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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling	
Laboratory:	SGS-AXYS, Sydney, British Columbia, Canada	
Laboratory Group:	WG65521-PAH_2	
Analyses/Method:	Polycyclic Aromatic Hydrocarbons (PAHs) / AXYS Method MLA-021 Rev12 Ver. 05	
Validation Level:	Stage 4	
AECOM Project Number:	60566335.2.12	
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SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on August 20-25, 2018.

Sample ID	Matrix/Sample Type
PDI-RB-XD-180820	Equipment blank
PDI-WS-T01-1808	Surface Water
PDI-WS-T02-1808	Surface Water
PDI-WS-T03-1808	Surface Water
PDI-WS-T04-1808	Surface Water
PDI-WS-T05-1808	Surface Water
PDI-WS-T06-1808	Surface Water
PDI-WS-T07-1808	Surface Water

Data validation activities were conducted with reference to:

- *AXYS Laboratory SOP MLA-021 Rev.12 Ver. 05: Analytical Method for the Determination of Polycyclic Aromatic Hydrocarbons (PAHs), Alkylated PAHs and Alkanes,*
- *USEPA Contract Laboratory Program National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016),*
- *Quality Assurance Project Plan, Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling, Portland Harbor Superfund Site (March 2018),* and the
- laboratory quality control (QC) limits.

The National Functional Guidelines were modified to accommodate the non-CLP methodologies. In the absence of method-specific information, laboratory QC limits, project-specific requirements and/or AECOM professional judgment were used as appropriate.

REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

✓	Data completeness (chain-of-custody (COC)/sample integrity)
✓	Holding times and sample preservation
✓	Mass resolution/chromatographic resolution
✓	Initial calibration/continuing calibration verification
✗	Laboratory blanks/equipment blanks
NA	Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓	Ongoing precision and recovery (OPR) results
NA	Field duplicate results
✗	Labeled compound recoveries
✗	Sample results/reporting issues

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. An NA indicates that the parameter was not included as part of this data set or was not applicable to this validation and therefore not reviewed. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

The data appear valid as qualified and may be used for decision making purposes. Select data points were qualified as estimated due to nonconformances of certain QC criteria (see discussion below). Qualified sample results are presented in Table 1.

RESULTS

Data Completeness (COC)/Sample Integrity

The data package was reviewed and found to meet acceptance criteria for completeness:

- The COCs were reviewed for completeness of information relevant to the samples and requested analyses, and for signatures indicating transfer of sample custody.
- The laboratory sample login sheet(s) were reviewed for issues potentially affecting sample integrity, including the condition of sample containers upon receipt at the laboratory.
- Completeness of analyses was verified by comparing the reported results to the COC requests.

Holding Times and Sample Preservation

Sample preservation and preparation/analysis holding times were reviewed for conformance with method criteria. All method QC acceptance criteria were met.

Mass Resolution/Chromatographic Resolution

The data were reviewed to ensure that

- the perfluorotributylamine (PFTBA) unit mass resolution at m/z 69/70 and 219/220 is demonstrated by the presence of a resolved peak at m/z 70 and m/z 220;
- the separation between benzo(b)fluoranthene and benzo(k)fluoranthene must be $\leq$ 75% of the valley height for equal concentrations, and
- the separation between phenanthrene and anthracene must be $\leq$ 30% of the valley height for equal concentrations.

All method QC acceptance criteria were met.

Initial Calibration/Continuing Calibration Verification

The data were reviewed to ensure that

- the signal/noise (S/N) and ion abundance ratio method acceptance criteria were met (as summarized by the laboratory);
- the initial calibration percent relative standard deviation (%RSD) method acceptance criteria were met for the native and labeled compounds;
- and the calibration verification standard (VER) method acceptance criteria were met.

All method QC acceptance criteria were met.

Laboratory Blanks/Equipment Blanks

Method and equipment rinsate blank results are evaluated as to whether there are contaminants detected above the estimated detection limit (EDL). Target compounds were detected in the method blank and equipment blank associated with the samples in this data set.

Compounds detected in the laboratory method blank and the equipment blank are summarized in Attachment A in Tables A-1 and A-2, respectively. It should be noted that significant contamination was found in the equipment blank associated with the samples in this data set. Consequently, the sample data were qualified on the basis of the equipment blank contamination as well as the laboratory method blank contamination.

The NFG guidance stipulates that a conservative approach should be taken and the reporting of false negative results should be avoided. Therefore, in order to avoid the reporting of false negative results, professional judgment was used to qualify the data in the manner summarized below.

The data were first qualified for laboratory method blank contamination on the following basis. As allowed in the NFG, a blank action limit (BAL) was determined as five times the method blank result.

- When the sample results were < the method blank result, the sample result was qualified as nondetect (U) at the sample result.
- When the sample result was $\geq$ the method blank result but $\leq$ the BAL, the sample result was qualified as estimated and potentially biased high (J+).
- When the sample result was > the BAL, the sample result was not qualified.

Qualified sample results are summarized in Table 1.

