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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:	K1803202
Analyses/Method:	Chlorinated Pesticides, Tributyltin, Polycyclic Aromatic Hydrocarbons (PAHs), bis(2-Ethylhexyl)phthalate, and Total Solids
Validation Level:	Stage 4
AECOM Project Number:	60566335, Task #2.12
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SUMMARY

The data quality review of 14 surface sediment samples collected on April 4 and April 5, 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/or 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 K1803202:

Sample ID	Laboratory ID
PDI-SG-B049-BL1	K1803202-001
PDI-SG-B048-BL1	K1803202-002
PDI-SG-B047-BL1	K1803202-003
PDI-SG-B051-BL1	K1803202-004
PDI-SG-B050-BL1	K1803202-005
PDI-SG-B055-BL1	K1803202-006
PDI-SG-B052-BL1	K1803202-007
PDI-SG-B053-BL1	K1803202-008
PDI-SG-B057-BL1	K1803202-009
PDI-SG-B056-BL1	K1803202-010
PDI-SG-B058-BL1	K1803202-011
PDI-SG-B060-BL1	K1803202-012
PDI-SG-B060-BL1-D (Duplicate of PDI-SG-B060-BL1)	K1803202-013
PDI-SG-B061-BL1	K1803202-014

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 temperature was recorded. No discrepancies related to sample identification were noted by ALS, and the cooler was received at a temperature within the EPA-recommended limits of greater than 0°C and less than or equal to 6°C.

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

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

   Chlorinated Pesticides by EPA Method 1699-modified – The percent difference (%D) for cis-nonachlor (-28.3%) was outside the control limit of ±25% and the percent recovery for heptachlor-13C10 (340%) was outside the control limits of 50-200% in the continuing calibration verification (CCV) analyzed on May 21, 2018. The results for cis-nonachlor and heptachlor in all samples reported in laboratory group K1803202 were qualified as estimated and flagged 'UJ' based on these CCV results.

   PAHs by Method 8270D-SIM – The %Ds in the following CCVs were outside the control limit of ±20%.

Analysis Date	Analyte	Percent Difference
4/23/18	Pyrene	30%
4/24/18	Pyrene	33%
5/1/18	2-Methylnaphthalene	-21%

   The results for pyrene in PDI-SG-B049-BL1, PDI-SG-B052-BL1, PDI-SG-B057-BL1, PDI-SG-B058-BL1, PDI-SG-B060-BL1, and PDI-SG-B060-BL1-D and 2-methylnaphthalene in PDI-SG-B055-BL1 were qualified as estimated and flagged 'J' based on these CCV results.

3. Blanks – Acceptable except as noted below:

   General – A rinsate blank was collected on April 11, 2018, was reported with laboratory group K1803515 (laboratory ID K1803515-024), and is applicable to the samples collected in this

laboratory group. The following analytes were detected in the rinsate blank at concentrations between the method detection limits (MDLs) and the reporting limits:

Analysis	Analyte	Result
Chlorinated Pesticides	4,4'-DDD	0.16 ng/L
	4,4'-DDE	0.16 ng/L
	4,4'-DDT	0.14 ng/L
	gamma-Chlordane	0.24 ng/L
	Heptachlor	0.18 ng/L
PAHs	Naphthalene	0.0025 ug/L
	Phenanthrene	0.0015 ug/L
	Pyrene	0.0012 ug/L
	Benz(a)anthracene	0.0028 ug/L
bis(2-Ethylhexyl)phthalate	bis(2-ethylhexyl)Phthalate	0.94 ug/L

The results for naphthalene, phenanthrene, benz(a)anthracene, and bis(2-ethyhexyl)phthalate in this rinsate blank were qualified as not detected and flagged 'U' at the reporting limits based on the associated method blank results. Data were not qualified based on rinsate blank detections.

PAHs by Method 8270D-SIM – Phenanthrene (0.98 ug/kg) was detected at a concentration between the MDL and the reporting limit in the method blank extracted on April 12, 2018. Phenanthrene was reported at concentrations above the reporting limits and significantly above the blank concentration in the samples associated with this method blank; therefore, data were not qualified based on this method blank result.

4. Surrogates – Acceptable except as noted below:

Chlorinated Pesticides by EPA Method 1699-modified – The percent recovery for the surrogate heptachlor-13C10 (226%) in PDI-SG-B052-BL1 exceeded the control limits of 10-177%. The result for heptachlor in PDI-SG-B052-BL1 was qualified based on the associated CCV results as described in Section 2; therefore, no further qualification based on this surrogate recovery was necessary.

