Data Validation Report

Project:	Portland Harbor
Laboratory:	Alpha Analytical Laboratory
Environmental Test Record (ETR):	1408032
Analyses/Method:	Polycyclic Aromatic Hydrocarbons (PAH), Petroleum Biomarkers, n-Alkanes and Total Petroleum Hydrocarbons (TPH), and Total Organic Carbon (TOC)

Summary

Nine sediment samples were collected in Portland Harbor, Oregon on August 11, 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 samples are associated with the laboratory ETR 1408032.

Sample ID	Laboratory ID	Matrix
PH14-S34-S	1408032-01	Sediment
PH14-S35-S	1408032-02	Sediment
PH14-S35-D	1408032-03	Sediment
PH14-S36-S	1408032-04	Sediment
PH14-S37-S	1408032-05	Sediment
PH14-S38-S	1408032-06	Sediment
PH14-S31-S	1408032-07	Sediment
PH14-S33-S	1408032-09	Sediment
PH14-S27-S	1408032-11	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.

Blanks– Acceptable except as noted below:

Method Blank: The method blank met the QC acceptance criteria for PAH and biomarkers (EPA Method 8270D). PAH were detected in the method blank below the reporting limit. However, with the exception of the analytes below, the associated sample results were either non-detect or were greater than ten times the blank concentration. Samples containing the below listed analytes 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.

[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,

PAH and Biomarker Compounds	Result	Unit	Lab Qualifier
cis/trans-Decalin	0.140	µg/Kg	J
Dibenzo(a,i)pyrene	.0705	µg/Kg	J
Dibenzo(a,h)pyrene	.0718	µg/Kg	J
1-Methyldibenzothiophene	.0215	µg/Kg	J

The method blank met the QC acceptance criteria for n-alkanes and TPH (EPA Method 8015D). n-Alkanes were detected in the method blank below the reporting limit. However, with the exception of the analytes below, the associated sample results were either non-detect or were greater than ten times the blank concentration. Samples containing the below listed analytes 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.

n-Alkanes and TPH Compounds	Result	Unit	Lab Qualifier
n-Decane (C10)	0.0016	mg/Kg	J
n-Pentadecane (C15)	0.00207	mg/Kg	J
n-Nonadecane (C19)	0.000467	mg/Kg	J

Rinsate Blank: One rinsate blank (PH14-RB1) was collected on August 11, 2014 (ETR 1408033) and is associated with the sediment samples in this ETR. 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 except as noted below:

The surrogate recovery for benzo(a)pyrene-d12 in field sample PH14-36-S was below the acceptance criteria of 40-120%. The results for benzo(b)fluoranthene, Benzo(j/k)fluoranthene, benzo(a)fluoranthene, benzo(e)pyrene, benzo(a)pyrene, perylene, indeno(1,2,3-cd)pyrene, dibenz(ah)anthracene, and benzo(ghi)perylene were "J" qualified.

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-S38-S	Anthracene	47	49	50 - 125	ok	30
	Phenanthrene	142	ok	50 - 125	ok	30
	Pyrene	ok	47	50 - 125	ok	30
	Chrysene/Triphenylene	29	48	50 - 125	ok	30
	Benzo[a]pyrene	29	38	50 - 125	ok	30
	Anthanthrene	1	1	50 - 125	ok	30
	Dibenzo(a,l)pyrene/dibenz(b,k)fluoranthene	22	29	50 - 125	ok	30

Sample ID	Analyte	MS (%)	MSD (%)	QC Limit (%)	RPD (%)	QC Limit (%)
	Naphtho(2,3-a)pyrene	-1	0	50 - 125	ok	30
	Dibenzo(a,i)pyrene	23	22	50 - 125	ok	30
	Dibenzo(a,h)pyrene	0	0	50 - 125	ok	30

The results for anthracene, phenanthrene, pyrene, chrysene/triphenylene, benzo(a)pyrene, anthanthrene, dibenzo(a,l)pyrene/ dibenz(b,k)fluoranthene, naphtho(2,3-a)pyrene, dibenzo(a,i)pyrene, and dibenzo(a,h)pyrene in the native sample were qualified as estimated and flagged "J" based on these MS/MSD results.

