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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling	
Laboratory:	Test America, Knoxville, Tennessee	
Service Request:	580-79946-3	
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 August 28, 2018.

Sample ID	Matrix/Sample Type
PDI-SG-B478	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),
- *EPA Method 1668B: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS* (USEPA, November 2008),
- *EPA Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS* (USEPA, April 2010),
- *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 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, 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 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 in this data set. An equipment blank was not submitted with the data package.

Detected compounds are summarized in Attachment A in Table A-1.

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 blank result:

- When the sample results were < the blank result, the sample result was qualified as nondetect (U) at the sample 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, 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.

Ongoing Precision and Recovery

The OPR %Rs 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 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. All method QC acceptance criteria were met.

Sample Results/Reporting Issues

During the Stage 4 data validation, it was discovered that the ion ratio QC limits entered into the laboratory's CHROM data system were incorrect for PCB-5 and PCB-159. Additionally, it was discovered that the CHROM data system did not always provide the area for one of the two ions when manually assigned by the analyst and this resulted in the ion ratio being reported as 0. A database query was performed by the laboratory to determine which results were impacted by these errors. The laboratory updated the CHROM data system to correct for these issues and affected samples were reprocessed. For samples analyzed after the discovery of these issues, all lab reports will indicate the correct QC limits for the ion ratios for PCB 5 and PCB 159. As an additional precaution, the laboratory continues to monitor the sample results in order to ensure all peak areas are being provided by the CHROM data system and the incidence of missing area results no longer exists.

It should be noted, that sample or standard results were not reprocessed for the following instances since the sample concentration or final reported result were not impacted.

- The pcb congener detected in a sample was determined to be found at a concentration that was less than the EDL. Consequently, the result is reported as not detected.
- The pcb congener was calculated and reported correctly in spite of the incorrect QC limit noted in the CHROM data system.

For the scenarios listed above, the ion ratio QC limits reported in the laboratory report will not reflect the corrected change to the CHROM data system.

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.

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" qualifier 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.

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-B478	SE	PCB-102	0.0016	0.00012	ng/g	JN	k
PDI-SG-B478	SE	PCB-11	0.015	0.00053	ng/g	J+	bl
PDI-SG-B478	SE	PCB-120	0.00093	0.000085	ng/g	JN	k
PDI-SG-B478	SE	PCB-123	0.0013	0.00044	ng/g	JN	k
PDI-SG-B478	SE	PCB-134	0.0038	0.00063	ng/g	JN	k
PDI-SG-B478	SE	PCB-137	0.0037	0.00054	ng/g	JN	k
PDI-SG-B478	SE	PCB-139	0.0016	0.00054	ng/g	JN	k
PDI-SG-B478	SE	PCB-140	0.0016	0.00054	ng/g	JN	k
PDI-SG-B478	SE	PCB-143	0.0038	0.00063	ng/g	JN	k
PDI-SG-B478	SE	PCB-144	0.0022	0.000035	ng/g	JN	k
PDI-SG-B478	SE	PCB-154	0.0016	0.000030	ng/g	JN	k
PDI-SG-B478	SE	PCB-158	0.0070	0.00038	ng/g	JN	k
PDI-SG-B478	SE	PCB-17	0.010	0.00012	ng/g	JN	k
PDI-SG-B478	SE	PCB-171	0.0048	0.00013	ng/g	JN	k
PDI-SG-B478	SE	PCB-173	0.0048	0.00013	ng/g	JN	k
PDI-SG-B478	SE	PCB-174	0.020	0.00012	ng/g	JN	k
PDI-SG-B478	SE	PCB-175	0.00057	0.00012	ng/g	JN	k
PDI-SG-B478	SE	PCB-176	0.0022	0.000090	ng/g	JN	k
PDI-SG-B478	SE	PCB-178	0.0045	0.00013	ng/g	JN	k
PDI-SG-B478	SE	PCB-19	0.0024	0.00015	ng/g	JN	k
PDI-SG-B478	SE	PCB-190	0.0042	0.000086	ng/g	J+	bl
PDI-SG-B478	SE	PCB-191	0.00066	0.000089	ng/g	JN	k
PDI-SG-B478	SE	PCB-195	0.0037	0.00027	ng/g	JN	k
PDI-SG-B478	SE	PCB-196	0.0030	0.000044	ng/g	JN	k
PDI-SG-B478	SE	PCB-197	0.00030	0.000034	ng/g	JN	k
PDI-SG-B478	SE	PCB-2	0.0018	0.00010	ng/g	JN	k
PDI-SG-B478	SE	PCB-3	0.00069	0.00011	ng/g	JN	k
PDI-SG-B478	SE	PCB-32	0.0067	0.000087	ng/g	JN	k
PDI-SG-B478	SE	PCB-37	0.0089	0.00025	ng/g	JN	k
PDI-SG-B478	SE	PCB-4	0.0051	0.00072	ng/g	JN	k
PDI-SG-B478	SE	PCB-45	0.0088	0.00071	ng/g	J+	bl
PDI-SG-B478	SE	PCB-48	0.0052	0.00067	ng/g	JN	k
PDI-SG-B478	SE	PCB-51	0.0088	0.00071	ng/g	J+	bl
PDI-SG-B478	SE	PCB-60	0.0076	0.00050	ng/g	JN	k
PDI-SG-B478	SE	PCB-67	0.0011	0.00043	ng/g	JN	k
PDI-SG-B478	SE	PCB-68	0.0022	0.00044	ng/g	J+	bl
PDI-SG-B478	SE	PCB-7	0.0011	0.00056	ng/g	JN	k
PDI-SG-B478	SE	PCB-77	0.0041	0.00047	ng/g	J+	bl

