

## Data Validation Report

|                       |   |                             |
|-----------------------|---|-----------------------------|
| Project:              | Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling |                             |
| Laboratory:           | SGS-AXYS, Sydney, British Columbia, Canada                              |                             |
| Laboratory Group:     | WG66481-PEST  |                             |
| Analyses/Method:      | Pesticides by HRGC/HRMS / EPA Method 1699                               |                             |
| 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 November 27-30, 2018 and December 1, 2018.

| Sample ID        | Matrix/Sample Type |
|------------------|--------------------|
| PDI-RB-XF-181129 | Equipment Blank    |
| PDI-WS-T01-1811  | Surface Water      |
| PDI-WS-T02-1811  | Surface Water      |
| PDI-WS-T03-1811  | Surface Water      |
| PDI-WS-T04-1812  | Surface Water      |
| PDI-WS-T05-1811  | Surface Water      |
| PDI-WS-T06-1811  | Surface Water      |
| PDI-WS-T07-1811  | Surface Water      |

Data validation activities were conducted with reference to:

- EPA Method 1699: *Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS* (December 2007) ,
- *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
- ✗ Labeled compound 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

Laboratory method blanks and equipment 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 equipment blank associated with the samples in this data set.

Detected compounds are summarized in Attachment A in Table A-1 and Table A-2. The results for the equipment blank PDI-RB-XF-181129 are provided for informational purposes only.

The NFG guidance stipulates that a conservative approach should be taken with regards to qualification of data 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 on the basis of laboratory method blank contamination. As allowed in the NFG, a blank action limit (BAL) was determined as five 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, 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.

#### **OPR Results**

The OPR %Rs were reviewed for conformance with the method QC acceptance criteria. All method QC acceptance criteria were met.

The laboratory noted in the case narrative that the solvent went to dryness for the OPR and samples PDI-WS-T06-1811 and PDI-RB-XF-181129 during sample extraction. A new OPR was created and extracted, replacing the original OPR. The OPR was re-united with the batch and the clean-up of the samples continued. Additional sample was not available for the re-extraction of these samples. No data validation actions were taken on this basis.

#### **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 Recoveries**

The labeled compound %Rs were reviewed for conformance with the QC acceptance criteria. The laboratory noted in the case narrative that the solvent went dry during the extraction procedure of samples PDI-WS-T06-1811 and PDI-RB-XF-181129 along with the associated OPR. Additional sample was not available for the re-extraction of these samples. However, given that the extract went dry after the addition of the labeled extraction standards and that isotope dilution quantification corrects for such losses, data are not considered significantly impacted. All labeled extraction standards met criteria in sample PDI-WS-T06-1811.

Nonconformances noted for sample PDI-RB-XF-181129 are summarized in Attachment A in Table A-3.

Samples were qualified as follows:

**Actions:** (Based on NFG 2016)

| Criteria <sup>1</sup>  |                           | Actions <sup>2</sup> |             |
|--|---------------------------|----------------------|-------------|
|  |                           | 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          |
| <sup>1</sup> See Table 7 in method 1613B for acceptance criteria<br><sup>2</sup> The 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.

It should be noted that the sample reported detection limit is the sample specific estimated detection limit (EDL) with the following exceptions. In cases where the EDL is less than the nominal concentration of 0.2 ng for hexachlorobenzene or 0.4 ng for the remaining compounds, then the EDL is raised to these nominal concentrations and are adjusted to include the appropriate preparation factors

### Lock Mass Interferences

The laboratory identified the presence of interferences of the mass ion as indicated by the monitored lock mass by qualifying the affected sample result with a G laboratory qualifier. These interferences may impact compound quantitation; therefore, the positive and nondetect results for affected samples were qualified as estimated (J/UJ).

### 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 identified the presence of EMPCs for the samples in this data set by qualifying affected results with a K laboratory qualifier. Samples were qualified as follows:

**Actions:** (Based on AECOM professional judgment)

| Criteria  | Actions   |
|---|---|
| A native target compound was reported by the laboratory as an EMPC.                                   | Report result as an EMPC and qualify as estimated and presumptively present (JN). |
| A labeled compound was flagged by the laboratory indicating all identification criteria were not met. | Qualify associated positive and nondetect results as estimated (J/UJ).            |

It should be noted that in instances of multiple nonconformances, the bias is considered indeterminate where there is a conflicting low and high bias or when a result does not exhibit a consistent bias. These results have an overall qualification of estimated (J) with the exception noted below.

When applicable, the "JN" qualifier was retained rather than replacement with the conventional overall "J" qualifier in instances where EMPC results were qualified for multiple quality control nonconformances.

Qualified sample results are summarized in Table 1.

