AECOM
6200 S. Quebec Street
Greenwood Village, CO 80111
303.694.2770 tel

Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling	
Laboratory:	Test America, Knoxville, Tennessee	
Service Request:	580-76952-3	
Analyses/Method:	Chlorinated Biphenyls by HRGC/HRMS / E1668A	
Validation Level:	Stage 2A	
AECOM Project Number:	60566335.2.12	
Prepared by:	Sheri Fling/AECOM	Completed on: 09/27/2018
Reviewed by:	Larry Brook/AECOM	File Name: 580-76952-3 DVR

SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on April 29, 2018.

Sample ID	Matrix/Sample Type
PDI-SG-S208	Sediment
PDI-SG-S216	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
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 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. The method QC acceptance criteria were met.

An extra container for each of the samples was provided to be archived frozen at the TestAmerica Sacramento laboratory pending potential additional analyses.

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 associated with the samples in this data set. An equipment rinsate blank was not submitted with this data set.

Method blank detected compounds are summarized in Attachment A, 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 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

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 (OPR)

The OPR percent recoveries (%Rs) and/or relative percent differences (RPDs) were reviewed for conformance with the method QC acceptance criteria. The method QC acceptance criteria were met.

Field Duplicate Results

A field duplicate was not submitted with this data set.

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. The method QC acceptance criteria were met.

It was noted in the case narrative that ion abundance ratios (IARs) were outside acceptance criteria for one or more of the labeled compounds associated with sample: PDI-SG-S216. Nonconformances are summarized in Attachment A, Table A-2.

Sample results were qualified as follows:

Actions: (Based on National Functional Guidelines 2016)

Criteria	Actions*	
	Detected	Nondetected
IAR criteria not met in sample but met in all associated calibration standards	J	UJ
IAR fails in sample and fails in any one of associated calibration standards	J	R

* Method-listed associated quantitation reference congener results were qualified

Sample Results/Reporting Issues

The 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.

Sample PDI-SG-S208 exhibited elevated noise or matrix interferences for one or more analytes causing elevation of the detection limit. Because the reporting limit for the affected analytes was raised to the EDL, no further action was taken.

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. The 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-S208	SE	PCB-123	0.064	0.011	ng/g	JN	k
PDI-SG-S208	SE	PCB-145	0.013	0.00097	ng/g	JN	k
PDI-SG-S208	SE	PCB-34	0.0064	0.0051	ng/g	JN	k
PDI-SG-S208	SE	PCB-48	0.15	0.068	ng/g	JN	k
PDI-SG-S208	SE	PCB-58	0.051	0.047	ng/g	JN	k
PDI-SG-S216	SE	PCB-10	0.0032	0.0015	ng/g	JN	k
PDI-SG-S216	SE	PCB-2	0.052	0.00084	ng/g	J	lc
PDI-SG-S216	SE	PCB-27	0.033	0.00072	ng/g	JN	k
PDI-SG-S216	SE	PCB-3	0.063	0.00099	ng/g	J	lc
PDI-SG-S216	SE	PCB-46	0.065	0.0095	ng/g	JN	k
PDI-SG-S216	SE	PCB-58	0.016	0.0052	ng/g	JN	k
PDI-SG-S216	SE	PCB-63	0.040	0.0047	ng/g	JN	k
PDI-SG-S216	SE	PCB-67	0.025	0.0050	ng/g	JN	k
PDI-SG-S216	SE	PCB-68	0.071	0.0047	ng/g	JN	k
PDI-SG-S216	SE	PCB-72	0.065	0.0053	ng/g	JN	k
PDI-SG-S216	SE	PCB-9	0.0079	0.0015	ng/g	JN	k

