Data Validation Report

Project:	Portland Harbor
Laboratory:	Alpha Analytical Laboratory
Environmental Test Record (ETR):	L1527785
Analyses/Method:	Polychlorinated biphenyls (PCBs), Chlorinated Pesticides, and Grain Size

Summary

Fifteen sediment samples were collected in Portland Harbor, Oregon on October 21, 2015, October 22, 2015, and October 23, 2015. Samples were analyzed for Polychlorinated biphenyls (PCBs) by EPA Method 8270D modified by selected ion monitoring mode (SIM), chlorinated pesticides hydrocarbons by EPA Method 8081B, and grain size by ASTM Method D422-63 by Alpha Analytical Laboratory located in Mansfield, Massachusetts. The laboratory provided Level 4 data packages containing samples results and associated quality assurance (QA) and quality control (QC) data, preparation logs, and raw instrument output. The following sediment samples are associated with the laboratory ETR L1527785.

Sample ID	Lab ID	Matrix
PH15-34-A	L1527785-01	Sediment
PH15-10-A	L1527785-02	Sediment
PH15-11-A	L1527785-03	Sediment
PH15-11-D	L1527785-04	Sediment
PH15-11-D-FD	L1527785-05	Sediment
PH15-28-A	L1527785-06	Sediment
PH15-31-A	L1527785-07	Sediment
PH15-40-A	L1527785-08	Sediment
PH15-40-B	L1527785-09	Sediment
PH15-40-C	L1527785-10	Sediment
PH15-40-D	L1527785-11	Sediment
PH15-43-A	L1527785-12	Sediment
PH15-50-A	L1527785-13	Sediment
PH15-50-C	L1527785-14	Sediment
PH15-50-D	L1527785-15	Sediment

The data have been independently validated using *USEPA Contact Laboratory Program National Functional Guidelines for Organic Superfund Methods Data Review* EPA-540-R-2017-002, dated January 2017. Validation includes reconstruction of the analytical data to verify that data are traceable and sufficiently complete in order for a qualified individual other than the originator to perform reconstruction of the data. The validation included the following checks:

- Sample Receipt/Transcription error check
- Sample preservation
- Sample holding times
- Tune Summary
- Initial calibration

- Continuing calibration verification (CCV)
- Laboratory blank contamination
- Equipment blank contamination
- Surrogate spike recoveries
- Internal Standard recoveries
- Matrix spike/Matrix spike duplicate (MS/MSD) recoveries, relative percent difference (RPD)
- Standard Reference Material Sediment accuracy check
- Laboratory control sample (LCS), LCS Duplicate (LCSD) recoveries, RPD values
- DDT/Endrin Breakdown
- Contract Required Quantitation Limit (CRQL)
- Field duplicate results
- Laboratory duplicate results
- Dual Column Confirmation
- Overall assessment of the data

Data validation is based on the QC criteria documented in *Portland Harbor Supplemental Sediment Study, Portland Oregon Quality Assurance Project Plan (QAPP),*[1] dated October 14, 2015, and the *Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Quality Assurance Project Plan (QAPP)*,[2] dated March 23, 2018. Data qualifiers assigned to results reported in this sample set are included in Table 1. Reason codes and explanations for qualified data are provided in Table 2.

Sample Receipt

Chain of custody documentation were reviewed for completeness of information relevant to the samples and requested analysis. Sample IDs and sample collection dates from the chain of custody records were matched to the reported data.

All coolers were received within 4 ± 2°C.

ORGANIC ANALYSES

Holding Time and Sample Preservation

All samples were extracted and analyzed within holding times.

GC/MS Instrument Performance Check – Acceptable

Initial Calibration and Continuing Calibration Verifications – Acceptable

Blanks – Acceptable

Method Blank: Acceptable. No data were qualified based on the method blank results.

[1] NewFields. (2015). Portland Harbor Supplemental Sediment Study, Portland Oregon Quality Assurance Project Plan (QAPP). October 14, 2015.
[2] AECOM and Geosyntec. 2018. Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site, Quality Assurance Project Plan. March 23, 2018,

Rinsate Blank: Three rinsate blanks were collected on October 23, 2015, October 26, and October 27, 2015 (PH15-03-RB, PH15-04-RB, PH15-05-RB, respectively [ETR 1510012 and ETR 150019]) and are associated with the samples in this ETR.

