

# Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling	
Laboratory:	Test America, Knoxville, Tennessee	
Service Request:	580-80635-9	
Analyses/Method:	Chlorinated Biphenyls by HRGC/HRMS / E1668A	
Validation Level:	Stage 2A	
AECOM Project Number:	60566335.2.12	
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## SUMMARY

The sample listed below was collected by AECOM in Portland Harbor in Portland, OR on August 17, 2018.

Sample ID	Matrix/Sample Type
PDI-SG-B474	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
X	Labeled compounds and labeled clean-up standard recoveries
X	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 (X) 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 sample was received on dry ice at -10 degrees Celsius and placed on hold. The method QC acceptance criteria were met.

An extra container for each sample was provided to be archived frozen at the TestAmerica Sacramento laboratory pending potential additional analyses. On October 23, 2018 the laboratory was instructed to analyze the extra container for sample PDI-SG-B474 for PCB congeners.

### 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 sample in this data set. An equipment blank was not submitted with this data set.

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. Method blank detected compounds are summarized in Attachment A, Table A-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 laboratory case narrative that ion abundance ratios (IARs) were outside acceptance criteria for one or more of the labeled compounds associated with sample PDI-SG-B474. 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.

### **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-B474	SE	PCB-1	0.00098	0.00013	ng/g	JN	k
PDI-SG-B474	SE	PCB-128	0.0074	0.0018	ng/g	JN	k
PDI-SG-B474	SE	PCB-16		0.00036	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-166	0.0074	0.0018	ng/g	JN	k
PDI-SG-B474	SE	PCB-17	0.0024	0.00033	ng/g	J	lc
PDI-SG-B474	SE	PCB-18	0.0031	0.00029	ng/g	JN	k,lc
PDI-SG-B474	SE	PCB-19		0.00040	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-20	0.0066	0.00043	ng/g	J	lc
PDI-SG-B474	SE	PCB-206	0.0061	0.0022	ng/g	JN	k
PDI-SG-B474	SE	PCB-21	0.0033	0.00042	ng/g	J+	lc,bl
PDI-SG-B474	SE	PCB-22	0.0023	0.00044	ng/g	J	lc
PDI-SG-B474	SE	PCB-23		0.00044	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-24		0.00027	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-25		0.00040	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-26	0.0012	0.00043	ng/g	J	lc
PDI-SG-B474	SE	PCB-27		0.00024	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-28	0.0066	0.00043	ng/g	J	lc
PDI-SG-B474	SE	PCB-29	0.0012	0.00043	ng/g	J	lc
PDI-SG-B474	SE	PCB-30	0.0031	0.00029	ng/g	JN	k,lc
PDI-SG-B474	SE	PCB-31	0.0059	0.00042	ng/g	J	lc
PDI-SG-B474	SE	PCB-32	0.0010	0.00023	ng/g	JN	k,lc
PDI-SG-B474	SE	PCB-33	0.0033	0.00042	ng/g	J+	lc,bl
PDI-SG-B474	SE	PCB-34		0.00046	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-35		0.00044	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-36		0.00043	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-37	0.0019	0.00044	ng/g	JN	k
PDI-SG-B474	SE	PCB-38		0.00046	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-39		0.00041	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-40	0.0030	0.0011	ng/g	JN	k,lc
PDI-SG-B474	SE	PCB-41	0.0030	0.0011	ng/g	JN	k,lc
PDI-SG-B474	SE	PCB-42	0.0028	0.0012	ng/g	J	lc
PDI-SG-B474	SE	PCB-43		0.0011	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-44	0.010	0.0010	ng/g	J	lc
PDI-SG-B474	SE	PCB-45		0.0012	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-46		0.0015	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-47	0.010	0.0010	ng/g	J	lc
PDI-SG-B474	SE	PCB-48		0.0011	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-49	0.0071	0.00094	ng/g	J	lc
PDI-SG-B474	SE	PCB-50		0.0011	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-51		0.0012	ng/g	UJ	lc