5. Internal Standards – Acceptable where applicable

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

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

Extraction Date	Analyte	LCS	Control limits
4/13/18	2,4'-DDT	141%	77-118%
	alpha-Chlordane	163%	74-130%
	cis-Nonachlor	195%	69-134%
	gamma-Chlordane	155%	76-128%
	trans-Nonachlor	183%	76-124%

alpha-Chlordane, cis-nonachlor, and gamma-chlordane were not detected in the associated samples; therefore, data were not qualified for these analytes based on these LCS results. The results for 2,4'-DDT in PDI-SG-B052-BL1 and trans-nonachlor in PDI-SG-B056-BL1 and PDI-SG-B058-BL1 were qualified as estimated and flagged 'J' based on these LCS results.

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

General - An MS/MSD was performed using PDI-SG-B047-BL1 for all organic analyses. Results were acceptable with the following exceptions.

Chlorinated Pesticides by EPA Method 1699-modified – The following percent recoveries for the following analytes were outside control limits in the MS/MSD.

Analyte	MS	MSD	Control Limits	RPD CL=40%
2,4'-DDE	ok	ok	32-169%	91%
alpha-Chlordane	223%	173%	31-156%	77%
cis-Nonachlor	327%	281%	27-144%	148%
gamma-Chlordane	192%	ok	31-158%	60%
trans-Nonachlor	244%	236%	35-153%	108%

ok – acceptable RPD – relative percent difference CL – control limit

Results for the pesticides noted in the above table were either reported as not detected or 2 out of 3 quality control parameters (MS, MSD, and/or RPD) were acceptable; therefore, no data were qualified based on these MS/MSD results.

PAHs by Method 8270D-SIM – The following percent recoveries were outside control limits in the MS/MSD.

Analyte	MS	MSD	Control Limits
Naphthalene	29%	31%	37-104%
Benzo(a)pyrene	38%	ok	39-130%
Benzo(g,h,i)perylene	34%	ok	35-140%

As the percent recoveries for benzo(a)pyrene and benzo(g,h,i)perylene in the MSD and the RPDs for the MS/MSD pair were acceptable, data were not qualified for benzo(a)pyrene and benzo(g,h,i)perylene in PDI-SG-B047-BL1. The result for naphthalene in PDI-SG-B047-BL1 was qualified as estimated and flagged 'J' based on these MS/MSD results.

8. Field Duplicate – Acceptable except as noted below:

General - A field duplicate was submitted for PDI-SG-B060-BL1 and identified as PDI-SG-B060-BL1-D. Results were comparable with the following exceptions.

Chlorinated Pesticides by EPA Method 1699-modified – The RPD for aldrin was not calculable for the PDI-SG-B060-BL1/PDI-SG-B060-BL1-D parent sample/field duplicate pair. The sample concentration for aldrin inPDI-SG-B060-BL1-D was less than five times the reporting limit; therefore, data were not qualified based on the field duplicate RPD.

PAHs by Method 8270D-SIM – The RPDs for the following analytes were more than 50% for the parent sample/field duplicate pair.

Analyte	RPD
Acenaphthylene	55%
Benzo(a)pyrene	60%
Benzo(b)fluoranthene	56%
Benzo(g,h,i)perylene	74%
Benzo(k)fluoranthene	60%
Chrysene	55%
Dibenzo(a,h)anthracene	61%
Indeno(1,2,3-cd)pyrene	63%

The results for these PAHs in PDI-SG-B060-BL1 and PDI-SG-B060-BL1-D were qualified as estimated and flagged 'J' based on the elevated field duplicate RPDs.

9. Calculation Checks – Acceptable

A calculation check was performed for sample results on one sample per calibration per method. The review confirmed the final results were correct as reported.

10. Reporting Limits and Chromatographic Review – Acceptable except as noted below:

General – Chromatograms/spectra were reviewed to confirm target analytes were properly identified. The review confirmed target analytes were properly identified and reported by the laboratory.

One or more results were flagged 'J' by the laboratory to indicate the reported concentrations were above the 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 the sediment samples in laboratory group K1803202.

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.

11. Other Items of Note:

Tributyltin by Krone et al. – The result for tributyltin in PDI-SG-B056-BL1 was flagged 'JP' by the laboratory to indicate that the RPD between the two analytical columns was more than 40%. The result for tributyltin in PDI-SG-B056-BL1 was qualified as estimated and flagged 'J' based on the dual-column RPD.