- PH15-03-RB is associated with: PH15-40-A, PH15-40-B, PH15-40-C, PH15-40-D, PH15-50-A, PH15-50-C, PH15-50-D, PH15-28-A, and PH15-10-A.
- PH15-04-RB is associated with: PH15-31-A, PH15-43-A, PH15-11-A, PH15-11-D, and PH15-11-D-FD.
- PH15-05-RB is associated with: PH15-34-A.

Detections of target compounds in rinsate blanks were evaluated relative to sediment method detection limits (MDL). No target analytes were found in rinsate blanks at relative concentrations at, or above, the sediment MDL. No data were qualified based on the rinsate blank results.

Surrogate Spikes – Acceptable.

The surrogates listed below had recoveries outside the QC acceptance criteria, however the surrogate outlier was within criteria on the confirmation column. Therefore no data were qualified based on the surrogate recovery results.

Sample ID	Surrogate Compound	Recovery (%)	QC Limit (%)
PH15-11-A	Decachlorobiphenyl	36300	30 – 150
PH15-11-D	Decachlorobiphenyl	10700	30 – 150
PH15-11-D-FD	Decachlorobiphenyl	1710	30 – 150
PH15-28-A	Decachlorobiphenyl	636	30 – 150
PH15-31-A	Decachlorobiphenyl	3210	30 – 150
PH15-40-A	Decachlorobiphenyl	2090	30 – 150
PH15-40-B	Decachlorobiphenyl	612	30 – 150
PH15-50-A	Decachlorobiphenyl	2890	30 – 150

Internal Standard Areas – Acceptable.

Laboratory Control Samples – Acceptable.

Matrix Spike/Spike Duplicate – Acceptable.

Standard Reference Material – Acceptable except as noted below:

The following percent recoveries were outside QC acceptance limits:

Compound	Recovery (%)	QC Limit (%)
CL4-BZ#56	172	40 – 140
CL8-BZ#201	178	40 – 140

No data were qualified based on the SRM recovery results.

Field Duplicate– Acceptable except as noted below:

A field duplicate was submitted for PH15-11-D and was identified as PH15-11-D-FD. The results for the field duplicates were comparable except as noted below.

Sample ID	Field Duplicate ID	Analyte	RPD (%)	QC Limit (%)
PH15-11-D	PH15-11-D-FD	CL5-BZ#110	59	50
		Pentachlorobiphenyls	103	50
		Hexachlorobiphenyls	104	50
		Heptachlorobiphenyls	102	50
		Total PCB	110	50

The results for the analytes listed above were qualified as estimated and flagged "J" based on elevated field duplicates.

Laboratory Duplicate– Acceptable except as noted below:

Sample ID	Analyte	RPD (%)	QC Limit (%)
PH15-31-A	4,4'-DDD	59	30
	2,4'-DDT	75	30
	Pentachlorobiphenyls	73	30
	Hexachlorobiphenyls	37	30
	Total PCB	96	30

The results for the analytes listed above were qualified as estimated and flagged "J" based on elevated laboratory duplicates.

Target Compound Identifications– Acceptable.

DDT/Endrin Breakdown – No DDT/Endrin % breakdown issues were noted during data validation.

Dual Column Confirmation – Acceptable.

Compound Quantitation and CRQLs – Acceptable.

CONVENTIONAL ANALYSES

Field Duplicate– Acceptable.

Laboratory Duplicate– Acceptable.

OVERALL ASSESSMENT OF DATA

The data reported in this laboratory ETR is considered usable for meeting the project objectives.

The completeness is calculated by the number of usable data points divided by the total number of data points generated, multiplied by 100. The completeness for the laboratory ETR is 100%.

Validation performed by and Date:

George Desreuisseau, Mike Mitchel and Kerylynn Krahforst, December 2018.

