

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: K1805180

Analyses/Method: Chlorinated Pesticides, Tributyltin, Polycyclic Aromatic Hydrocarbons (PAHs), bis(2-Ethylhexyl)phthalate, and Total Solids

Validation Level: Stage 2A

AECOM Project

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File Name: K1805180 DVR

SUMMARY

The data quality review of 11 surface sediment samples and two rinsate blanks collected between June 1 and June 3, 2018, has been completed. Samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/MS/MS), tributyltin by Krone et al (sediments) or Unger et al. (waters), 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, and/or Unger, MA et al., *Determination of Butyltins in Natural Waters by Flame Photometric Detection of Hexane Derivatives and Mass Spectrometric Confirmation*, *Chemosphere*, 1886,16(4):461-470. 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 K1805180:

Sample ID	Laboratory ID
PDI-SG-B186-BL1	K1805180-001
PDI-SG-B182-BL1	K1805180-002
PDI-SG-B187-BL1	K1805180-003
PDI-SG-B179-BL1	K1805180-004
PDI-SG-B110-BL1	K1805180-005
PDI-SG-B181-BL1	K1805180-006
PDI-SG-B189-BL1	K1805180-007
PDI-SG-B189-BL1-D (Duplicate of PDI-SG-B189-BL1)	K1805180-008
PDI-SG-B316-BL1	K1805180-009
PDI-SG-B317-BL1	K1805180-010
PDI-SG-B255-BL1	K1805180-011



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Sample ID	Laboratory ID
PDI-RB-VV-180602 (rinsate blank)	K1805180-012
PDI-RB-VV-180603 (rinsate blank)	K1805180-013

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 above the EPA-recommended limits of greater than 0°C and less than or equal to 6°C at 6.8°C. Data were not qualified based on the elevated cooler temperature.

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:

Chlorinated Pesticides by EPA Method 1699-modified – Samples PDI-SG-B182-BL1, PDI-SG-B179-BL1, PDI-SG-B189-D-BL1, and PDI-SG-B255-BL1 were re-extracted between 38 and 40 days past the method-recommended holding time of 14 days after sample collection. Per ALS-Kelso protocol, the samples were frozen in archive after the initial extractions and the samples were thawed for less than 14 days; therefore, the samples were not re-extracted outside the holding times.

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

General – Two rinsate blanks were collected on June 2 and June 3, 2018, were reported with this laboratory group (IDs K1808150-012 and K1808150-013), and are applicable to the samples collected in this laboratory group. The following analytes were detected in these rinsate blanks between the method detection limits (MDLs) and the reporting limits:

Sample Identification	Analysis	Analyte	Result
K1805180-012 (PDI-RB-VV-180602)	Chlorinated Pesticides	trans-Nonachlor	0.19 ng/L
	PAHs	Naphthalene	0.0071 ug/L
		2-Methylnaphthalene	0.0049 ug/L

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Sample Identification	Analysis	Analyte	Result
K1805180-012 (PDI-RB-VV-180602) (continued)	PAHs (continued)	Phenanthrene	0.0025 ug/L
		Fluoranthene	0.0015 ug/L
		Pyrene	0.0017 ug/L
		Benz(a)anthracene	0.0025 ug/L
	bis(2-Ethylhexyl)phthalate	bis(2-Ethylhexyl)phthalate	0.18 ug/L
K1805180-013 (PDI-RB-VV-180603)	PAHs	Naphthalene	0.0056 ug/L
		2-Methylnaphthalene	0.0030 ug/L
		Phenanthrene	0.0022 ug/L
		Benz(a)anthracene	0.0021 ug/L
	bis(2-Ethylhexyl)phthalate	bis(2-Ethylhexyl)phthalate	0.44 ug/L

One or more of these results were qualified as not detected based on the associated method blank results. Sediment data were not qualified based on rinsate blank detections.

PAHs by EPA Method 8270D-SIM – The following analytes were detected at concentrations between the MDLs and the reporting limits in the method blanks.

Extraction Date	Analyte	Result
June 5, 2018 (water)	Phenanthrene	0.0019 ug/L
	Fluoranthene	0.0014 ug/L
	Pyrene	0.0018 ug/L
	Benz(a)anthracene	0.0029 ug/L
	Chrysene	0.0010 ug/L
June 14, 2018 (sediment)	Fluoranthene	0.076 ug/kg
	Pyrene	0.075 ug/kg
	Benz(a)anthracene	0.050 ug/kg

Chrysene was not detected in the samples associated with the method blank extracted on June 5, 2018; therefore, data were not qualified for chrysene based on this method blank result. The results for phenanthrene, fluoranthene, pyrene, and benz(a)anthracene in PDI-RB-VV-180602 and phenanthrene and benz(a)anthracene in PDI-RB-VV-180603 were reported at concentrations between the MDLs and reporting limits, were qualified as not detected, and were flagged 'U' at the reporting limits based on the method blank extracted on June 5, 2018.