Sample ID	Matrix	Compound	Result	EDL	Units	Validation Qualifiers	Validation Reason
PDI-SG-B478	SE	PCB-82	0.0066	0.00014	ng/g	JN	k
PDI-SG-B478	SE	PCB-98	0.0016	0.00012	ng/g	JN	k

Attachment A

Nonconformance Summary Tables

Table A-1 - Laboratory Blanks

Blank ID	Compound	Result	ML	Units	BAL	Associated Samples
MB 140-23946/5-B	PCB-109	0.00273	0.00012	ng/g	0.01365	PDI-SG-B478
	PCB-11	0.00366	0.0034	ng/g	0.0183	
	PCB-110	0.00264	0.00010	ng/g	0.0132	
	PCB-115	0.00264	0.00010	ng/g	0.0132	
	PCB-118	0.00230	0.00022	ng/g	0.0115	
	PCB-119	0.00273	0.00012	ng/g	0.01365	
	PCB-125	0.00273	0.00012	ng/g	0.01365	
	PCB-126	0.00115	0.00023	ng/g	0.00575	
	PCB-147	0.00120	0.00067	ng/g	0.006	
	PCB-149	0.00120	0.00067	ng/g	0.006	
	PCB-156	0.00184	0.00054	ng/g	0.0092	
	PCB-157	0.00184	0.00054	ng/g	0.0092	
	PCB-169	0.00124	0.00033	ng/g	0.0062	
	PCB-189	0.00114	0.00016	ng/g	0.0057	
	PCB-190	0.00109	0.000082	ng/g	0.00545	
	PCB-198	0.00147	0.000069	ng/g	0.00735	
	PCB-199	0.00147	0.000069	ng/g	0.00735	
	PCB-20	0.00582	0.00068	ng/g	0.0291	
	PCB-28	0.00582	0.00068	ng/g	0.0291	
	PCB-40	0.00232	0.00044	ng/g	0.0116	
	PCB-41	0.00232	0.00044	ng/g	0.0116	
	PCB-44	0.00750	0.00039	ng/g	0.0375	
	PCB-45	0.00248	0.00046	ng/g	0.0124	
	PCB-47	0.00750	0.00039	ng/g	0.0375	
	PCB-51	0.00248	0.00046	ng/g	0.0124	
	PCB-52	0.00262	0.00044	ng/g	0.0131	
	PCB-60	0.000849	0.00033	ng/g	0.004245	
	PCB-61	0.00522	0.00031	ng/g	0.0261	
	PCB-64	0.00103	0.00029	ng/g	0.00515	
	PCB-65	0.00750	0.00039	ng/g	0.0375	
	PCB-66	0.00257	0.00031	ng/g	0.01285	
	PCB-68	0.00179	0.00029	ng/g	0.00895	
	PCB-70	0.00522	0.00031	ng/g	0.0261	
	PCB-71	0.00232	0.00044	ng/g	0.0116	
	PCB-74	0.00522	0.00031	ng/g	0.0261	

Blank ID	Compound	Result	ML	Units	BAL	Associated Samples
	PCB-76	0.00522	0.00031	ng/g	0.0261	
	PCB-77	0.000868	0.00031	ng/g	0.00434	
	PCB-86	0.00273	0.00012	ng/g	0.01365	
	PCB-87	0.00273	0.00012	ng/g	0.01365	
	PCB-95	0.00214	0.00015	ng/g	0.0107	
	PCB-97	0.00273	0.00012	ng/g	0.01365	

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