Staff Scientists - NewFields

Table 1. QA/QC Summary Review

Sdg	SoilSampID	Lab_ID	AnalMeth	Analyte	Result	Lab_Flag	Units	NFG Result	NFG Qualifier	validator_ reason_code
L1527785	PH15-11-D	L1527785-04	8270D-SIM/680(M)	Heptachlorobiphenyls	0.314		ug/kg		J	fd
L1527785	PH15-11-D	L1527785-04	8270D-SIM/680(M)	Hexachlorobiphenyls	0.602		ug/kg		J	fd
L1527785	PH15-11-D	L1527785-04	8270D-SIM/680(M)	Pentachlorobiphenyls	0.422		ug/kg		J	fd
L1527785	PH15-11-D	L1527785-04	8270D-SIM/680(M)	Total Pcb	1.34		ug/kg		J	fd
L1527785	PH15-11-D	L1527785-04	8270D-SIM/680(M)	Cl5-BZ#110	0.138		ug/kg		J	fd
L1527785	PH15-11-D-FD	L1527785-05	8270D-SIM/680(M)	Total Pcb	4.63		ug/kg		J	fd
L1527785	PH15-11-D-FD	L1527785-05	8270D-SIM/680(M)	Cl5-BZ#110	0.254		ug/kg		J	fd
L1527785	PH15-11-D-FD	L1527785-05	8270D-SIM/680(M)	Heptachlorobiphenyls	0.974		ug/kg		J	fd
L1527785	PH15-11-D-FD	L1527785-05	8270D-SIM/680(M)	Hexachlorobiphenyls	1.91		ug/kg		J	fd
L1527785	PH15-11-D-FD	L1527785-05	8270D-SIM/680(M)	Pentachlorobiphenyls	1.33		ug/kg		J	fd
L1527785	PH15-31-A	L1527785-07	8270D-SIM/680(M)	Hexachlorobiphenyls	0.206		ug/kg		J	ld
L1527785	PH15-31-A	L1527785-07	8270D-SIM/680(M)	Pentachlorobiphenyls	0.152		ug/kg		J	ld
L1527785	PH15-31-A	L1527785-07	8270D-SIM/680(M)	Total Pcb	0.501		ug/kg		J	ld
L1527785	PH15-31-A-DUP	WG836768-4	8270D-SIM/680(M)	Total Pcb	1.43	Q	ug/kg		J	ld
L1527785	PH15-31-A-DUP	WG836768-4	8270D-SIM/680(M)	Hexachlorobiphenyls	0.298	Q	ug/kg		J	ld
L1527785	PH15-31-A-DUP	WG836768-4	8270D-SIM/680(M)	Pentachlorobiphenyls	0.326	Q	ug/kg		J	ld
L1527785	PH15-31-A	L1527785-07	EPA 8081B	4,4'-DDD	0.91	IPD	ug/kg		J	ld
L1527785	PH15-31-A	L1527785-07	EPA 8081B	2,4'-DDT	0.485	D	ug/kg		J	ld
L1527785	PH15-31-A-DUP	WG836766-4	EPA 8081B	2,4'-DDT	0.22	QD	ug/kg		J	ld
L1527785	PH15-31-A-DUP	WG836766-4	EPA 8081B	4,4'-DDD	1.67	IPQD	ug/kg		J	ld

Table 2. Reason Codes and Explanations

Reason Code	Explanation
bf	Field blank contamination
bl	Laboratory blank contamination
C	Calibration issue
el	Clean-up standard recovery
d	Reporting limit raised due to chromatographic interference
fd	Field duplicate RPDs
h	Holding Times
i	Internal standard areas
k	Estimated Maximum Possible Concentration (EMPC)
l	LCS or OPR recoveries
le	Labeled compound recovery
ld	Laboratory duplicate RPDs
lp	Laboratory control sample laboratory control sample duplicate RPDs
m	Matrix spike recovery
md	Matrix spike/matrix spike duplicate RPDs
nb	Negative laboratory blank contamination
p	Chemical preservation issue
r	Dual column RPD
q	Quantitation issue
s	Surrogate recovery
su	Ion suppression
t	Temperature preservation issue
x	Percent solids
y	Serial dilution results
z	ICS results