The results for fluoranthene, pyrene, and benz(a)anthracene in the sediment samples associated with the method blank extracted on June 14, 2018, were reported at concentrations significantly higher than the method blank concentrations; therefore, data were not qualified based on these method blank results.

4. Surrogates – Acceptable except as noted below:

Chlorinated Pesticides by EPA Method 1699-modified – The percent recovery for aldrin-13C12 (9%) was below the control limits of 10-143% in PDI-SG-B186-BL1. The result for aldrin in PDI-SG-B186-BL1 was qualified as estimated and flagged 'UJ' based on this surrogate result.

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With the exceptions of 4,4'-DDD-d4 and 4,4'-DDT-d4, all surrogates were recovered outside the control limits in the parent sample associated with batch QC (sample PDI-SG-S069, reported under separate cover). No data reported with this sample set were qualified based on these surrogate recoveries.

PAHs by EPA Method 8270D-SIM – The percent recoveries for the following surrogates were outside the control limits.

Sample	Surrogate	Percent Recovery	Control Limits
PDI-SG-B182-BL1	Fluorene-d10	270%	42-106%
	Terphenyl-d14	114%	41-102%
PDI-SG-B179-BL1	Fluorene-d10	279%	42-106%
	Terphenyl-d14	104%	41-102%

The samples listed in the table above were analyzed at 1,000x dilutions; therefore, data were not qualified based on these surrogate recoveries in these samples.

bis(2 Ethylhexyl)phthalate by EPA Method 8270D – The percent recoveries for p-terphenyl-d14 exceeded the control limits of 30-102% in the following samples.

Sample	Percent Recovery
PDI-SG-B182-BL1	121%
PDI-SG-B179-BL1	108%
LCS (batch KQ1807452)	103%

Data were not qualified based on surrogate recoveries in QC samples. The sediment samples listed in the table above were analyzed at 10x dilutions; therefore, data were not qualified based on these surrogate recoveries in these samples.

- Internal Standards – Acceptable where applicable
- 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 limits
June 5, 2018 (water)	2,4'-DDD	75%	77%	79-113%
	Aldrin	70%	80%	81-113%
June 14, 2018 (sediment)	2,4'-DDT	133%	NA	77-118%
	cis-Nonachlor	151%	NA	69-134%
	Heptachlor	115%	NA	81-114%
	trans-Nonachlor	131%	NA	76-124%
June 25, 2018 (sediment)	cis-Nonachlor	183%	NA	69-134%
	trans-Nonachlor	129%	NA	76-124%

NA – not applicable

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The results for 2,4'-DDD and aldrin in PDI-RB-VV-180602 and PDI-RB-VV-180603 were qualified as estimated and flagged 'UJ' based on the LCS/LCSD extracted on June 5, 2018.

cis-Nonachlor and heptachlor were not detected in the samples associated with the LCS extracted on June 14, 2018; therefore, data were not qualified for cis-nonachlor and heptachlor based on these elevated LCS recoveries. The results for 2,4'-DDT in PDI-SG-B189-BL1 and PDI-SG-B189-BL1-D and trans-nonachlor in PDI-SG-B316-BL1 were qualified as estimated and flagged 'J' based on the June 14, 2018, LCS results.

cis-Nonachlor and trans-nonachlor were not detected in the samples associated with the LCS extracted on June 25, 2018; therefore, data were not qualified for these analytes based on these elevated LCS recoveries.

Tributyltin by Krone et al. – The percent recovery for tributyltin (123%) was outside the control limits of 32-122% in the LCS extracted on June 6, 2018. The percent recovery for tributyltin in the LCSD and the relative percent difference for the LCS/LCSD pair were acceptable; therefore, data were not qualified based on the LCS recovery.

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

General – MS/MSDs were not performed in association with the rinsate blank samples. Precision and accuracy were assessed using the LCS/LCSD results.

Chlorinated Pesticides by EPA Method 1699-modified – MS/MSDs were performed using PDI-SG-S015 (laboratory group K1805178, discussed under separate cover) and PDI-SG-S069 (laboratory group K1804214, discussed under separate cover). Data in this laboratory group were not qualified based on these MS/MSD results. Qualification, if any, is discussed in the associated data validation report.

