

Data Validation Report

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| Project: | Portland Harbor |
| Laboratory: | Alpha Analytical Laboratory |
| Environmental Test Record (ETR): | 1408041 |
| Analyses/Method: | Polycyclic Aromatic Hydrocarbons (PAH), Petroleum Biomarkers, n-Alkanes and Total Petroleum Hydrocarbons (TPH), and Total Organic Carbon (TOC) |

Summary

Eighteen sediment samples were collected in Portland Harbor, Oregon on August 18, 2014 and August 19, 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 and 1408041.

| Sample ID | Lab ID | Matrix |
|--------------|------------|----------|
| PH14-S03-S | 1408041-01 | Sediment |
| PH14-S03-Z | 1408041-02 | Sediment |
| PH14-S05-S | 1408041-03 | Sediment |
| PH14-S05-Z | 1408041-04 | Sediment |
| PH14-S06-S | 1408041-05 | Sediment |
| PH14-S06-Z | 1408041-06 | Sediment |
| PH14-S10-S | 1408041-07 | Sediment |
| PH14-S10-Z | 1408041-08 | Sediment |
| PH14-S08-S | 1408041-09 | Sediment |
| PH14-S08-Z | 1408041-10 | Sediment |
| PH14-S12-S | 1408041-11 | Sediment |
| PH14-S12-Z | 1408041-12 | Sediment |
| PH14-S13-S | 1408041-13 | Sediment |
| PH14-S13-Z | 1408041-14 | Sediment |
| PH14-S17-S | 1408041-15 | Sediment |
| PH14-S17-Z | 1408041-16 | Sediment |
| PH14-S17-Z-D | 1408041-17 | Sediment |
| PH14-S19-S | 1408041-18 | 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 $4 \pm 2^\circ\text{C}$.

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:

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,



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. All samples were either non-detect or greater than ten times the blank concentration. No samples were qualified based on the method blank results.

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 n-decane, the associated sample results were either non-detect or greater than ten times the blank concentration. Samples containing n-decane 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: One rinsate blank (PH14-RB2) was collected on August 19, 2014 (ETR 1408040) 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.

Internal Standard Areas – Acceptable.

Laboratory Control Samples – Acceptable except as noted below:

| Analytes | LCS (%) | LCSD (%) | QC Limit (%) | RPD (%) | QC Limit (%) |
|---------------|---------|----------|--------------|---------|--------------|
| n-Nonane (C9) | 43 | 48 | 50 - 125 | ok | 30 |

The results for n-nonane were qualified as estimated and “J” flagged on the low LCS/LCSD recoveries.

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-S03-Z | n-Nonane (C9) | 33 | 39 | 50 - 125 | ok | 30 |
| | n-Decane (C10) | 44 | 47 | 50 - 125 | ok | 30 |

The results for n-nonane and n-decane were qualified as estimated and flagged “J” based on these MS/MSD results.

Standard Reference Material – Acceptable.

Field Duplicate– Acceptable except as noted below:

A field duplicate was submitted for PH14-S17-Z and was identified as PH14-S17-Z-D. The results for the field duplicates were comparable except as noted below:

| Sample ID | Field Duplicate ID | Analyte | RPD (%) | QC Limit (%) |
|------------|--------------------|----------------------------------|---------|--------------|
| PH14-S17-Z | PH14-S17-Z-D | 2,6,10 Trimethyltridecane (1470) | 63 | 50 |



| | | | | |
|--|--|-------------------------|----|----|
| | | n-Dotriacontane (C32) | 89 | 50 |
| | | n-Hexatriacontane (C36) | 71 | 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-S03-S | C1-Decalins | 41 | 30 |
| | C2-Decalins | 40 | 30 |
| | Dibenzofuran | 33 | 30 |
| | C4-Phenanthrenes/Anthracenes | 57 | 30 |
| | Retene | 90 | 30 |

