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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling	
Laboratory:	Test America, Knoxville, Tennessee	
Service Request:	580-80213-8	
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 September 7, 2018.

Sample ID	Matrix/Sample Type
PDI-RB-VV-090718	Equipment Blank

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. 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 and the equipment blank in this data set. The equipment blank contamination, after laboratory method blank actions were applied, is summarized below for informational purposes only.

Blank ID	Compound	Result	ML	Units
PDI-RB-VV-090718	PCB-11	0.012	0.0048	ng/L
PDI-RB-VV-090718	PCB-18	0.0044	0.00053	ng/L
PDI-RB-VV-090718	PCB-21	0.0024	0.00058	ng/L
PDI-RB-VV-090718	PCB-30	0.0044	0.00053	ng/L
PDI-RB-VV-090718	PCB-31	0.0031	0.00058	ng/L
PDI-RB-VV-090718	PCB-33	0.0024	0.00058	ng/L
PDI-RB-VV-090718	PCB-52	0.0029	0.0010	ng/L

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 $\geq$ the 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.

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 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 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.

It should be noted that the overall bias is considered to be indeterminate in cases where cumulative nonconformances do not show a consistent bias or in cases of the presence of conflicting high and low biases.

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.

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-VV-090718	WQ	PCB-101		0.0026	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-109		0.0028	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-11	0.012	0.0048	ng/L	JN	bl,k
PDI-RB-VV-090718	WQ	PCB-110		0.0017	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-113		0.0026	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-115		0.0017	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-118		0.0012	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-119		0.0028	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-125		0.0028	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-129		0.0042	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-138		0.0042	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-147		0.0020	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-149		0.0020	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-160		0.0042	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-163		0.0042	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-18	0.0044	0.00053	ng/L	J+	bl
PDI-RB-VV-090718	WQ	PCB-183		0.0019	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-185		0.0019	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-20		0.0025	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-209 (decachlorobiphenyl)		0.0010	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-21	0.0024	0.00058	ng/L	JN	bl,k
PDI-RB-VV-090718	WQ	PCB-28		0.0025	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-3		0.0015	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-30	0.0044	0.00053	ng/L	J+	bl
PDI-RB-VV-090718	WQ	PCB-31	0.0031	0.00058	ng/L	J+	bl
PDI-RB-VV-090718	WQ	PCB-33	0.0024	0.00058	ng/L	JN	bl,k
PDI-RB-VV-090718	WQ	PCB-44		0.0062	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-47		0.0062	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-52	0.0029	0.0010	ng/L	JN	k
PDI-RB-VV-090718	WQ	PCB-61		0.0018	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-65		0.0062	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-70		0.0018	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-74		0.0018	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-76		0.0018	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-86		0.0028	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-87		0.0028	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-90		0.0026	ng/L	U	bl
PDI-RB-VV-090718	WQ	PCB-97		0.0028	ng/L	U	bl

Attachment A

Nonconformance Summary Tables

Table A-1 - Laboratory Blanks

Blank ID	Compound	Result	ML	Units	BAL	Associated Samples
MB 140-24007/6-A	PCB-101	0.00382	0.00015	ng/L	0.0191	PDI-RB-VV-090718
	PCB-105	0.00333	0.00023	ng/L	0.01665	
	PCB-109	0.00374	0.00014	ng/L	0.0187	
	PCB-11	0.0114	0.0017	ng/L	0.057	
	PCB-110	0.00455	0.00012	ng/L	0.02275	
	PCB-113	0.00382	0.00015	ng/L	0.0191	
	PCB-114	0.000541	0.00022	ng/L	0.002705	
	PCB-115	0.00455	0.00012	ng/L	0.02275	
	PCB-116	0.00172	0.00014	ng/L	0.0086	
	PCB-117	0.00172	0.00014	ng/L	0.0086	
	PCB-118	0.00706	0.00023	ng/L	0.0353	
	PCB-119	0.00374	0.00014	ng/L	0.0187	
	PCB-125	0.00374	0.00014	ng/L	0.0187	
	PCB-126	0.000852	0.00025	ng/L	0.00426	
	PCB-128	0.00218	0.00044	ng/L	0.0109	
	PCB-129	0.0106	0.00045	ng/L	0.053	
	PCB-138	0.0106	0.00045	ng/L	0.053	
	PCB-141	0.000915	0.00053	ng/L	0.004575	
	PCB-144	0.000742	0.000063	ng/L	0.00371	
	PCB-146	0.00110	0.00050	ng/L	0.0055	
	PCB-147	0.00335	0.00057	ng/L	0.01675	
	PCB-149	0.00335	0.00057	ng/L	0.01675	
	PCB-153	0.00708	0.00040	ng/L	0.0354	
	PCB-156	0.00430	0.00048	ng/L	0.0215	
	PCB-157	0.00430	0.00048	ng/L	0.0215	
	PCB-158	0.00192	0.00036	ng/L	0.0096	
	PCB-16	0.00165	0.00017	ng/L	0.00825	
	PCB-160	0.0106	0.00045	ng/L	0.053	
	PCB-163	0.0106	0.00045	ng/L	0.053	
	PCB-164	0.000760	0.00040	ng/L	0.0038	
	PCB-166	0.00218	0.00044	ng/L	0.0109	
	PCB-168	0.00708	0.00040	ng/L	0.0354	
	PCB-17	0.00145	0.00016	ng/L	0.00725	
	PCB-170	0.00753	0.00024	ng/L	0.03765	