Tributyltin by Krone et al. – An MS/MSD was performed using PDI-SG-B189-BL1. Results were acceptable.

PAHs by EPA Method 8270D-SIM – An MS/MSD was performed using sediment sample PDI-SG-S015 (laboratory group K1805178, discussed under separate cover). Data in this laboratory group were not qualified based on these MS/MSD results. Qualification, if any, is discussed in the associated data validation report.

bis(2 Ethylhexyl)phthalate by EPA Method 8270D – An MS/MSD was performed using PDI-SG-B255-BL1. Results were acceptable.

8. Field Duplicate – Acceptable except as noted below:

General – A field duplicate was submitted for PDI-SG-B189-BL1 and identified as PDI-SG-B189-BL1-D. Results were comparable except as noted below.

Chlorinated Pesticides by EPA Method 1699-modified – The RPDs for 4,4'-DDE (55%) and 4,4'-DDT (73%) were more than 50% for the PDI-SG-S189-BL1/ PDI-SG-S189-BL1-D field duplicate pair. The results for 4,4'-DDT were less than five times the reporting limits; therefore, data were not qualified for 4,4'-DDT based on this field duplicate RPD. The results for 4,4'-DDE were qualified as estimated and flagged 'J' in PDI-SG-B189-BL1 and PDI-SG-B189-BL1-D based on this parent sample/field duplicate RPD.



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9. 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 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 K1805180.

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 ANALYSES

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

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

Laboratory duplicates were performed using PDI-SG-B186-BL1 and PDI-SG-S015 (laboratory group K1805178, discussed under separate cover). Results were comparable.

3. Field Duplicate – Acceptable

A field duplicate was submitted for PDI-SG-B189-BL1 and identified as PDI-SG-B189-BL1-D. Results were comparable.

4. 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 K1805180 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-B186-BL1	K1805180-001	CWA1699M	Aldrin	0.79 U	ug/kg	0.79 UJ	s
PDI-SG-B189-BL1	K1805180-007	CWA1699M	2,4'-DDT	1.0	ug/kg	1.0 J	l
PDI-SG-B189-BL1	K1805180-007	CWA1699M	4,4'-DDE	5.7	ug/kg	5.7 J	fd
PDI-SG-B189-BL1-D	K1805180-008	CWA1699M	2,4'-DDT	1.3	ug/kg	1.3 J	l
PDI-SG-B189-BL1-D	K1805180-008	CWA1699M	4,4'-DDE	10	ug/kg	10 J	fd
PDI-SG-B316-BL1	K1805180-009	CWA1699M	trans-Nonachlor	1.4 J	ug/kg	1.4 J	l
PDI-RB-VV-180602	K1805180-012	CWA1699M	2,4'-DDD	0.55 U	ng/L	0.55 UJ	l
PDI-RB-VV-180602	K1805180-012	CWA1699M	Aldrin	1.1 U	ng/L	1.1 UJ	l
PDI-RB-VV-180602	K1805180-012	SW8270DSIM	Phenanthrene	0.0025 J	ug/L	0.020 U	bl
PDI-RB-VV-180602	K1805180-012	SW8270DSIM	Fluoranthene	0.0015 J	ug/L	0.020 U	bl
PDI-RB-VV-180602	K1805180-012	SW8270DSIM	Pyrene	0.0017 J	ug/L	0.020 U	bl
PDI-RB-VV-180602	K1805180-012	SW8270DSIM	Benz(a)anthracene	0.0025 J	ug/L	0.020 U	bl
PDI-RB-VV-180603	K1805180-013	CWA1699M	2,4'-DDD	0.54 U	ng/L	0.54 UJ	l
PDI-RB-VV-180603	K1805180-013	CWA1699M	Aldrin	1.1 U	ng/L	1.1 UJ	l
PDI-RB-VV-180603	K1805180-013	SW8270DSIM	Phenanthrene	0.0022 J	ug/L	0.022 U	bl
PDI-RB-VV-180603	K1805180-013	SW8270DSIM	Benz(a)anthracene	0.0021 J	ug/L	0.022 U	bl

Notes:

- bl - method blank contamination
- fd - field duplicate RPD
- J - estimated value
- l - laboratory control sample recoveries
- ng/L - nanogram per liter
- s - surrogate recovery
- RPD - relative percent difference
- ug/kg - microgram per kilogram
- ug/L - microgram per liter
- 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.