Data Validation Report

Project:	Portland Harbor
Laboratory:	Alpha Analytical Laboratory
Environmental Test Record (ETR):	1408033
Analyses/Method:	Polycyclic Aromatic Hydrocarbons (PAH), petroleum biomarkers , and n-Alkanes and Total Petroleum Hydrocarbons (TPH)

Summary

One rinsate blank was collected in Portland Harbor, Oregon on August 11, 2014. The sample was analyzed for polycyclic aromatic hydrocarbons (PAH) and petroleum biomarkers by EPA Method 8270D modified by selected ion monitoring mode (SIM), and n-alkanes and total petroleum hydrocarbons (TPH) by EPA Method 8015D by Alpha Analytical Laboratory located in Mansfield, Massachusetts. The laboratory provided Level 4 data packages containing sample results and associated quality assurance (QA) and quality control (QC) data, preparation logs, and raw instrument output. The following water sample is associated with the laboratory ETR 1408033.

Sample ID	Lab ID	Matrix
PH14-RB1	1408033-01	Water

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

General: The rinsate blank (PH14-RB1) was extracted twice: the original sample was extracted within 7-days of sampling, the second extraction was outside the 14-day holding time. The original analysis was reviewed for data quality purposes.

Holding Time and Sample Preservation

The sample was extracted and analyzed within holding times with one exception. The PAH analysis was performed 45 days from extraction. The sample extract was properly preserved. Based on professional judgement (i.e., surrogate recoveries met QC criteria and proper extract storage), no data qualification was necessary.

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. With the exception of naphthalene, PAH were detected in the method blank below the reporting limit. Samples containing the below listed analytes were qualified as not detected, and were flagged "U" at the reporting limit based on the method blank result.

PAH Compounds	Result	Unit	Lab Qualifier
Naphthalene	299	ng/L	
C1-Naphthalenes	1.95	ng/L	J
Biphenyl	0.712	ng/L	J
Fluorene	0.826	ng/L	J
Phenanthrene	1.07	ng/L	J

[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 Compounds	Result	Unit	Lab Qualifier
Dibenzothiophene	0.547	ng/L	J
Fluoranthene	0.540	ng/L	J
Pyrene	0.402	ng/L	J
Cyclopenta(cd)pyrene	1.69	ng/L	J
Coronene	1.93	ng/L	J
2-Methylnaphthalene	1.44	ng/L	J
1-Methylnaphthalene	1.14	ng/L	J

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. With the exception of the analytes below, 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.

n-Alkanes and TPH Compounds	Result	Unit	Lab Qualifier
n-Nonane (C9)	0.00455	µg/L	J
n-Decane (C10)	0.00682	µg/L	J
n-Tetradecane (C14)	0.0170	µg/L	J
n-Pentadecane (C15)	0.0852	µg/L	J
n-Hexacosane (C26)	0.0125	µg/L	J
n-Heptacosane (C27)	0.0170	µg/L	J
n-Nonacosane (C29)	0.0114	µg/L	J
n-Triacontane (C30)	0.0125	µg/L	J
n-Hentriacontane (C31)	0.0125	µg/L	J
Total Saturated Hydrocarbons	0.1800	µg/L	J

Surrogate Spikes – Acceptable.

Internal Standard Areas – Acceptable.

Laboratory Control Samples – Acceptable.

Target Compound Identifications– 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, January 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
1408033	PH14-RB1	1408033-01	EPA 8270D	2-Methylnaphthalene	0.00601	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	1-Methylnaphthalene	0.0047	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	Pyrene	0.000607	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	Biphenyl	0.00306	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	C1-Naphthalenes	0.00982	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	Coronene	0.00151	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	Cyclopenta(cd)pyrene	0.00278	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	Dibenzothiophene	0.000986	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	Fluoranthene	0.00118	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	Fluorene	0.00627	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	Naphthalene	0.251	B	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8270D	Phenanthrene	0.0063	JB	ug/L	0.0114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8015M	n-Hentriacontane (C31)	0.000025	JB	mg/L	0.00114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8015M	n-Heptacosane (C27)	0.0000864	JB	mg/L	0.00114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8015M	n-Hexacosane (C26)	0.0000386	JB	mg/L	0.00114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8015M	n-Nonacosane (C29)	0.0000398	JB	mg/L	0.00114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8015M	n-Nonane (C9)	0.000025	JB	mg/L	0.00114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8015M	n-Pentadecane (C15)	0.0000852	JB	mg/L	0.00114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8015M	n-Tetradecane (C14)	0.0000227	JB	mg/L	0.00114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8015M	n-Triacontane (C30)	0.0000364	JB	mg/L	0.00114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8015M	n-Decane (C10)	0.00000568	JB	mg/L	0.00114	U	bl
1408033	PH14-RB1	1408033-01	EPA 8015M	Total Saturated Hydrocarbons	0.000958	JB	mg/L	0.00114	U	bl

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