

Data Validation Report

Project:	Portland Harbor
Laboratory:	Alpha Analytical Laboratory
Environmental Test Record (ETR):	1510023
Analyses/Method:	Polycyclic Aromatic Hydrocarbons (PAH), and n-Alkanes and Total Petroleum Hydrocarbons (TPH), and Total Organic Carbon (TOC)

Summary

Eight sediment samples were collected in Portland Harbor, Oregon on October 21, 2015 and October 22, 2015. Samples were analyzed for polycyclic aromatic hydrocarbons (PAH) by EPA Method 8270D modified by selected ion monitoring mode (SIM), n-alkanes and total petroleum hydrocarbons (TPH) by EPA Method 8015D, and total organic carbon (TOC) by EPA Method 9060A 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 ETRs 1510023.

Sample ID	Lab ID	Matrix
PH15-10-A	1510023-01	Sediment
PH15-11-A	1510023-02	Sediment
PH15-11-D	1510023-03	Sediment
PH15-11-D-FD	1510023-04	Sediment
PH15-29-A	1510023-05	Sediment
PH15-29-C	1510023-06	Sediment
PH15-38-A	1510023-07	Sediment
PH15-38-C	1510023-08	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
- Calculation checks
- Contract Required Quantitation Limit (CRQL)
- Field duplicate results
- Laboratory duplicate results
- 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. No discrepancies noted.

All coolers were received within $4 \pm 2^{\circ}\text{C}$.

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 except as noted below:

Method Blank: The method blank met the QC acceptance criteria for PAH. PAH were detected in the method blank below the reporting limit. The associated sample results were either non-detect or were greater than 10X the blank concentration.

The method blank met the QC acceptance criteria for n-alkanes and TPH. n-Alkanes were detected in the method blank below the reporting limit. However, with the exception of the analytes below, n-octacosane, the associated sample results were either non-detect or were greater than 10X the blank concentration. Samples containing the below listed analytes at concentrations below the reporting limit were qualified as not detected, and were flagged “U” at the reporting limit based on the method blank result.

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,



n-Alkanes and TPH Compounds	Result	Unit	Lab Qualifier
n-Nonane (C9)	0.00240	mg/Kg	J
n-Octadecane (C18)	0.0584	mg/Kg	CJ
n-Docosane (C22)	0.00140	mg/Kg	J
n-Tricosane (C23)	0.00280	mg/Kg	J
n-Pentacosane (C25)	0.0966	mg/Kg	CJ

Rinsate Blank: Two rinsate blanks were collected on October 23, 2015 and October 26, 2015 (PH15-03-RB, and PH15-04-RB, respectively [ETRs 1510012 and 1510019, respectively]) and are associated with the samples in this ETR.

- PH15-03-RB is associated with: PH15-10-A, PH15-38-A, and PH15-38-C.
- PH15-04-RB is associated with: PH15-11-A, PH15-11-D, PH15-11-D-FD, PH15-29-A, and PH15-29-C.

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.

Internal Standard Areas – Acceptable.

Laboratory Control Samples – Acceptable.

Matrix Spike/Spike Duplicate – Acceptable except as noted below:

The following percent recoveries were outside QC limits:

Sample ID	Analyte	MS (%)	MSD (%)	QC Limit (%)	RPD (%)	QC Limit (%)
PH15-29-C	Naphthalene	-273	-295	50 - 125	ok	30
	Acenaphthene	-98	-25	50 - 125	49	30
	Fluorene	-58	-26	50 - 125	ok	30

The results for analytes listed above in the native sample were qualified as estimated and flagged "J" based on these MS/MSD results.

The samples contained highly elevated PAHs indicative tar-derived residues. Heterogeneity of sample matrix expected and reconciles with QC exceedance. The precision and accuracy of the method was demonstrated by the results of the LCS/LCSD. In addition, a PAH standard reference material (SRM 1941b), was reported with this ETR and met the QC acceptance criteria. The results of the SRM demonstrate accuracy has been achieved for this ETR.

Standard Reference Material – Acceptable.

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	Analytes	RPD (%)	QC Limit (%)
PH15-11-D	C2-Benzo(b)thiophenes	71	50
	C3-Benzo(b)thiophenes	79	50
	C3-Naphthalenes	61	50
	C4-Naphthalenes	50	50
	Acenaphthene	87	50
	Fluorene	96	50
	C1-Fluorennes	74	50
	Dibenzothiophene	88	50
	C1-Dibenzothiophenes	52	50
	Carbazole	106	50
	4-Methyldibenzothiophene	50	50
	2/3-Methyldibenzothiophene	52	50
	3-Methylphenanthrene	52	50
	n-Pentadecane (C15)	96	50
	n-Hexadecane (C16)	108	50
	n-Nonadecane (C19)	64	50
	n-Hexatriacontane (C36)	66	50

The samples contained highly elevated PAHs indicative tar-derived residues. Heterogeneity of sample matrix expected and reconciles with QC exceedance. The results for the analytes exceeding the QC criteria were qualified as estimated and flagged "J" based on elevated field duplicates.

Laboratory Duplicate— Acceptable except as noted below:

Sample ID	Analytes	RPD (%)	QC Limit (%)
PH15-29-A	n-Pentadecane (C15)	42	30
	Pristane	110	30
	n-Tricosane (C23)	92	30
	n-Pentacosane (C25)	40	30
	n-Heptacosane (C27)	73	30
	n-Nonacosane (C29)	64	30
	n-Dotriacontane (C32)	93	30
	n-Tritriacontane (C33)	96	30
	n-Tetratriacontane (C34)	47	30
	n-Pentatriacontane (C35)	106	30
	n-Heptatriacontane (C37)	92	30
	Total Saturated Hydrocarbons	69	30
	Total Petroleum Hydrocarbons (C9-C44)	80	30

Ninety-seven percent (97%) of the PAH results for the laboratory duplicates exceeded the QC limit of 30%. The samples contained elevated PAHs indicative tar-derived residues. Heterogeneity of sample matrix expected and reconciles with QC exceedance. The precision of the method was demonstrated by



the results of the LCS/LCSD. The results for the analytes were qualified as estimated and flagged “J” based on elevated laboratory duplicates.

Target Compound Identifications– Acceptable.

Compound Quantitation and CRQLs – Acceptable.

CONVENTIONAL ANALYSES

Holding Time and Sample Preservation – Acceptable.

Initial Calibration and Continuing Calibration Verifications – Acceptable.

Blanks– Acceptable.

Matrix Spike– Acceptable except as noted below:

Sample ID	Analytes	MS (%)	QC Limit (%)
PH15-33-A	TOC	74	50 - 125

The TOC results in the native sample were qualified as estimated and flagged “J” based on these MS results.

Standard Reference Material – Acceptable

Field Duplicate– Acceptable except as noted below:

Sample ID	Analytes	RPD (%)	QC Limit (%)
PH15-11-D	TOC	124	50

The result for TOC was qualified as estimated and flagged “J” based on the laboratory duplicate results.

Laboratory Duplicate– Acceptable except as noted below:

Sample ID	Analytes	RPD (%)	QC Limit (%)
PH15-10-A	TOC	40	25

The result for TOC was qualified as estimated and flagged “J” based on the laboratory duplicate results.

Compound Quantitation and CRQLs – 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.

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Staff Scientists - NewFields

Sdg	SoilSamplD	Lab_ID	AnalMeth	Analyte		Result	Lab_Flag	Units	NFG	NFG	validator_
									Result	Qualifier	reason_code
1510023	PH15-11-D-FD	1510023-04	EPA 9060	Total Organic Carbon		0.931		%	J		fd

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