Data Validation Report

Project:	Portland Harbor
Laboratory:	Alpha Analytical Laboratory
Environmental Test Record (ETR):	1408042
Analyses/Method:	Polycyclic Aromatic Hydrocarbons (PAH), Petroleum Biomarkers, n-Alkanes and Total Petroleum Hydrocarbons (TPH), and Total Organic Carbon (TOC)

Summary

Sixteen sediment samples were collected in Portland Harbor, Oregon on August 19 and August 20, 2014. Samples were analyzed for polycyclic aromatic hydrocarbons (PAH) and petroleum biomarkers 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 ETR 1408042.

Client ID	Lab ID	Matrix
PH14-S19-Z	1408042-01	Sediment
PH14-S20-S	1408042-02	Sediment
PH14-S20-Z	1408042-03	Sediment
PH14-S22-S	1408042-04	Sediment
PH14-S22-Z	1408042-05	Sediment
PH14-S24-S	1408042-06	Sediment
PH14-S24-Z	1408042-07	Sediment
PH14-S25-S	1408042-08	Sediment
PH14-S25-Z	1408042-09	Sediment
PH14-S26-S	1408042-10	Sediment
PH14-S32-S	1408042-11	Sediment
PH14-T04-S	1408042-13	Sediment
PH14-T04-Z	1408042-14	Sediment
PH14-T04-Z-D	1408042-15	Sediment
PH14-T03-S	1408042-16	Sediment
PH14-T03-Z	1408042-17	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 Sediment Forensic Chemistry Study, Portland Harbor Oregon Quality Assurance Project Plan (QAPP),*[1] dated July 29, 2014, 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 ± 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

[1] NewFields. (2014). Portland Harbor Sediment Forensic Chemistry Study, Portland Harbor Oregon Quality Assurance Project Plan (QAPP). July 29, 2014.
[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,

Blanks– Acceptable except as noted below:

Method Blank: The method blank met the QC acceptance criteria for PAH and biomarkers. PAH were detected in the method blank below the reporting limit, however the associated sample results were either non-detect or were greater than ten times the blank concentration. Data qualification was not necessary.

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 n-decane and n-tetradecane, the associated sample results were either non-detect or were greater than ten times the blank concentration. Samples containing n-decane and n-tetradecane at concentrations below the reporting limit and less than ten times the blank result were qualified as not detected, and were flagged "U" at the reporting limit based on the method blank result.

Rinsate Blank: Two rinsate blanks were collected on August 19, 2014 and August 20, 2014 (PH14-RB2 and PH14-RB3, respectively [ETR 1408040]) and are associated with the samples in this ETR.

- PH14-RB2 is associated with: PH14-S19-Z, PH14-S20-S, PH14-S20-Z, PH14-S22-S, PH14-S22-Z, PH14-S22-Z, PH14-S24-S, PH14-S24-Z, PH14-S24-Z, PH14-S25-S, PH14-S25-S, PH14-S25-Z, PH14-S25-Z, PH14-S26-S, and PH14-S32-S.

- PH14-RB3 is associated with: PH14-T04-S, PH14-T04-Z, PH14-T04-Z-D, PH14-T03-S, and PH14-T03-Z.

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 (%)
PH14-S20-S	Benzo[j]fluoranthene/Benzo[k]fluoranthene	170	208	50 - 125	ok	30
	Indeno[1,2,3-cd]pyrene	ok	139	50 - 125	ok	30
	Anthanthrene	40	42	50 - 125	ok	30
	n-Octacosane (C28)	ok	134	50 - 125	ok	30
	n-Triacontane (C30)	ok	138	50 - 125	ok	30

The results for benzo[j]fluoranthene/benzo[k]fluoranthene, indeno[1,2,3-cd]pyrene, anthanthrene, n-octacosane, and n-triacontane in the native sample were qualified as estimated and flagged "J" based on these MS/MSD results.

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 PH14-T04-Z and was identified as PH14-T04-Z-D. The results for the field duplicates were comparable except as noted below

Sample ID	Field Duplicate ID	Analyte	RPD (%)	QC Limit (%)
PH14-T04-Z	PH14-T04-Z-D	n-Triacontane (C30)	84	50
		n-Tritriacontane (C33)	52	50
		n-Pentatriacontane (C35)	61	50
		n-Hexatriacontane (C36)	64	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	Analytes	RPD (%)	QC Limit (%)
PH14-S19-Z	cis/trans-Decalin	34	30
	Benzothiophene	51	30
	7H-Benzo(c)fluorene	33	30
	Benzo[b]fluoranthene	31	30
	Indeno[1,2,3-cd]pyrene	36	30
	Benzo[g,h,i]perylene	36	30
	Coronene	33	30

The relative percent difference (RPD) for sample PH14-S19-Z and its duplicate exceeded the limit (30%) for seven (7) compounds with reported concentrations that are greater than the CRQL in either the native or duplicate extract. The RPD's for the LCS/LCSD show that precision of the method was established.

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

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/Spike Duplicate – Acceptable.

Standard Reference Material – Acceptable.

Field Duplicate– Acceptable.

Laboratory Duplicate– Acceptable.

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.

