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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site Surface Sediment – Stratified Random
Laboratory:	ALS Environmental, Kelso, WA
Laboratory Group:	K1805714
Analyses/Method:	Chlorinated Pesticides, Tributyltin, Polycyclic Aromatic Hydrocarbons (PAHs), bis(2-Ethylhexyl)phthalate, and Total Solids
Validation Level:	Stage 2A
AECOM Project Number:	60566335 Task #2.12
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SUMMARY

The data quality review of 15 surface sediment samples collected between May 20 and May 31, 2018, has been completed. Samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/MS/MS), tributyltin by Krone et al., PAHs by EPA Method 8270D modified by selected ion monitoring (SIM), bis(2-ethylhexyl)phthalate by EPA Method 8270D, and total solids by EPA Method 160.3-modified at ALS Environmental (ALS) located in Kelso, Washington. The analyses were performed in general accordance with the methods specified in EPA's *Test Methods for Evaluating Solid Waste* (SW-846), *Method 1699: Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS*, December 2007 (modified by ALS SOP SVM-PESTMS2), and *Methods for Chemical Analysis of Water and Wastes*, March 1983, 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*, Environmental Conservation Division, Northwest and Alaska Fisheries Center, National Marine Fisheries Service, NOAA, November,1988. 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 K1805714:

Sample ID	Laboratory ID
PDI-SG-B208-BL1	K1805714-001
PDI-SG-B389-BL1	K1805714-002
PDI-SG-B391-BL1	K1805714-003
PDI-SG-B392-BL1	K1805714-004
PDI-SG-B428-BL1	K1805714-005
PDI-SG-B427-BL1	K1805714-006
PDI-SG-B426-BL1	K1805714-007
PDI-SG-B415-BL1	K1805714-008
PDI-SG-B320-BL1	K1805714-009
PDI-SG-B404-BL1	K1805714-010
PDI-SG-B419-BL1	K1805714-011
PDI-SG-B421-BL1	K1805714-012
PDI-SG-B422-BL1	K1805714-013

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Sample ID	Laboratory ID
PDI-SG-B192-BL1	K1805714-014
PDI-SG-B183-BL1	K1805714-015

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 results reported in this sample set are included in Table 1.

SAMPLE RECEIPT

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. One cooler was received at a temperature below the EPA-recommended limits of greater than 0°C and less than or equal to 6°C at -1.0°C. Sample containers were intact upon laboratory receipt; therefore, data were not qualified based on the low cooler temperature. The samples in this laboratory group were frozen after sample collection and held in the AECOM storage facility until they were shipped to ALS on June 15, 2018.

ORGANIC ANALYSES

Samples were analyzed for chlorinated pesticides, tributyltin, PAHs, and bis(2-ethylhexyl)phthalate by the methods identified in the introduction to this report.

1. Holding Times – Acceptable except as noted below:

   General – Samples in this laboratory group were extracted 33-73 days past the method-recommended holding time of 14 days after sample collection. The samples were frozen immediately after collection and held in the AECOM storage facility freezer prior to shipment to ALS-Kelso. Per ALS-Kelso protocol, the samples were frozen in archive until extraction and were thawed for less than 14 days; therefore, the sample was not re-extracted outside the holding time.

2. Initial and Continuing Calibration Verifications – Acceptable

3. Blanks – Acceptable

   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. Data were not qualified based on rinsate blank results.

4. Surrogates – Acceptable

5. Internal Standards – Acceptable where applicable

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

   Chlorinated Pesticides by EPA Method 1699-modified – The percent recoveries for the following analytes in the LCS/LCSDs were outside the control limits:

Extraction Date	Analyte	LCS	LCSD	Control limit
July 19, 2018	2,4'-DDT	127%	123%	77-118%
	4,4'-DDE	134%	ok	66-132%
	Aldrin	ok	123%	74-122%
	alpha-Chlordane	164%	164%	74-130%
	cis-Nonachlor	200%	197%	69-134%
	gamma-Chlordane	147%	155%	76-128%
	Heptachlor	132%	131%	81-114%
	trans-Nonachlor	192%	181%	76-124%
August 15, 2018	alpha-Chlordane	158%	NA	74-130%
	cis-Nonachlor	307%	NA	69-134%
	gamma-Chlordane	148%	NA	76-128%
	Heptachlor	80%	NA	81-114%
	trans-Nonachlor	173%	NA	76-124%

NA – not applicable ok - acceptable

   The results for alpha-chlordane, gamma-chlordane, and trans-nonachlor in PDI-SG-B392-BL1 were qualified as estimated and flagged 'J' based on the LCS/LCSD extracted on July 19, 2018. The results for all other chlorinated pesticides associated with this LCS/LCSD were either not detected in the associated samples or 2 out of 3 quality control parameters (LCS, LCSD, and/or relative percent difference [RPD]) were acceptable; therefore, no data were qualified based on these LCS/LCSD results.