CONVENTIONAL ANALYSES

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

1. Holding Times – Acceptable

2. Field Duplicate – Acceptable

   A field duplicate was submitted for PDI-SG-B060-BL1 and identified as PDI-SG-B060-BL1-D. Results were comparable.

3. Laboratory Duplicate – Acceptable

   Laboratory duplicates were performed using PDI-SG-B047-BL1 and PDI-SG-B058-BL1. Results were comparable.

4 Calculation Checks – Acceptable

   A calculation check was performed for sample results on one sample per calibration. The review confirmed the final results were correct as reported.

5 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 K1803202 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Stratified Random
ALS Kelso Laboratory Group: K1803202

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B049-BL1	K1803202-001	CWA1699M	cis-Nonachlor	0.56 U	ug/kg	0.56 UJ	c
PDI-SG-B049-BL1	K1803202-001	CWA1699M	Heptachlor	0.56 U	ug/kg	0.56 UJ	c
PDI-SG-B049-BL1	K1803202-001	SW8270DSIM	Pyrene	240 D	ug/kg	240 J	c
PDI-SG-B048-BL1	K1803202-002	CWA1699M	cis-Nonachlor	0.49 U	ug/kg	0.49 UJ	c
PDI-SG-B048-BL1	K1803202-002	CWA1699M	Heptachlor	0.31 U	ug/kg	0.31 UJ	c
PDI-SG-B047-BL1	K1803202-003	CWA1699M	cis-Nonachlor	0.60 U	ug/kg	0.60 UJ	c
PDI-SG-B047-BL1	K1803202-003	CWA1699M	Heptachlor	0.60 U	ug/kg	0.60 UJ	c
PDI-SG-B047-BL1	K1803202-003	SW8270DSIM	Naphthalene	22	ug/kg	22 J	m
PDI-SG-B051-BL1	K1803202-004	CWA1699M	cis-Nonachlor	0.61 U	ug/kg	0.61 UJ	c
PDI-SG-B051-BL1	K1803202-004	CWA1699M	Heptachlor	0.61 U	ug/kg	0.61 UJ	c
PDI-SG-B050-BL1	K1803202-005	CWA1699M	cis-Nonachlor	0.49 U	ug/kg	0.49 UJ	c
PDI-SG-B050-BL1	K1803202-005	CWA1699M	Heptachlor	0.33 U	ug/kg	0.33 UJ	c
PDI-SG-B055-BL1	K1803202-006	CWA1699M	cis-Nonachlor	0.49 U	ug/kg	0.49 UJ	c
PDI-SG-B055-BL1	K1803202-006	CWA1699M	Heptachlor	0.40 U	ug/kg	0.40 UJ	c
PDI-SG-B055-BL1	K1803202-006	SW8270DSIM	2-Methylnaphthalene	4.3	ug/kg	4.3 J	c
PDI-SG-B052-BL1	K1803202-007	CWA1699M	2,4-DDT	0.89	ug/kg	0.89 J	l
PDI-SG-B052-BL1	K1803202-007	CWA1699M	cis-Nonachlor	0.49 U	ug/kg	0.49 UJ	c
PDI-SG-B052-BL1	K1803202-007	CWA1699M	Heptachlor	0.46 U	ug/kg	0.46 UJ	c
PDI-SG-B052-BL1	K1803202-007	SW8270DSIM	Pyrene	2300 D	ug/kg	2300 J	c
PDI-SG-B053-BL1	K1803202-008	CWA1699M	cis-Nonachlor	0.49 U	ug/kg	0.49 UJ	c
PDI-SG-B053-BL1	K1803202-008	CWA1699M	Heptachlor	0.34 U	ug/kg	0.34 UJ	c
PDI-SG-B057-BL1	K1803202-009	CWA1699M	cis-Nonachlor	0.49 U	ug/kg	0.49 UJ	c
PDI-SG-B057-BL1	K1803202-009	CWA1699M	Heptachlor	0.41 U	ug/kg	0.41 UJ	c
PDI-SG-B057-BL1	K1803202-009	SW8270DSIM	Pyrene	270 D	ug/kg	270 J	c
PDI-SG-B056-BL1	K1803202-010	CWA1699M	cis-Nonachlor	0.57 U	ug/kg	0.57 UJ	c
PDI-SG-B056-BL1	K1803202-010	CWA1699M	Heptachlor	0.57 U	ug/kg	0.57 UJ	c