The recovery of dibenzo[a,h]pyrene from the MS/MSD samples was 0%. The result for dibenzo[a,h]pyrene in the native sample (PH14-S38-S) was "R" qualified based on the 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-S35-S and was identified as PH14-S35-D. The results for the field duplicates were comparable except as noted below

Sample ID	Field Duplicate ID	Analyte	RPD (%)	QC Limit (%)
PH14-S35-S	PH14-S35-D	C1-Naphthalenes	58	50
		Benzo[a]pyrene	52	50
		Anthanthrene	130	50
		Naphtho(2,3-a)pyrene	78	50
		2-Methylnaphthalene	66	50
		17a(H),21b(H)-25-Norhopane	59	50

The results for the analytes listed above in the native sample 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-S35-S	n-Hexacosane (C26)	31	30
	n-Octacosane (C28)	60	30
	n-Nonacosane (C29)	33	30
	n-Triacontane (C30)	148	30
	n-Hentriacontane (C31)	54	30
	n-Dotriacontane (C32)	155	30
	n-Tritriacontane (C33)	100	30
	n-Tetratriacontane (C34)	77	30

Sample ID	Analytes	RPD (%)	QC Limit (%)
	n-Pentatriacontane (C35)	112	30
	n-Hexatriacontane (C36)	93	30
	Total Saturated Hydrocarbons	54	30

The relative percent difference (RPD) for sample PH14-S35-S and its duplicate exceeded the limit (30%) for eleven (11) compounds with reported concentrations that are greater than the CRQL in either the native or duplicate extract. Similar results were exhibited from the initial extraction (not reported). The RPD's for the LCS/LCSD showing 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 except as noted below:

Sample ID	Analyte	MS (%)	MSD (%)	QC Limit (%)	RPD (%)	QC Limit (%)
PH14-S27-S	TOC	70	ok	75 - 125	ok	25

The result for TOC in sample PH14-27-S was qualified as estimated and flagged "J" based on this MS/MSD results.

Standard Reference Material – Acceptable.

Field Duplicate– Acceptable

Laboratory Duplicate– Acceptable except as noted below:

Sample ID	Analytes	RPD (%)	QC Limit (%)
PH14-S27-S	TOC	33	25

The result for TOC in sample PH14-27-S was qualified as estimated and flagged "J" based on the MS/MSD 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 99.95%.

One analyte (dibenzo[a,h]pyrene) in one sample is "R" qualified. The recovery of dibenzo[a,h]pyrene from the MS/MSD samples was 0%. The result for dibenzo[a,h]pyrene in the native sample (PH14- S38- S) was "R" qualified based on the MS/MSD results.

Validation performed by and Date:

George Desreuisseau, Mike Mitchel and Kerylynn Krahforst, February 2019.