The data were subsequently qualified for equipment blank contamination on the following basis. Again, as allowed in the NFG, a blank action limit (BAL) was determined as five times the equipment blank result.

- When the sample result was ≤ the BAL, the sample result was qualified as estimated and potentially biased high (J+).
- When the sample result was > the BAL, the sample result was not qualified.

Qualified sample results are summarized in Table 1.

MS/MSD Results

MS/MSD analyses were not performed on a sample in this data set. No data validation actions were taken on this basis.

OPR Results

The OPR percent recoveries (%Rs) were reviewed for conformance with the method QC acceptance criteria. All method QC acceptance criteria were met.

Field Duplicate Results

A field duplicate pair was not submitted with this data set. No data validation actions were taken on this basis.

Labeled Compound Recoveries

The labeled compound %Rs were reviewed for conformance with the QC acceptance criteria. All method QC acceptance criteria were met.

The laboratory spikes the XAD resin with anthracene-d_{10} prior to deployment to the field. The laboratory established QC acceptance limits of 70-130% were met with one exception. The %R for anthracene-d_{10} in sample PDI-WS-T02-1808 (37.1%) fell below the QC acceptance limits. The positive PAH results in this sample were qualified as estimated (J).

Sample Results/Reporting Issues

All sample results detected at concentrations less than the lowest calibration standard but greater than the EDL are qualified by the laboratory as estimated (J). This "J" qualifier is retained during data validation.

Compound Identification

The data were reviewed to ensure that

- the absolute retention time, ion abundance ratios, SIM ion co-maximization, and S/N method acceptance criteria were met for compound identification.

Samples were qualified as follows:

Actions: (Based on NFG 2016 and AECOM professional judgment)

Criteria	Actions
A native target compound was reported by the laboratory as an EMPC.	Report result as an EMPC and qualify as estimated and presumptively present (JN).
A labeled compound was flagged by the laboratory indicating all identification criteria were not met.	Qualify associated positive and nondetect results as estimated (J/UJ).

It should be noted that in instances of multiple nonconformances, the bias is considered indeterminate in cases where a conflicting low and high bias exists or when a result does not exhibit a consistent bias. These results have an overall qualification of estimated (J) with the exception noted below.

When applicable, the "JN" qualifier was retained rather than replacement with the conventional overall "J" qualifier in instances where EMPC results were qualified for multiple quality control nonconformances.

Qualified sample results are shown in Table 1.

Compound Quantitation

The naphthalene result in all samples was qualified with the laboratory qualifier "MAX" and is defined as an estimated maximum value. The XAD resin is known to degrade to naphthalene over time; therefore, the naphthalene concentration reported for the sample may be affected if degradation of the XAD resin had occurred. All results flagged as "MAX" by the laboratory have been qualified as estimated and potentially biased high (J+).

It should be noted that a distinction between the term "EMPC" as noted above in the compound identification discussion and the term "MAX" as previously discussed must be made. The use of the term "EMPC" as noted in the compound identification section is used to indicate that not all identification criteria have been met, yet a concentration was reported for the affected compound. This conservative approach to report these results is taken due to the potential toxicity of these compounds. The laboratory uses a laboratory qualifier of "K" to indicate the occurrence of this "EMPC". In the case of the "MAX" designation, all identification criteria have been met, but results may be elevated due to the fact that degradation of the resin may contribute to the sample concentration; therefore, the laboratory considers these results to be an estimated maximum amount due to this potential contribution.

Verification of calculations was performed on a subset of the data as deemed appropriate. No discrepancies were noted.

QUALIFICATION ACTIONS

Sample results qualified as a result of validation actions are summarized in Table 1. All actions are described above.