Blank ID	Compound	Result	ML	Units	BAL	Associated Samples
	PCB-171	0.00188	0.00026	ng/L	0.0094	
	PCB-172	0.000807	0.00026	ng/L	0.004035	
	PCB-173	0.00188	0.00026	ng/L	0.0094	
	PCB-174	0.00365	0.00024	ng/L	0.01825	
	PCB-177	0.00202	0.00025	ng/L	0.0101	
	PCB-178	0.000525	0.00025	ng/L	0.002625	
	PCB-179	0.000616	0.00019	ng/L	0.00308	
	PCB-18	0.00358	0.00014	ng/L	0.0179	
	PCB-180	0.0158	0.00019	ng/L	0.079	
	PCB-183	0.00497	0.00023	ng/L	0.02485	
	PCB-185	0.00497	0.00023	ng/L	0.02485	
	PCB-187	0.00352	0.00022	ng/L	0.0176	
	PCB-189	0.000827	0.000093	ng/L	0.004135	
	PCB-190	0.00144	0.00017	ng/L	0.0072	
	PCB-191	0.000554	0.00017	ng/L	0.00277	
	PCB-193	0.0158	0.00019	ng/L	0.079	
	PCB-194	0.00386	0.000093	ng/L	0.0193	
	PCB-195	0.00125	0.00010	ng/L	0.00625	
	PCB-196	0.00193	0.000019	ng/L	0.00965	
	PCB-198	0.00331	0.000019	ng/L	0.01655	
	PCB-199	0.00331	0.000019	ng/L	0.01655	
	PCB-20	0.00362	0.00016	ng/L	0.0181	
	PCB-203	0.000936	0.000017	ng/L	0.00468	
	PCB-209 (decachlorobiphenyl)	0.00341	0.000019	ng/L	0.01705	
	PCB-21	0.00189	0.00016	ng/L	0.00945	
	PCB-22	0.000667	0.00016	ng/L	0.003335	
	PCB-28	0.00362	0.00016	ng/L	0.0181	
	PCB-3	0.00181	0.00019	ng/L	0.00905	
	PCB-30	0.00358	0.00014	ng/L	0.0179	
	PCB-31	0.00227	0.00016	ng/L	0.01135	
	PCB-32	0.000825	0.00011	ng/L	0.004125	
	PCB-33	0.00189	0.00016	ng/L	0.00945	
	PCB-37	0.00125	0.00016	ng/L	0.00625	
	PCB-44	0.0117	0.0012	ng/L	0.0585	
	PCB-47	0.0117	0.0012	ng/L	0.0585	
	PCB-56	0.00200	0.00096	ng/L	0.01	
	PCB-61	0.00430	0.00092	ng/L	0.0215	
	PCB-65	0.0117	0.0012	ng/L	0.0585	
	PCB-70	0.00430	0.00092	ng/L	0.0215	

Blank ID	Compound	Result	ML	Units	BAL	Associated Samples
	PCB-74	0.00430	0.00092	ng/L	0.0215	
	PCB-76	0.00430	0.00092	ng/L	0.0215	
	PCB-8	0.00271	0.0016	ng/L	0.01355	
	PCB-83	0.00201	0.00018	ng/L	0.01005	
	PCB-85	0.00172	0.00014	ng/L	0.0086	
	PCB-86	0.00374	0.00014	ng/L	0.0187	
	PCB-87	0.00374	0.00014	ng/L	0.0187	
	PCB-88	0.000819	0.00017	ng/L	0.004095	
	PCB-90	0.00382	0.00015	ng/L	0.0191	
	PCB-91	0.000819	0.00017	ng/L	0.004095	
	PCB-92	0.00173	0.00017	ng/L	0.00865	
	PCB-95	0.00205	0.00018	ng/L	0.01025	
	PCB-97	0.00374	0.00014	ng/L	0.0187	
	PCB-99	0.00201	0.00018	ng/L	0.01005	

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