery	Control Limits
Arsenic Dorm-4	6.8	6.8	100%	6.52-7.36
Arsenic Tort-3	59.5	66.2	111%	47.8-79.4
Mercury Dorm-4	0.41	0.33	80%	0.177-0.795
Mercury Tort-3	0.292	0.238	82%	0.076-0.669

9. Serial Dilution – Acceptable

Total Arsenic by Method 6020A – A serial dilution was performed using PDI-TF-SMB037. Results were comparable.

10. ICP Interference Check Samples – Acceptable

11. Reporting Limits – Acceptable

Total Arsenic by Method 6020A – The results for arsenic in PDI-TF-SMB128 and PDI-TF-SMB109 were flagged 'J' by the laboratory to indicate the reported concentrations were between the MDLs the reporting limits. As described above, laboratory 'J'-flagged results are considered estimated results.

CONVENTIONAL ANALYSIS

Samples were analyzed for total solids by ALS SOP MET-TISP.

1. Holding Times – Acceptable
2. Laboratory Duplicate – Acceptable

A laboratory duplicate was performed using PDI-TF-SMB037. Results were comparable.

3. Reporting Limits – Acceptable

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 K1810425 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Fish Tissue
ALS Kelso Laboratory Group: K1810425

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-TF-SMB028	K1810425-001	SW7471B	Mercury	0.118	mg/kg	0.118 J	bl
PDI-TF-SMB039	K1810425-002	SW7471B	Mercury	0.122	mg/kg	0.122 J	bl
PDI-TF-SMB040	K1810425-003	SW7471B	Mercury	0.175	mg/kg	0.175 J	bl
PDI-TF-SMB020	K1810425-004	SW7471B	Mercury	0.125	mg/kg	0.125 J	bl
PDI-TF-SMB010	K1810425-005	SW7471B	Mercury	0.070	mg/kg	0.070 J	bl
PDI-TF-SMB022	K1810425-006	SW7471B	Mercury	0.139	mg/kg	0.139 J	bl
PDI-TF-SMB024	K1810425-007	SW7471B	Mercury	0.137	mg/kg	0.137 J	bl
PDI-TF-SMB021	K1810425-008	SW7471B	Mercury	0.190	mg/kg	0.190 J	bl
PDI-TF-SMB023	K1810425-009	SW7471B	Mercury	0.160	mg/kg	0.160 J	bl
PDI-TF-SMB015	K1810425-010	SW7471B	Mercury	0.106	mg/kg	0.106 J	bl
PDI-TF-SMB037	K1810425-011	SW7471B	Mercury	0.136	mg/kg	0.136 J	bl
PDI-TF-SMB011	K1810425-012	SW7471B	Mercury	0.145	mg/kg	0.145 J	bl
PDI-TF-SMB128	K1810425-013	SW7471B	Mercury	0.130	mg/kg	0.130 J	bl
PDI-TF-SMB128	K1810425-013	SW8270DSIM	Bis(2-ethylhexyl)phthalate	580 JD	ug/kg	580 J	bl
PDI-TF-SMB053	K1810425-014	SW7471B	Mercury	0.171	mg/kg	0.171 J	bl
PDI-TF-SMB009	K1810425-015	SW7471B	Mercury	0.081	mg/kg	0.081 J	bl
PDI-TF-SMB058	K1810425-016	SW7471B	Mercury	0.075	mg/kg	0.075 J	bl
PDI-TF-SMB066	K1810425-017	SW7471B	Mercury	0.114	mg/kg	0.114 J	bl
PDI-TF-SMB109	K1810425-018	SW7471B	Mercury	0.133	mg/kg	0.133 J	bl
PDI-TF-SMB069	K1810425-019	SW7471B	Mercury	0.079	mg/kg	0.079 J	bl
PDI-TF-SMB056	K1810425-020	SW7471B	Mercury	0.100	mg/kg	0.100 J	bl

Notes:

- bl - laboratory blank contamination
- D - result was reported from a dilution
- J - estimated value
- mg/kg - milligram per kilogram
- ug/kg - microgram per kilogram