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
1408032	PH14-S27-S	1408032-11X	EPA 8270D	1-Methyldibenzothiophen	0.203	JB	µg/Kg	0.953	U	bl
1408032	PH14-S27-S	1408032-11X	EPA 8270D	cis/trans-Decalin	0.601	JB	µg/Kg	0.953	U	bl
1408032	PH14-S27-S	1408032-11X	EPA 8270D	Dibenzo(a,h)pyrene	0.162	JB	µg/Kg	0.953	U	bl
1408032	PH14-S27-S	1408032-11X	EPA 8270D	Dibenzo(a,i)pyrene	0.548	JB	µg/Kg	0.953	U	bl
1408032	PH14-S35-D	1408032-03X	EPA 8270D	Benzo[a]pyrene	116		µg/Kg		J	fd
1408032	PH14-S35-D	1408032-03X	EPA 8270D	C1-Naphthalenes	14.3		µg/Kg		J	fd
1408032	PH14-S35-D	1408032-03X	EPA 8270D	Naphtho(2,3-a)pyrene	15.4		µg/Kg		J	fd
1408032	PH14-S35-D	1408032-03X	EPA 8270D	Dibenzo(a,h)pyrene	1.74		µg/Kg		J	fd
1408032	PH14-S35-D	1408032-03X	EPA 8270D	2-Methylnaphthalene	15.3		µg/Kg		J	fd
1408032	PH14-S35-D	1408032-03X	EPA 8270D	17a(H),21b(H)-25-Norhopa	40.2		µg/Kg		J	fd
1408032	PH14-S35-D	1408032-03X	EPA 8270D	Anthanthrene	28.2		µg/Kg		J	fd
1408032	PH14-S35-D	1408032-03X	EPA 8270D	Dibenzo(a,i)pyrene	6.52		µg/Kg		J	fd
1408032	PH14-S35-S	1408032-02X	EPA 8270D	2-Methylnaphthalene	7.74		µg/Kg		J	fd
1408032	PH14-S35-S	1408032-02X	EPA 8270D	17a(H),21b(H)-25-Norhopa	21.9		µg/Kg		J	fd
1408032	PH14-S35-S	1408032-02X	EPA 8270D	Benzo[a]pyrene	68.3		µg/Kg		J	fd
1408032	PH14-S35-S	1408032-02X	EPA 8270D	C1-Naphthalenes	7.86		µg/Kg		J	fd
1408032	PH14-S35-S	1408032-02X	EPA 8270D	cis/trans-Decalin	1.21	JB	µg/Kg	1.36	U	bl
1408032	PH14-S35-S	1408032-02X	EPA 8270D	Dibenzo(a,h)pyrene	0	U	µg/Kg		J	fd
1408032	PH14-S35-S	1408032-02X	EPA 8270D	Dibenzo(a,i)pyrene	3		µg/Kg		J	fd
1408032	PH14-S35-S	1408032-02X	EPA 8270D	Naphtho(2,3-a)pyrene	6.77		µg/Kg		J	fd
1408032	PH14-S35-S	1408032-02X	EPA 8270D	Anthanthrene	6.01		µg/Kg		J	fd
1408032	PH14-S36-S	1408032-04X	EPA 8270D	Benzo[a]pyrene	35		µg/Kg		J	S
1408032	PH14-S36-S	1408032-04X	EPA 8270D	Perylene	42.6		µg/Kg		J	S
1408032	PH14-S36-S	1408032-04X	EPA 8270D	Indeno[1,2,3-cd]pyrene	58.9		µg/Kg		J	S
1408032	PH14-S36-S	1408032-04X	EPA 8270D	Dibenz[ah]anthracene/Dib	12.1		µg/Kg		J	S
1408032	PH14-S36-S	1408032-04X	EPA 8270D	Benzo[j]fluoranthene/Ben	55.4		µg/Kg		J	S
1408032	PH14-S36-S	1408032-04X	EPA 8270D	Benzo[b]fluoranthene	68.1		µg/Kg		J	S
1408032	PH14-S36-S	1408032-04X	EPA 8270D	Benzo[a]fluoranthene	9.31		µg/Kg		J	S
1408032	PH14-S36-S	1408032-04X	EPA 8270D	Benzo[g,h,i]perylene	78.3		µg/Kg		J	S
1408032	PH14-S36-S	1408032-04X	EPA 8270D	Benzo[e]pyrene	59.3		µg/Kg		J	S
1408032	PH14-S38-S	1408032-06X	EPA 8270D	Anthanthrene	4.11		µg/Kg		J	m
1408032	PH14-S38-S	1408032-06X	EPA 8270D	Phenanthrene	73.1		µg/Kg		J	m
1408032	PH14-S38-S	1408032-06X	EPA 8270D	Naphtho(2,3-a)pyrene	4.34		µg/Kg		J	m
1408032	PH14-S38-S	1408032-06X	EPA 8270D	Dibenzo(a,l)pyrene/dibenz	22		µg/Kg		J	m
1408032	PH14-S38-S	1408032-06X	EPA 8270D	Dibenzo(a,i)pyrene	1.45	J	µg/Kg		J	m
1408032	PH14-S38-S	1408032-06X	EPA 8270D	Dibenzo(a,h)pyrene	0	U	µg/Kg		R	m
1408032	PH14-S38-S	1408032-06X	EPA 8270D	Chrysene/Triphenylene	111		µg/Kg		J	m
1408032	PH14-S38-S	1408032-06X	EPA 8270D	Anthracene	17.5		µg/Kg		J	m
1408032	PH14-S38-S	1408032-06X	EPA 8270D	Pyrene	173		µg/Kg		J	m
1408032	PH14-S38-S	1408032-06X	EPA 8270D	Benzo[a]pyrene	46.4		µg/Kg		J	m
1408032	PH14-S27-S	1408032-11X	EPA 8015M	n-Decane (C10)	0.00562	JB	mg/Kg	0.0953	U	bl
1408032	PH14-S27-S	1408032-11X	EPA 8015M	n-Pentadecane (C15)	0.00238	JB	mg/Kg	0.0953	U	bl