   The result for heptachlor in PDI-SG-B208-BL1 was qualified as estimated and flagged 'UJ' based on the LCS extracted on August 15, 2018. The other chlorinated pesticides associated with this LCS were not detected in the associated sample; therefore, no data were qualified based on these LCS results.

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

   Chlorinated Pesticides by EPA Method 1699-modified – An MS/MSD was performed using PDI-SG-B208-BL1. The following percent recoveries were outside the control limits:

Analyte	MS	MSD	Control limit
alpha-Chlordane	171%	183%	31-156%
cis-Nonachlor	318%	348%	27-144%
trans-Nonachlor	189%	204%	35-153%

   The chlorinated pesticides noted in the above table were not detected in PDI-SG-B208-BL1; therefore, no data were qualified based on these MS/MSD results.

   Tributyltin by Krone et al. – An MS/MSD was performed using PDI-SG-B194-BL1 (laboratory group K1806076, discussed under separate cover). Results were acceptable.

PAHs by EPA Method 8270D-SIM – An MS/MSD was performed using PDI-SG-B404-BL1. The percent recoveries for fluoranthene (30%) and pyrene (27%) in the MSD were outside the control limits of 42-130% and 33-125%, respectively. The percent recoveries for fluoranthene and pyrene in the MS and the RPDs for the MS/MSD pair were acceptable; therefore, data were not qualified for these analytes based on the MSD results.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – An MS/MSD was performed using PDI-SG-B392-BL1. Results were acceptable.

8. Reporting Limits – Acceptable except as noted below:

General – One or more results were flagged 'J' by the laboratory to indicate the reported concentrations were above the method detection limits (MDLs) but below the reporting limits. 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.

Chlorinated Pesticides by EPA Method 1699-modified – The reporting limits for one or more pesticides reported as not detected in multiple samples were elevated due to the moisture content and/or dilution due to matrix interference. The reporting limits and MDLs for dieldrin exceeded the cleanup level in all sediment samples reported in laboratory group K1805714.

Tributyltin by Krone et al. – The reporting limits for tributyltin reported as not detected in multiple samples were elevated due to moisture content. The elevated reporting limits and MDLs do not exceed the cleanup level.

CONVENTIONAL ANALYSIS

Soil samples were analyzed for total solids by EPA Method 160.3-modified.

1. Holding Times – Acceptable except as noted below:

Samples reported in this laboratory group were frozen and held in archive at the AECOM storage facility immediately after collection. The frozen samples were submitted to ALS on June 16, 2018, and the total solids analyses were performed on June 19, 2018, 12 to 23 days past the 7-day holding time indicated for total solids described in the QAPP. No data qualifiers were assigned based on the holding time exceedance.

2. Laboratory Duplicate – Acceptable

Laboratory duplicates were performed using PDI-SG-B392-BL1 and PDI-SG-B192-BL1 Results were comparable.

3. Reporting Limits – Acceptable

OVERALL ASSESSMENT OF DATA

The data reported in this laboratory group is considered usable for meeting project objectives. The completeness for laboratory group K1805714 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Stratified Random
ALS Kelso Laboratory Group: K1805714

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B208-BL1	K1805714-001	CWA1699M	Heptachlor	1.5 U	ug/kg	1.5 UJ	l
PDI-SG-B392-BL1	K1805714-004	CWA1699M	alpha-Chlordane	1.4 J	ug/kg	1.4 J	l
PDI-SG-B392-BL1	K1805714-004	CWA1699M	gamma-Chlordane	3.1 J	ug/kg	3.1 J	l
PDI-SG-B392-BL1	K1805714-004	CWA1699M	trans-Nonachlor	1.8 J	ug/kg	1.8 J	l

Notes:
J - estimated value
l - laboratory control sample recoveries
ug/kg - microgram per kilogram
U - Compound was analyzed for, but not detected above the value shown.