Sample ID	Matrix	Compound	Result	EDL	Units	Validation Qualifiers	Validation Reason
PDI-SG-B474	SE	PCB-52	0.014	0.0011	ng/g	J	lc
PDI-SG-B474	SE	PCB-53		0.0011	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-54		0.000070	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-55		0.00084	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-56	0.0043	0.00084	ng/g	J	lc
PDI-SG-B474	SE	PCB-57		0.00085	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-58		0.00086	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-59		0.00081	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-60	0.0022	0.00085	ng/g	J	lc
PDI-SG-B474	SE	PCB-61	0.018	0.00080	ng/g	J	lc
PDI-SG-B474	SE	PCB-62		0.00081	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-63		0.00078	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-64	0.0041	0.00077	ng/g	J	lc
PDI-SG-B474	SE	PCB-65	0.010	0.0010	ng/g	J	lc
PDI-SG-B474	SE	PCB-66	0.011	0.00080	ng/g	J	lc
PDI-SG-B474	SE	PCB-67		0.00073	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-68		0.00075	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-69	0.0071	0.00094	ng/g	J	lc
PDI-SG-B474	SE	PCB-70	0.018	0.00080	ng/g	J	lc
PDI-SG-B474	SE	PCB-71	0.0030	0.0011	ng/g	JN	k,lc
PDI-SG-B474	SE	PCB-72		0.00083	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-73		0.0011	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-74	0.018	0.00080	ng/g	J	lc
PDI-SG-B474	SE	PCB-75		0.00081	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-76	0.018	0.00080	ng/g	J	lc
PDI-SG-B474	SE	PCB-78		0.00086	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-79		0.00074	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-80		0.00073	ng/g	UJ	lc
PDI-SG-B474	SE	PCB-82	0.0034	0.00048	ng/g	JN	k
PDI-SG-B474	SE	PCB-95	0.018	0.00045	ng/g	JN	k

## Attachment A

## Nonconformance Summary Tables

Table A-1 – Laboratory Blanks

Blank ID	Compound	Result	QL	Unit	BAL	Associated Samples
MB 140-25237/16-B	PCB-109	0.00110	0.060	ng/g	0.0055	PDI-SG-B474
	PCB-110	0.00101	0.020	ng/g	0.00505	
	PCB-115	0.00101	0.020	ng/g	0.00505	
	PCB-118	0.000694	0.010	ng/g	0.00347	
	PCB-119	0.00110	0.060	ng/g	0.0055	
	PCB-125	0.00110	0.060	ng/g	0.0055	
	PCB-129	0.00303	0.040	ng/g	0.01515	
	PCB-138	0.00303	0.040	ng/g	0.01515	
	PCB-147	0.00262	0.020	ng/g	0.0131	
	PCB-149	0.00262	0.020	ng/g	0.0131	
	PCB-153	0.00194	0.020	ng/g	0.0097	
	PCB-160	0.00303	0.040	ng/g	0.01515	
	PCB-163	0.00303	0.040	ng/g	0.01515	
	PCB-168	0.00194	0.020	ng/g	0.0097	
	PCB-174	0.00144	0.010	ng/g	0.0072	
	PCB-180	0.00187	0.020	ng/g	0.00935	
	PCB-183	0.00103	0.020	ng/g	0.00515	
	PCB-185	0.00103	0.020	ng/g	0.00515	
	PCB-193	0.00187	0.020	ng/g	0.00935	
	PCB-20	0.000922	0.020	ng/g	0.00461	
	PCB-21	0.000822	0.020	ng/g	0.00411	
	PCB-28	0.000922	0.020	ng/g	0.00461	
	PCB-33	0.000822	0.020	ng/g	0.00411	
	PCB-37	0.000360	0.010	ng/g	0.0018	
	PCB-44	0.00183	0.030	ng/g	0.00915	
	PCB-47	0.00183	0.030	ng/g	0.00915	
	PCB-52	0.000983	0.010	ng/g	0.004915	
	PCB-61	0.00110	0.040	ng/g	0.0055	
	PCB-65	0.00183	0.030	ng/g	0.00915	
	PCB-66	0.000657	0.010	ng/g	0.003285	
	PCB-70	0.00110	0.040	ng/g	0.0055	
	PCB-74	0.00110	0.040	ng/g	0.0055	
	PCB-76	0.00110	0.040	ng/g	0.0055	
	PCB-83	0.00101	0.020	ng/g	0.00505	
PCB-86	0.00110	0.060	ng/g	0.0055		
PCB-87	0.00110	0.060	ng/g	0.0055		
PCB-95	0.00151	0.010	ng/g	0.00755		
PCB-97	0.00110	0.060	ng/g	0.0055		
PCB-99	0.00101	0.020	ng/g	0.00505		

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

<b>Sample ID</b>	<b>Labeled Compound and Labeled Clean-up Standard</b>	<b>Ion Abundance Ratio</b>	<b>Lower Limit</b>	<b>Upper limit</b>
PDI-SG-B474	PCB-54L	0.90	0.65	0.89
	PCB-19L	1.29	0.88	1.20

**Attachment B****Qualifier Codes and Explanations**

<b>Qualifier</b>	<b>Explanation</b>
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