ATTACHMENTS

Attachment A: Nonconformance Summary Tables

Attachment B: Qualifier Codes and Explanations

Attachment C: Reason Codes and Explanations

Table 1 - Data Validation Summary of Qualified Data

Sample ID	Matrix	Compound	Result	EDL	Units	Validation Qualifiers	Validation Reason
PDI-RB-XD-180820	WQ	Chrysene	2.83	0.953	ng/sample	J+	bl
PDI-RB-XD-180820	WQ	Naphthalene	831000	139	ng/sample	J+	q
PDI-WS-T01-1808	WS	Benz(a)anthracene	64.7	0.908	ng/sample	JN	k
PDI-WS-T01-1808	WS	Indeno(1,2,3-cd)pyrene	6.73	4.26	ng/sample	JN	k
PDI-WS-T01-1808	WS	Naphthalene	2260	3.71	ng/sample	J+	be,q
PDI-WS-T02-1808	WS	Benz(a)anthracene	93.6	1.07	ng/sample	JN	fs,k
PDI-WS-T02-1808	WS	Benzo(a)pyrene	15.1	2.65	ng/sample	J	fs
PDI-WS-T02-1808	WS	Benzo(b)fluoranthene	21.7	1.87	ng/sample	J	fs
PDI-WS-T02-1808	WS	Benzo(g,h,i)perylene	8.36	1.64	ng/sample	J	fs
PDI-WS-T02-1808	WS	Benzo(j,k)fluoranthene	17.8	2.01	ng/sample	J	fs
PDI-WS-T02-1808	WS	Chrysene	150	1.07	ng/sample	J	fs
PDI-WS-T02-1808	WS	Dibenz(a,h)anthracene	2.92	2.01	ng/sample	JN	fs,k
PDI-WS-T02-1808	WS	Indeno(1,2,3-cd)pyrene	8.67	1.97	ng/sample	JN	fs,k
PDI-WS-T02-1808	WS	Naphthalene	1180	5.03	ng/sample	J	be,fs,q
PDI-WS-T03-1808	WS	Benz(a)anthracene	157	2.14	ng/sample	JN	k
PDI-WS-T03-1808	WS	Benzo(g,h,i)perylene	9.52	1.55	ng/sample	JN	k
PDI-WS-T03-1808	WS	Indeno(1,2,3-cd)pyrene	7.92	2.01	ng/sample	JN	k
PDI-WS-T03-1808	WS	Naphthalene	1910	7.36	ng/sample	J+	be,q
PDI-WS-T04-1808	WS	Benz(a)anthracene	95.7	2.39	ng/sample	JN	k
PDI-WS-T04-1808	WS	Benzo(g,h,i)perylene	7.05	1.83	ng/sample	JN	k
PDI-WS-T04-1808	WS	Dibenz(a,h)anthracene	3.21	1.76	ng/sample	JN	k
PDI-WS-T04-1808	WS	Indeno(1,2,3-cd)pyrene	7.19	2.21	ng/sample	JN	k
PDI-WS-T04-1808	WS	Naphthalene	1980	7.29	ng/sample	J+	be,q
PDI-WS-T05-1808	WS	Benz(a)anthracene	47.0	0.775	ng/sample	JN	k
PDI-WS-T05-1808	WS	Benzo(a)pyrene	3.52	2.13	ng/sample	JN	k
PDI-WS-T05-1808	WS	Naphthalene	1850	6.92	ng/sample	J+	be,q
PDI-WS-T06-1808	WS	Benz(a)anthracene	18.6	1.20	ng/sample	JN	k
PDI-WS-T06-1808	WS	Benzo(a)pyrene	3.34	2.63	ng/sample	JN	k
PDI-WS-T06-1808	WS	Benzo(g,h,i)perylene	3.08	2.10	ng/sample	JN	k
PDI-WS-T06-1808	WS	Benzo(j,k)fluoranthene	2.44	1.96	ng/sample	JN	k
PDI-WS-T06-1808	WS	Naphthalene	1800	5.10	ng/sample	J+	be,q
PDI-WS-T07-1808	WS	Benz(a)anthracene	18.2	0.685	ng/sample	JN	k
PDI-WS-T07-1808	WS	Benzo(g,h,i)perylene	3.39	1.63	ng/sample	JN	k
PDI-WS-T07-1808	WS	Benzo(j,k)fluoranthene	2.99	1.70	ng/sample	JN	k
PDI-WS-T07-1808	WS	Dibenz(a,h)anthracene	2.04	0.994	ng/sample	JN	k
PDI-WS-T07-1808	WS	Indeno(1,2,3-cd)pyrene	2.54	1.98	ng/sample	JN	k
PDI-WS-T07-1808	WS	Naphthalene	2030	6.34	ng/sample	J+	be,q

Attachment A

Nonconformance Summary Tables

Table A-1 - Laboratory Blanks

Blank ID	Compound	Result	RL	BAL	Units	Associated Samples
WG65521-101	Naphthalene	44.2	5.98	221	ng/sample	PDI-RB-XD-180820
	Chrysene	1.04	0.983	5.20	ng/sample	PDI-WS-T01-1808 PDI-WS-T02-1808 PDI-WS-T03-1808 PDI-WS-T04-1808 PDI-WS-T05-1808 PDI-WS-T06-1808 PDI-WS-T07-1808

Table A-2 - Field Blanks

Blank ID	Compound	Result	RL	BAL	Units	Associated Samples
PDI-RB-XD-180820	Naphthalene	831000	139	4155000	ng/sample	PDI-WS-T01-1808
	Chrysene	2.83	0.953	14.2	ng/sample	PDI-WS-T02-1808 PDI-WS-T03-1808 PDI-WS-T04-1808 PDI-WS-T05-1808 PDI-WS-T06-1808 PDI-WS-T07-1808

Attachment B

Qualifier Codes and Explanations

Qualifier	Explanation
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample with a potential low bias.
J+	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample with a potential high bias.
JN	The analyte was tentatively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Attachment C

Reason Codes and Explanations

Reason Code	Explanation
be	Equipment blank contamination
bf	Field blank contamination
bl	Laboratory blank contamination
c	Calibration issue
cl	Clean-up standard recovery
d	Reporting limit raised due to chromatographic interference
fd	Field duplicate RPDs
fs	Field spike recovery
h	Holding times
i	Internal standard areas
k	Estimated Maximum Possible Concentration (EMPC)
l	LCS or OPR recoveries
lc	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