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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling	
Laboratory:	Test America, Knoxville, Tennessee	
Service Request:	580-77234-3	
Analyses/Method:	Chlorinated Biphenyls by HRGC/HRMS / E1668A	
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 May 9, 2018.

Sample ID	Matrix/Sample Type
PDI-SG-B078-BL1	Sediment

Data validation activities were conducted with reference to:

- *EPA Method 1668A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS* (USEPA, August 2003),
- *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
✗ Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓ Ongoing precision and recovery results

NA	Field duplicate results
✓	Labeled compounds and labeled clean-up standard 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 or negated 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

Method 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 blanks associated with the samples in this data set.

An equipment blank was not analyzed within this sample delivery group (SDG).

The NFG guidance stipulates that a conservative approach should be taken with regards to qualification of PCB congeners due to the toxicity of these compounds 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 following manner. As allowed in the NFG, a blank action limit (BAL) was determined as 5 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 and $\leq$ the BAL, the sample result was qualified as estimated and potentially biased high (J+).

- When the sample result was > the BAL, sample result was not qualified.

Qualified sample results are summarized in Table 1.

MS/MSD Results

The MS/MSD percent recoveries (%Rs) and relative percent differences (RPDs) were reviewed for conformance with the QC acceptance criteria.

Nonconformances are summarized in Attachment A in Table A-1. Samples were qualified as follows:

Actions: (Based on AECOM professional judgment in the absence of NFG guidance)

Qualify results	MS/MSD %Rs			MS/MSD RPD
	<10% R*	10% R to Lower Limit	➢ Upper Limit	> QC Limit
Detected Results	J-	J-	J+	J
Non-Detected Results	R	UJ	Accept	Accept
*AECOM professional judgment used to establish a minimum criterion of 10% R				
Notes: Qualifications should be applied to the affected compound in the unspiked sample only unless all data appear to be impacted. If the sample result is > 4x the spike added concentration, no action is taken based on AECOM professional judgment.				

Qualified sample results are shown in Table 1.

Ongoing Precision and Recovery

The OPR %Rs and/or RPDs were reviewed for conformance with the method QC acceptance criteria. All method QC acceptance criteria were met.

Field Duplicate Results

A field duplicate was not submitted for this sample delivery group (SDG).

Labeled Compounds and Labeled Clean-up Standard Recoveries

The labeled compounds and labeled clean-up standard %Rs were reviewed for conformance with the QC acceptance criteria. No QC outliers were noted in the samples.

Sample Results/Reporting Issues

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

The laboratory qualified the sample results with a "q" to indicate that the ion abundance ratio was outside of the QC acceptance limits; the result should be considered as an Estimated Maximum Possible Concentration (EMPC). These results were qualified as estimated and tentatively identified (JN). Qualified sample results are summarized in Table 1.

It should be noted that the "JN" qualifier was retained rather than replacement with the conventional overall "J", “J+”, and “J-“ qualifiers in instances where sample results were qualified for multiple quality control nonconformances.

Percent Solids Content

The percent solids data were reviewed since the amount of moisture in a solid sample may have an impact on data representativeness. Due to the extremely low solubility of PCB congeners in water, these analytes should be contained in the solid phase. Consequently, the NFG guidance does not stipulate a percent solids criterion. If applicable, EPA Regional guidance is used when assessing percent solids content. In the absence of EPA Regional guidance, AECOM uses 30% solids (from the NFG semivolatile guidance) as a benchmark to evaluate the percent solids content and professional judgment is used to determine the necessity to qualify data. Data were not qualified on the basis of percent solids content.

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-SG-B078-BL1	SE	PCB-105	1.3	0.0055	ng/g	J-	m
PDI-SG-B078-BL1	SE	PCB-145	0.0035	0.000066	ng/g	JN	k
PDI-SG-B078-BL1	SE	PCB-148	0.0028	0.000088	ng/g	JN	k
PDI-SG-B078-BL1	SE	PCB-150	0.0044	0.000059	ng/g	JN	k
PDI-SG-B078-BL1	SE	PCB-152	0.0048	0.000064	ng/g	JN	k
PDI-SG-B078-BL1	SE	PCB-156	0.69	0.0035	ng/g	J-	m
PDI-SG-B078-BL1	SE	PCB-157	0.69	0.0035	ng/g	J-	m
PDI-SG-B078-BL1	SE	PCB-2	0.0056	0.00029	ng/g	JN	bl,k
PDI-SG-B078-BL1	SE	PCB-24	0.0037	0.00043	ng/g	JN	k
PDI-SG-B078-BL1	SE	PCB-3	0.011	0.00033	ng/g	J+	bl
PDI-SG-B078-BL1	SE	PCB-5	0.0027	0.00052	ng/g	JN	k
PDI-SG-B078-BL1	SE	PCB-54	0.0038	0.000035	ng/g	JN	k

Attachment A

Nonconformance Summary Tables

Table A-1 - MS/MSD Results

Sample ID	Compound	MS % Recovery	MSD % Recovery	Lower Limit	Upper Limit	RPD	RPD Limit
PDI-SG-B078-BL1	PCB-105	0	0	50	150	ok	50
	PCB-156	ok	47	50	150	ok	50
	PCB-157	ok	47	50	150	ok	50

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