PDI-SG-B056-BL1	K1803202-010	CWA1699M	trans-Nonachlor	0.41 J	ug/kg	0.41 J	l
PDI-SG-B056-BL1	K1803202-010	Unger et al.	Tri-n-butyltin	1.3 JP	ug/kg	1.3 J	r
PDI-SG-B058-BL1	K1803202-011	CWA1699M	cis-Nonachlor	0.49 U	ug/kg	0.49 UJ	c
PDI-SG-B058-BL1	K1803202-011	CWA1699M	Heptachlor	0.49 U	ug/kg	0.49 UJ	c
PDI-SG-B058-BL1	K1803202-011	CWA1699M	trans-Nonachlor	0.38 J	ug/kg	0.38 J	l
PDI-SG-B058-BL1	K1803202-011	SW8270DSIM	Pyrene	240 D	ug/kg	240 J	c
PDI-SG-B060-BL1	K1803202-012	CWA1699M	cis-Nonachlor	0.49 U	ug/kg	0.49 UJ	c
PDI-SG-B060-BL1	K1803202-012	CWA1699M	Heptachlor	0.41 U	ug/kg	0.41 UJ	c
PDI-SG-B060-BL1	K1803202-012	SW8270DSIM	Acenaphthylene	28 D	ug/kg	28 J	fd
PDI-SG-B060-BL1	K1803202-012	SW8270DSIM	Pyrene	1100 D	ug/kg	1100 J	c
PDI-SG-B060-BL1	K1803202-012	SW8270DSIM	Chrysene	280 D	ug/kg	280 J	fd
PDI-SG-B060-BL1	K1803202-012	SW8270DSIM	Benzo(b)fluoranthene	230 D	ug/kg	230 J	fd
PDI-SG-B060-BL1	K1803202-012	SW8270DSIM	Benzo(k)fluoranthene	80 D	ug/kg	80 J	fd
PDI-SG-B060-BL1	K1803202-012	SW8270DSIM	Benzo(a)pyrene	280 D	ug/kg	280 J	fd
PDI-SG-B060-BL1	K1803202-012	SW8270DSIM	Indeno(1,2,3-cd)pyrene	210 D	ug/kg	210 J	fd
PDI-SG-B060-BL1	K1803202-012	SW8270DSIM	Dibenz(a,h)anthracene	32 D	ug/kg	32 J	fd
PDI-SG-B060-BL1	K1803202-012	SW8270DSIM	Benzo(g,h,i)perylene	240 D	ug/kg	240 J	fd
PDI-SG-B060-BL1-D	K1803202-013	CWA1699M	cis-Nonachlor	0.49 U	ug/kg	0.49 UJ	c
PDI-SG-B060-BL1-D	K1803202-013	CWA1699M	Heptachlor	0.40 U	ug/kg	0.40 UJ	c
PDI-SG-B060-BL1-D	K1803202-013	SW8270DSIM	Acenaphthylene	16 D	ug/kg	16 J	fd
PDI-SG-B060-BL1-D	K1803202-013	SW8270DSIM	Pyrene	1300 D	ug/kg	1300 J	c
PDI-SG-B060-BL1-D	K1803202-013	SW8270DSIM	Chrysene	160 D	ug/kg	160 J	fd
PDI-SG-B060-BL1-D	K1803202-013	SW8270DSIM	Benzo(b)fluoranthene	130 D	ug/kg	130 J	fd
PDI-SG-B060-BL1-D	K1803202-013	SW8270DSIM	Benzo(k)fluoranthene	43 D	ug/kg	43 J	fd
PDI-SG-B060-BL1-D	K1803202-013	SW8270DSIM	Benzo(a)pyrene	150 D	ug/kg	150 J	fd
PDI-SG-B060-BL1-D	K1803202-013	SW8270DSIM	Indeno(1,2,3-cd)pyrene	110 D	ug/kg	110 J	fd
PDI-SG-B060-BL1-D	K1803202-013	SW8270DSIM	Dibenz(a,h)anthracene	17 D	ug/kg	17 J	fd
PDI-SG-B060-BL1-D	K1803202-013	SW8270DSIM	Benzo(g,h,i)perylene	110 D	ug/kg	110 J	fd
PDI-SG-B061-BL1	K1803202-014	CWA1699M	cis-Nonachlor	0.50 U	ug/kg	0.50 UJ	c
PDI-SG-B061-BL1	K1803202-014	CWA1699M	Heptachlor	0.50 U	ug/kg	0.50 UJ	c

c - calibration issue
D - result reported from a dilution
fd - field duplicate RPD
J - estimated value
l - LCS recoveries
m - matrix spike recovery
P - dual-column RPD greater than 40%
r - dual column RPD
RPD - relative percent difference
ug/kg - microgram per kilogram
U - Compound was analyzed for, but not detected above the value shown.