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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:	WG67276-PAH	
Analyses/Method:	Polycyclic Aromatic Hydrocarbons (PAHs) / AXYS Method MLA-021 (Rev12 Ver. 05)	
Validation Level:	Stage 2A	
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 January 26-27, 2019 and February 17-18, 2019.

Sample ID	Matrix/Sample Type
PDI-RB-XD-190127	Equipment Blank
PDI-WS-T01-1902	Surface Water
PDI-WS-T02-1902	Surface Water
PDI-WS-T03-1902	Surface Water
PDI-WS-T04-1902	Surface Water
PDI-WS-T05-1902	Surface Water
PDI-WS-T06-1901	Surface Water
PDI-WS-T07-1901	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
✗	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.

Laboratory Blanks/Equipment Blanks

Laboratory method blank 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 laboratory 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 with regards to naphthalene was found in the equipment blank associated with the samples in this data set. Consequently, the sample data for naphthalene 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 $\leq$ 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.

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 %Rs and/or RPDs were reviewed for conformance with the method QC acceptance criteria. All 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 for all samples with the following exception. The %R (185%) for anthracene-d_{10} in sample PDI-WS-T01-1902 exceeded the QC acceptance limits. Qualification of the data on this basis is not required.

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.

Estimated Maximum Possible Concentrations (EMPCs)

The data were reviewed to identify sample results that were indicated by the laboratory to be estimated maximum possible concentrations (EMPCs) because of identification criteria not being met.

The laboratory identified the presence of EMPCs for the samples in this data set by qualifying affected results with a “K” laboratory qualifier. Samples were qualified as follows:

Actions: (Based on 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 where there is a conflicting low and high bias 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 summarized 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.

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-190127	WQ	Chrysene	0.952	0.663	ng/sample	J+	bl
PDI-RB-XD-190127	WQ	Naphthalene	920	4.10	ng/sample	J+	q
PDI-WS-T01-1902	WS	Naphthalene	2050	6.23	ng/sample	J+	be,q
PDI-WS-T02-1902	WS	Benz(a)anthracene	8.05	0.767	ng/sample	JN	k
PDI-WS-T02-1902	WS	Naphthalene	1430	21.1	ng/sample	J+	be,q
PDI-WS-T03-1902	WS	Benzo(j,k)fluoranthene	3.75	2.58	ng/sample	JN	k
PDI-WS-T03-1902	WS	Naphthalene	1870	20.3	ng/sample	J+	be,q
PDI-WS-T04-1902	WS	Indeno(1,2,3-cd)pyrene	3.25	1.69	ng/sample	JN	k
PDI-WS-T04-1902	WS	Naphthalene	19100	53.0	ng/sample	J+	q
PDI-WS-T05-1902	WS	Naphthalene	1130	8.54	ng/sample	J+	be,q
PDI-WS-T06-1901	WS	Indeno(1,2,3-cd)pyrene	2.61	1.70	ng/sample	JN	k
PDI-WS-T06-1901	WS	Naphthalene	2120	3.70	ng/sample	J+	be,q
PDI-WS-T07-1901	WS	Naphthalene	1510	9.76	ng/sample	J+	be,q

Attachment A

Nonconformance Summary Tables

Table A-1 - Lab Blanks

Blank ID	Compound	Result	QL	BAL	Units	Associated Samples
WG67276-101	Chrysene	0.616	80.7	3.08	ng/sample	PDI-RB-XD-190127
	Naphthalene	27.1	79.2	135.5	ng/sample	PDI-WS-T01-1902 PDI-WS-T02-1902 PDI-WS-T03-1902 PDI-WS-T04-1902 PDI-WS-T05-1902 PDI-WS-T06-1901 PDI-WS-T07-1901

Table A-2 - Field Blanks

Blank ID	Compound	Result	QL	BAL	Units	Associated Samples
PDI-RB-XD-190127	Chrysene	0.952	80.9	4.76	ng/sample	PDI-WS-T01-1902
	Naphthalene	920	79.5	4600	ng/sample	PDI-WS-T02-1902 PDI-WS-T03-1902 PDI-WS-T04-1902 PDI-WS-T05-1902 PDI-WS-T06-1901 PDI-WS-T07-1901

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
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