Attachment A

Nonconformance Summary Tables

Table A-1 – Laboratory Blanks

Blank ID	Compound	Result	QL	Unit	BAL	Associated Samples
MB 140-20224/17-B	PCB-1	0.000462	0.010	ng/g	0.00231	PDI-SG-S208 PDI-SG-S216
	PCB-101	0.00110	0.030	ng/g	0.0055	
	PCB-109	0.000374	0.060	ng/g	0.00187	
	PCB-11	0.00122	0.020	ng/g	0.0061	
	PCB-110	0.000718	0.020	ng/g	0.00359	
	PCB-113	0.00110	0.030	ng/g	0.0055	
	PCB-115	0.000718	0.020	ng/g	0.00359	
	PCB-118	0.000433	0.010	ng/g	0.002165	
	PCB-119	0.000374	0.060	ng/g	0.00187	
	PCB-12	0.000864	0.020	ng/g	0.00432	
	PCB-125	0.000374	0.060	ng/g	0.00187	
	PCB-129	0.000345	0.040	ng/g	0.001725	
	PCB-13	0.000864	0.020	ng/g	0.00432	
	PCB-132	0.000143	0.010	ng/g	0.000715	
	PCB-138	0.000345	0.040	ng/g	0.001725	
	PCB-139	0.0000746	0.020	ng/g	0.000373	
	PCB-140	0.0000746	0.020	ng/g	0.000373	
	PCB-147	0.000354	0.020	ng/g	0.00177	
	PCB-149	0.000354	0.020	ng/g	0.00177	
	PCB-15	0.000545	0.010	ng/g	0.002725	
	PCB-153	0.000774	0.020	ng/g	0.00387	
	PCB-160	0.000345	0.040	ng/g	0.001725	
	PCB-163	0.000345	0.040	ng/g	0.001725	
	PCB-167	0.000151	0.010	ng/g	0.000755	
	PCB-168	0.000774	0.020	ng/g	0.00387	
	PCB-170	0.000118	0.010	ng/g	0.00059	
	PCB-179	0.0000245	0.010	ng/g	0.000123	
	PCB-180	0.000465	0.020	ng/g	0.002325	
	PCB-183	0.000503	0.020	ng/g	0.002515	
	PCB-185	0.000503	0.020	ng/g	0.002515	
	PCB-19	0.000649	0.010	ng/g	0.003245	
	PCB-193	0.000465	0.020	ng/g	0.002325	
	PCB-20	0.000366	0.020	ng/g	0.00183	
	PCB-202	0.0000612	0.010	ng/g	0.000306	
	PCB-24	0.000165	0.010	ng/g	0.000825	
	PCB-28	0.000366	0.020	ng/g	0.00183	
	PCB-31	0.000727	0.020	ng/g	0.003635	
	PCB-32	0.000802	0.010	ng/g	0.00401	
	PCB-4	0.000938	0.020	ng/g	0.00469	
	PCB-44	0.00121	0.030	ng/g	0.00605	
	PCB-47	0.00121	0.030	ng/g	0.00605	
	PCB-52	0.000690	0.010	ng/g	0.00345	
	PCB-61	0.000582	0.040	ng/g	0.00291	

Blank ID	Compound	Result	QL	Unit	BAL	Associated Samples
	PCB-65	0.00121	0.030	ng/g	0.00605	
	PCB-70	0.000582	0.040	ng/g	0.00291	
	PCB-74	0.000582	0.040	ng/g	0.00291	
	PCB-76	0.000582	0.040	ng/g	0.00291	
	PCB-83	0.000404	0.020	ng/g	0.00202	
	PCB-86	0.000374	0.060	ng/g	0.00187	
	PCB-87	0.000374	0.060	ng/g	0.00187	
	PCB-90	0.00110	0.030	ng/g	0.0055	
	PCB-94	0.000177	0.010	ng/g	0.000885	
	PCB-95	0.000652	0.010	ng/g	0.00326	
	PCB-97	0.000374	0.060	ng/g	0.00187	
	PCB-99	0.000404	0.020	ng/g	0.00202	

Table A-2 – Ion Abundance Ratios for Labeled Compound and Labeled Clean-Up Standard

Sample ID	Labeled Compound and Labeled Clean-up Standard	Ion Abundance Ratio	Lower Limit	Upper limit
PDI-SG-S216	PCB-3L	3.69	2.66	3.60

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
ma	Multiple analyses. Sample analyzed more than once, a value from another analysis should be used.
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