1408032	PH14-S31-S	1408032-07X	EPA 8015M	n-Decane (C10)	0.0091	JB	mg/Kg	0.134	U	bl
1408032	PH14-S33-S	1408032-09X	EPA 8015M	n-Pentadecane (C15)	0.00986	JB	mg/Kg	0.107	U	bl
1408032	PH14-S33-S	1408032-09X	EPA 8015M	n-Decane (C10)	0.00546	JB	mg/Kg	0.107	U	bl
1408032	PH14-S34-S	1408032-01X	EPA 8015M	n-Decane (C10)	0.00675	JB	mg/Kg	0.125	U	bl
1408032	PH14-S34-S	1408032-01X	EPA 8015M	n-Pentadecane (C15)	0.0192	JB	mg/Kg	0.125	U	bl
1408032	PH14-S35-D	1408032-03X	EPA 8015M	n-Decane (C10)	0.00785	JB	mg/Kg	0.14	U	bl
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Hexatriacontane (C36)	0.482		mg/Kg		J	ld
1408032	PH14-S35-S	1408032-02X	EPA 8015M	Total Saturated Hydrocarb	15.8		mg/Kg		J	ld
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Tritriacontane (C33)	0.669		mg/Kg		J	ld
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Triacontane (C30)	0.167		mg/Kg		J	ld
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Tetratriacontane (C34)	0.935		mg/Kg		J	ld
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Pentatriacontane (C35)	0.453		mg/Kg		J	ld
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Nonacosane (C29)	3.46		mg/Kg		J	ld
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Hexacosane (C26)	0.228		mg/Kg		J	ld
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Hentriacontane (C31)	1.64		mg/Kg		J	ld
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Dotriacontane (C32)	0.186		mg/Kg		J	ld
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Decane (C10)	0.00597	JB	mg/Kg	0.136	U	bl
1408032	PH14-S35-S	1408032-02X	EPA 8015M	n-Octacosane (C28)	0.444		mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	n-Nonacosane (C29)	4.81	¤	mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	Total Saturated Hydrocarb	27.6	¤	mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	n-Tritriacontane (C33)	2.02	¤	mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	n-Triacontane (C30)	1.12	¤	mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	n-Tetratriacontane (C34)	2.1	¤	mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	n-Octacosane (C28)	0.821	¤	mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	n-Hexatriacontane (C36)	1.32	¤	mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	n-Hexacosane (C26)	0.31	¤	mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	n-Hentriacontane (C31)	2.85	¤	mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	n-Dotriacontane (C32)	1.45	¤	mg/Kg		J	ld
1408032	PH14-S35-S-DUP	1408032-02XD	EPA 8015M	n-Pentatriacontane (C35)	1.6	¤	mg/Kg		J	ld
1408032	PH14-S36-S	1408032-04X	EPA 8015M	n-Decane (C10)	0.0097	JB	mg/Kg	0.156	U	bl

Sdg	SoilSampID	Lab_ID	AnalMeth	Analyte	Result	Lab_Flag	Units	NFG Result	NFG Qualifier	validator_ reason_code
1408032	PH14-S37-S	1408032-05X	EPA 8015M	n-Decane (C10)	0.0107	JB	mg/Kg	0.151	U	bl
1408032	PH14-S38-S	1408032-06X	EPA 8015M	n-Decane (C10)	0.00676	JB	mg/Kg	0.147	U	bl
1408032	PH14-S27-S	1408032-11	EPA 9060	Total Organic Carbon	1.28		%		J	m, ld
1408032	PH14-S27-S-DUP	1408032-11D	EPA 9060	Total Organic Carbon	0.921	¤	%		J	ld

Table 2. Reason Codes and Explanations

Reason Code	Explanation
be	Equipment blank contamination
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)
I	LCS or OPR recoveries
Ie	Labeled compound recovery
Id	Laboratory duplicate RPDs
Ip	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