The relative percent difference (RPD) for sample PH14-S03-S and its duplicate exceeded the limit (30%) for five (5) compounds with reported concentrations that are greater than the QL 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 except as noted below:

| Sample ID | Analyte | MS (%) | QC Limit (%) |
|------------|---------|--------|--------------|
| PH14-S08-S | TOC | 185 | 75 - 125 |

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

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 |
|---------|----------------|-------------|-----------|----------------------------------|----------|----------|-------|------------|---------------|-----------------------|
| 1408041 | PH14-S03-S | 1408041-01 | EPA 8270D | C1-Decalins | 4.47 | | µg/Kg | J | | ld |
| 1408041 | PH14-S03-S | 1408041-01 | EPA 8270D | Retene | 294 | | µg/Kg | J | | ld |
| 1408041 | PH14-S03-S | 1408041-01 | EPA 8270D | Dibenzofuran | 12 | | µg/Kg | J | | ld |
| 1408041 | PH14-S03-S | 1408041-01 | EPA 8270D | C4-Phenanthrenes/Anthracenes | 112 | | µg/Kg | J | | ld |
| 1408041 | PH14-S03-S | 1408041-01 | EPA 8270D | C2-Decalins | 13.4 | | µg/Kg | J | | ld |
| 1408041 | PH14-S03-S-DUP | 1408041-01D | EPA 8270D | C1-Decalins | 2.97 | ¤ | µg/Kg | J | | ld |
| 1408041 | PH14-S03-S-DUP | 1408041-01D | EPA 8270D | Dibenzofuran | 16.9 | ¤ | µg/Kg | J | | ld |
| 1408041 | PH14-S03-S-DUP | 1408041-01D | EPA 8270D | Retene | 111 | ¤ | µg/Kg | J | | ld |
| 1408041 | PH14-S03-S-DUP | 1408041-01D | EPA 8270D | C2-Decalins | 8.96 | ¤ | µg/Kg | J | | ld |
| 1408041 | PH14-S03-S-DUP | 1408041-01D | EPA 8270D | C4-Phenanthrenes/Anthracenes | 62.5 | ¤ | µg/Kg | J | | ld |
| 1408041 | PH14-S03-S | 1408041-01 | EPA 8015M | n-Nonane (C9) | 0.00172 | J | mg/Kg | J | | l |
| 1408041 | PH14-S05-S | 1408041-03 | EPA 8015M | n-Nonane (C9) | 0.00109 | J | mg/Kg | J | | l |
| 1408041 | PH14-S05-Z | 1408041-04 | EPA 8015M | n-Nonane (C9) | 0.000836 | J | mg/Kg | J | | l |
| 1408041 | PH14-S06-S | 1408041-05 | EPA 8015M | n-Nonane (C9) | 0.000964 | J | mg/Kg | J | | l |
| 1408041 | PH14-S06-Z | 1408041-06 | EPA 8015M | n-Nonane (C9) | 0.0005 | J | mg/Kg | J | | l |
| 1408041 | PH14-S08-S | 1408041-09 | EPA 8015M | n-Nonane (C9) | 0.00118 | J | mg/Kg | J | | l |
| 1408041 | PH14-S08-Z | 1408041-10 | EPA 8015M | n-Nonane (C9) | 0.00153 | J | mg/Kg | J | | l |
| 1408041 | PH14-S10-S | 1408041-07 | EPA 8015M | n-Nonane (C9) | 0.0105 | J | mg/Kg | J | | l |
| 1408041 | PH14-S10-Z | 1408041-08 | EPA 8015M | n-Nonane (C9) | 0.0297 | J | mg/Kg | J | | l |
| 1408041 | PH14-S12-S | 1408041-11 | EPA 8015M | n-Nonane (C9) | 0.00056 | J | mg/Kg | J | | l |