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
1408042	PH14-S19-Z	1408042-01	EPA 8270D	Coronene	815		µg/Kg		J	ld
1408042	PH14-S19-Z	1408042-01	EPA 8270D	Indeno[1,2,3-cd]pyrene	1900		µg/Kg		J	ld
1408042	PH14-S19-Z	1408042-01	EPA 8270D	cis/trans-Decalin	41.3		µg/Kg		J	ld
1408042	PH14-S19-Z	1408042-01	EPA 8270D	Benzothiophene	83		µg/Kg		J	ld
1408042	PH14-S19-Z	1408042-01	EPA 8270D	Benzo[g,h,i]perylene	2500		µg/Kg		J	ld
1408042	PH14-S19-Z	1408042-01	EPA 8270D	Benzo[b]fluoranthene	2140		µg/Kg		J	ld
1408042	PH14-S19-Z	1408042-01	EPA 8270D	7H-Benzo(c)fluorene	328		µg/Kg		J	ld
1408042	PH14-S19-Z-DUP	1408042-01D	EPA 8270D	Benzothiophene	140	¤	µg/Kg		J	ld
1408042	PH14-S19-Z-DUP	1408042-01D	EPA 8270D	Indeno[1,2,3-cd]pyrene	2720	¤	µg/Kg		J	ld
1408042	PH14-S19-Z-DUP	1408042-01D	EPA 8270D	7H-Benzo(c)fluorene	459	¤	µg/Kg		J	ld
1408042	PH14-S19-Z-DUP	1408042-01D	EPA 8270D	cis/trans-Decalin	58.2	¤	µg/Kg		J	ld
1408042	PH14-S19-Z-DUP	1408042-01D	EPA 8270D	Benzo[g,h,i]perylene	3600	¤	µg/Kg		J	ld
1408042	PH14-S19-Z-DUP	1408042-01D	EPA 8270D	Benzo[b]fluoranthene	2940	¤	µg/Kg		J	ld
1408042	PH14-S19-Z-DUP	1408042-01D	EPA 8270D	Coronene	1130	¤	µg/Kg		J	ld
1408042	PH14-S20-S	1408042-02	EPA 8270D	Indeno[1,2,3-cd]pyrene	404		µg/Kg		J	m
1408042	PH14-S20-S	1408042-02	EPA 8270D	Benzo[j]fluoranthene/Benzo[k]fluoranthene	392		µg/Kg		J	m
1408042	PH14-S20-S	1408042-02	EPA 8270D	Anthanthrene	162		µg/Kg		J	m
1408042	PH14-S20-S	1408042-02	EPA 8015M	n-Decane (C10)	0.00983	JB	mg/Kg	0.179	U	bl
1408042	PH14-S20-Z	1408042-03	EPA 8015M	n-Decane (C10)	0.0183	JB	mg/Kg	0.38	U	bl
1408042	PH14-S24-S	1408042-06	EPA 8015M	n-Decane (C10)	0.0146	JB	mg/Kg	0.188	U	bl
1408042	PH14-S26-S	1408042-10	EPA 8015M	n-Tetradecane (C14)	0.00889	JB	mg/Kg	0.165	U	bl
1408042	PH14-S26-S	1408042-10	EPA 8015M	n-Decane (C10)	0.00691	JB	mg/Kg	0.165	U	bl
1408042	PH14-S32-S	1408042-11	EPA 8015M	n-Decane (C10)	0.00799	JB	mg/Kg	0.125	U	bl
1408042	PH14-T03-S	1408042-16	EPA 8015M	n-Decane (C10)	0.00856	JB	mg/Kg	0.168	U	bl
1408042	PH14-T03-Z	1408042-17	EPA 8015M	n-Decane (C10)	0.00781	JB	mg/Kg	0.12	U	bl
1408042	PH14-T04-S	1408042-13	EPA 8015M	n-Decane (C10)	0.00882	JB	mg/Kg	0.17	U	bl
1408042	PH14-T04-Z	1408042-14	EPA 8015M	n-Decane (C10)	0.00946	JB	mg/Kg	0.158	U	bl
1408042	PH14-T04-Z-D	1408042-15	EPA 8015M	n-Decane (C10)	0.00896	JB	mg/Kg	0.16	U	bl
1408042	PH14-T04-Z	1408042-14	EPA 8015M	n-Pentatriacontane (C35)	0.492		mg/Kg		J	fd
1408042	PH14-T04-Z	1408042-14	EPA 8015M	n-Tritriacontane (C33)	0.763		mg/Kg		J	fd
1408042	PH14-T04-Z	1408042-14	EPA 8015M	n-Triacontane (C30)	0.428		mg/Kg		J	fd
1408042	PH14-T04-Z	1408042-14	EPA 8015M	n-Hexatriacontane (C36)	0.503		mg/Kg		J	fd
1408042	PH14-T04-Z-D	1408042-15	EPA 8015M	n-Tritriacontane (C33)	0.449		mg/Kg		J	fd
1408042	PH14-T04-Z-D	1408042-15	EPA 8015M	n-Pentatriacontane (C35)	0.261		mg/Kg		J	fd
1408042	PH14-T04-Z-D	1408042-15	EPA 8015M	n-Hexatriacontane (C36)	0.26		mg/Kg		J	fd
1408042	PH14-T04-Z-D	1408042-15	EPA 8015M	n-Triacontane (C30)	0.174		mg/Kg		J	fd
1408042	PH14-S20-S	1408042-02	EPA 8015M	n-Octacosane (C28)	0.38		mg/Kg		J	m
1408042	PH14-S20-S	1408042-02	EPA 8015M	n-Triacontane (C30)	0.236		mg/Kg		J	m, md

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