

Data Validation Report

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|-----------------------|-------------------------------------------------------------------------|---------------|-----------------|
| Project: | Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling | | |
| Laboratory: | Test America, West Sacramento, California | | |
| Laboratory Group: | 580-78378-2 | | |
| Analyses/Method: | Clean Water Act - Dioxins and Furans / CWA1613B | | |
| Validation Level: | Stage 2A | | |
| AECOM Project Number: | 60566335.2.12 | | |
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| Reviewed by: | Waverly Braunstein/AECOM | File Name: | 580-78378-2_DVR |

SUMMARY

The sample listed below was collected by AECOM in Portland Harbor in Portland, OR on June 25, 2018.

| Sample ID | Matrix/Sample Type |
|-----------------|--------------------|
| PDI-SG-B264-BL1 | Sediment |

Data validation activities were conducted with reference to:

- EPA Method 1613B: *Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS (October 1994)*,
- 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 (OPR) results
- NA Field duplicate results

- X Labeled compound and 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 and/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

Laboratory method blanks and equipment blank results are evaluated as to whether there are contaminants detected above the estimated detection limit (EDL). An equipment blank was not submitted with the data package. Target compounds were detected in the blanks associated with the samples in this data set.

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 PCDD/PCDFs 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 and \leq the BAL, and, 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.

OPR Results

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 Compound and Clean-up Standard Recoveries

The labeled compounds and labeled clean-up standard %Rs were reviewed for conformance with the QC acceptance criteria.

Nonconformances are summarized in Attachment A in Table A-2. Samples were qualified as follows:

Actions: (Based on NFG 2016)

| Criteria ¹ | | Actions ² | |
|-----------------------------------------------------|---------------------------|----------------------|-------------|
| | | Detected | Nondetected |
| %R > Upper Acceptance Limit | | J | UJ |
| %R >10% but < Lower Acceptance Limit | | J | UJ |
| %R <10% | | See below | |
| <10% and S/N >10:1 | | J | R |
| <10% and S/N <10:1 | | R | R |
| Ion abundance ratio criteria not met | Calibration compliant | J | UJ |
| | Calibration non-compliant | J | R |
| Clean-up Standard Recovery < Lower Acceptance Limit | | J | UJ |

¹See Table 7 in method 1613B for acceptance criteria

²The dioxin method is performed using isotope dilution technique; therefore, professional judgment was applied and bias codes were not included in data qualification.

Qualified sample results are summarized in Table 1.

Sample Results/Reporting Issues

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.

Due to the matrix, the initial volumes used for the sediment sample deviated from the standard procedure. The reporting limits (RLs) have been adjusted proportionately.

Second Column Confirmation (2,3,7,8-TCDF)

The sample data were reviewed to ensure that results for 2,3,7,8-TCDF when analyzed on a DB-5 (or equivalent) column were confirmed on a second column (i.e., DB-225 or equivalent) when isomer specificity is not achieved. All sample results requiring confirmation were confirmed and results were reported from the confirmation column.

Estimated Maximum Possible Concentrations (EMPCs)

The data were reviewed to identify sample results that were indicated by the laboratory to be estimated maximum possible concentrations (EMPCs) because of identification criteria not being met.

The laboratory qualified all sample results with a "q" laboratory qualifier to indicate that the ion ratio criterion was not met. Results qualified with the "q" laboratory qualifier were qualified as estimated and tentatively identified (JN). Qualified sample results are shown 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 dioxins and furans 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. Qualification on this basis was not required.

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-B264-BL1 | SE | 1,2,3,4,6,7,8-HpCDF | 0.011 | 0.00034 | ug/kg | JN | k,lc |
| PDI-SG-B264-BL1 | SE | 1,2,3,6,7,8-HxCDF | 0.00056 | 0.00015 | ug/kg | JN | k |
| PDI-SG-B264-BL1 | SE | 1,2,3,7,8,9-HxCDF | 0.00018 | 0.000081 | ug/kg | J+ | bl |
| PDI-SG-B264-BL1 | SE | 1,2,3,7,8-PeCDF | 0.00040 | 0.000072 | ug/kg | J+ | bl |
| PDI-SG-B264-BL1 | SE | 2,3,7,8-TCDD | 0.00030 | 0.000069 | ug/kg | JN | k |

Attachment A

Nonconformance Summary Tables

Table A-1 - Lab Blanks

| Blank ID | Compound | Result | QL | BAL | Units | Associated Samples |
|-------------------|---------------------|-----------|--------|-----------|-------|--------------------|
| MB 320-232323/1-A | 1,2,3,4,6,7,8-HpCDD | 0.000229 | 0.0050 | 0.001145 | ug/kg | PDI-SG-B264-BL1 |
| | 1,2,3,4,6,7,8-HpCDF | 0.000120 | 0.0050 | 0.0006 | ug/kg | |
| | 1,2,3,4,7,8,9-HpCDF | 0.000117 | 0.0050 | 0.000585 | ug/kg | |
| | 1,2,3,4,7,8-HxCDD | 0.0000973 | 0.0050 | 0.0004865 | ug/kg | |
| | 1,2,3,7,8,9-HxCDF | 0.000144 | 0.0050 | 0.00072 | ug/kg | |
| | 1,2,3,7,8-PeCDF | 0.0000879 | 0.0050 | 0.0004395 | ug/kg | |
| | OCDD | 0.000919 | 0.010 | 0.004595 | ug/kg | |
| | OCDF | 0.000134 | 0.010 | 0.00067 | ug/kg | |

Table A-2 - Labeled Compound Recoveries

| Sample ID | Labeled Compound | % Recovery | Lower Limit | Upper Limit |
|-----------------|-------------------------|------------|-------------|-------------|
| PDI-SG-B264-BL1 | 13C-1,2,3,4,6,7,8-HpCDF | 23 | 28 | 143 |

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 |