| 1408041 | PH14-S12-Z | 1408041-12 | EPA 8015M | n-Nonane (C9) | 0.000749 | J | mg/Kg | J | | l |
| 1408041 | PH14-S13-S | 1408041-13 | EPA 8015M | n-Nonane (C9) | 0.000538 | J | mg/Kg | J | | l |
| 1408041 | PH14-S13-Z | 1408041-14 | EPA 8015M | n-Nonane (C9) | 0.000629 | J | mg/Kg | J | | l |
| 1408041 | PH14-S17-S | 1408041-15 | EPA 8015M | n-Nonane (C9) | 0.0016 | J | mg/Kg | J | | l |
| 1408041 | PH14-S17-Z | 1408041-16 | EPA 8015M | n-Nonane (C9) | 0.00219 | J | mg/Kg | J | | l |
| 1408041 | PH14-S17-Z-D | 1408041-17 | EPA 8015M | n-Nonane (C9) | 0.00245 | J | mg/Kg | J | | l |
| 1408041 | PH14-S19-S | 1408041-18 | EPA 8015M | n-Nonane (C9) | 0.00739 | J | mg/Kg | J | | l |
| 1408041 | PH14-S03-Z | 1408041-02 | EPA 8015M | n-Nonane (C9) | 0.000799 | J | mg/Kg | J | | l, m |
| 1408041 | PH14-S03-S | 1408041-01 | EPA 8015M | n-Decane (C10) | 0.00516 | JB | mg/Kg | 0.143 | U | bl |
| 1408041 | PH14-S05-S | 1408041-03 | EPA 8015M | n-Decane (C10) | 0.00397 | JB | mg/Kg | 0.137 | U | bl |
| 1408041 | PH14-S05-Z | 1408041-04 | EPA 8015M | n-Decane (C10) | 0.00176 | JB | mg/Kg | 0.0929 | U | bl |
| 1408041 | PH14-S06-S | 1408041-05 | EPA 8015M | n-Decane (C10) | 0.00537 | JB | mg/Kg | 0.138 | U | bl |
| 1408041 | PH14-S06-Z | 1408041-06 | EPA 8015M | n-Decane (C10) | 0.00125 | JB | mg/Kg | 0.0834 | U | bl |
| 1408041 | PH14-S12-S | 1408041-11 | EPA 8015M | n-Decane (C10) | 0.00314 | JB | mg/Kg | 0.112 | U | bl |
| 1408041 | PH14-S12-Z | 1408041-12 | EPA 8015M | n-Decane (C10) | 0.00267 | JB | mg/Kg | 0.107 | U | bl |
| 1408041 | PH14-S13-S | 1408041-13 | EPA 8015M | n-Decane (C10) | 0.00236 | JB | mg/Kg | 0.107 | U | bl |
| 1408041 | PH14-S13-Z | 1408041-14 | EPA 8015M | n-Decane (C10) | 0.00262 | JB | mg/Kg | 0.105 | U | bl |
| 1408041 | PH14-S03-Z | 1408041-02 | EPA 8015M | n-Decane (C10) | 0.00489 | JB | mg/Kg | 0.0999 | U J | bl, m |
| 1408041 | PH14-S17-Z | 1408041-16 | EPA 8015M | n-Dotriacontane (C32) | 0.384 | | mg/Kg | J | | fd |
| 1408041 | PH14-S17-Z | 1408041-16 | EPA 8015M | 2,6,10 Trimethyltridecane (1470) | 0.165 | | mg/Kg | J | | fd |
| 1408041 | PH14-S17-Z | 1408041-16 | EPA 8015M | n-Hexatriacontane (C36) | 0.377 | | mg/Kg | J | | fd |
| 1408041 | PH14-S17-Z-D | 1408041-17 | EPA 8015M | n-Hexatriacontane (C36) | 0.18 | | mg/Kg | J | | fd |
| 1408041 | PH14-S17-Z-D | 1408041-17 | EPA 8015M | n-Dotriacontane (C32) | 0.148 | | mg/Kg | J | | fd |
| 1408041 | PH14-S17-Z-D | 1408041-17 | EPA 8015M | 2,6,10 Trimethyltridecane (1470) | 0.315 | | mg/Kg | J | | fd |
| 1408041 | PH14-S08-S | 1408041-09 | EPA 9060 | Total Organic Carbon | 2.31 | | % | J | | m |

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 |