## 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-XF-181129 | WQ     | 2,4-DDT           |        | 0.286  | ng/sample | UJ                    | lc                |
| PDI-RB-XF-181129 | WQ     | 4,4'-DDT          |        | 0.483  | ng/sample | UJ                    | lc                |
| PDI-RB-XF-181129 | WQ     | Aldrin            |        | 0.0543 | ng/sample | UJ                    | lc                |
| PDI-RB-XF-181129 | WQ     | alpha-Chlordane   | 0.113  | 0.0696 | ng/sample | JN                    | k                 |
| PDI-RB-XF-181129 | WQ     | cis-Nonachlor     | 0.091  | 0.0543 | ng/sample | JN                    | k                 |
| PDI-RB-XF-181129 | WQ     | Hexachlorobenzene | 0.059  | 0.0271 | ng/sample | JN                    | bl,k              |
| PDI-RB-XF-181129 | WQ     | Oxychlordane      |        | 0.0711 | ng/sample | UJ                    | lc,q              |
| PDI-WS-T01-1811  | WS     | Aldrin            | 0.079  | 0.0493 | ng/sample | JN                    | k                 |
| PDI-WS-T01-1811  | WS     | alpha-Chlordane   | 0.174  | 0.0493 | ng/sample | JN                    | k                 |
| PDI-WS-T01-1811  | WS     | cis-Nonachlor     | 0.177  | 0.0493 | ng/sample | JN                    | k                 |
| PDI-WS-T01-1811  | WS     | Oxychlordane      | 0.065  | 0.0493 | ng/sample | J                     | q                 |
| PDI-WS-T01-1811  | WS     | trans-Chlordane   | 0.194  | 0.0493 | ng/sample | JN                    | k                 |
| PDI-WS-T01-1811  | WS     | trans-Nonachlor   | 0.247  | 0.0493 | ng/sample | J+                    | bl                |
| PDI-WS-T02-1811  | WS     | Aldrin            | 0.126  | 0.0519 | ng/sample | JN                    | k                 |
| PDI-WS-T02-1811  | WS     | cis-Nonachlor     | 0.262  | 0.0519 | ng/sample | JN                    | k                 |
| PDI-WS-T02-1811  | WS     | Oxychlordane      | 0.113  | 0.0519 | ng/sample | JN                    | k,q               |
| PDI-WS-T03-1811  | WS     | Aldrin            | 0.057  | 0.0533 | ng/sample | JN                    | k                 |
| PDI-WS-T03-1811  | WS     | cis-Nonachlor     | 0.117  | 0.0533 | ng/sample | JN                    | k                 |
| PDI-WS-T03-1811  | WS     | Oxychlordane      | 0.071  | 0.0533 | ng/sample | JN                    | k,q               |
| PDI-WS-T03-1811  | WS     | trans-Nonachlor   | 0.230  | 0.0533 | ng/sample | J+                    | bl                |
| PDI-WS-T04-1812  | WS     | Aldrin            | 0.063  | 0.0504 | ng/sample | JN                    | k                 |
| PDI-WS-T04-1812  | WS     | alpha-Chlordane   | 0.237  | 0.0504 | ng/sample | JN                    | k                 |
| PDI-WS-T04-1812  | WS     | Oxychlordane      | 0.053  | 0.0504 | ng/sample | JN                    | k,q               |
| PDI-WS-T04-1812  | WS     | trans-Nonachlor   | 0.252  | 0.0504 | ng/sample | JN                    | bl,k              |
| PDI-WS-T05-1811  | WS     | cis-Nonachlor     | 0.332  | 0.0497 | ng/sample | JN                    | k                 |
| PDI-WS-T05-1811  | WS     | Oxychlordane      | 0.063  | 0.0497 | ng/sample | JN                    | k,q               |
| PDI-WS-T06-1811  | WS     | Aldrin            | 0.086  | 0.0520 | ng/sample | JN                    | k                 |
| PDI-WS-T06-1811  | WS     | cis-Nonachlor     | 0.165  | 0.0520 | ng/sample | JN                    | k                 |
| PDI-WS-T06-1811  | WS     | Oxychlordane      | 0.069  | 0.0520 | ng/sample | J                     | q                 |
| PDI-WS-T06-1811  | WS     | trans-Nonachlor   | 0.371  | 0.0520 | ng/sample | JN                    | k                 |
| PDI-WS-T07-1811  | WS     | Aldrin            | 0.056  | 0.0533 | ng/sample | JN                    | k                 |
| PDI-WS-T07-1811  | WS     | Oxychlordane      | 0.054  | 0.0533 | ng/sample | J                     | q                 |
| PDI-WS-T07-1811  | WS     | trans-Chlordane   | 0.302  | 0.0533 | ng/sample | JN                    | k                 |

## Attachment A

## Nonconformance Summary Tables

Table A-1 - Lab Blanks

| Blank ID    | Compound          | Result | QL   | BAL   | Units     | Associated Samples  |
|-------------|-------------------|--------|------|-------|-----------|---|
| WG66481-101 | Hexachlorobenzene | 0.056  | 2.56 | 0.280 | ng/sample | PDI-RB-XF-181129<br>PDI-WS-T01-1811<br>PDI-WS-T02-1811<br>PDI-WS-T03-1811<br>PDI-WS-T04-1812<br>PDI-WS-T05-1811<br>PDI-WS-T06-1811<br>PDI-WS-T07-1811 |
|             | trans-Nonachlor   | 0.061  | 4.36 | 0.305 | ng/sample |   |

Table A-2 - Field Blanks

| Blank ID         | Compound          | Result | QL   | Units     | Associated Samples  |
|------------------|-------------------|--------|------|-----------|---|
| PDI-RB-XF-181129 | Hexachlorobenzene | 0.059  | 2.71 | ng/sample | PDI-WS-T01-1811<br>PDI-WS-T02-1811<br>PDI-WS-T03-1811<br>PDI-WS-T04-1812<br>PDI-WS-T05-1811<br>PDI-WS-T06-1811<br>PDI-WS-T07-1811 |
|                  | alpha-Chlordane   | 0.113  | 5.44 | ng/sample |   |
|                  | cis-Nonachlor     | 0.091  | 5.42 | ng/sample |   |

Table A-3 - Labeled Compound Recoveries

| Sample ID        | Labeled Compound   | % Recovery | Lower Limit | Upper Limit |
|------------------|--------------------|------------|-------------|-------------|
| PDI-RB-XF-181129 | 13C-Chlordane, oxy | 25.5       | 30          | 200         |
|                  | 13C-Aldrin         | 29.2       | 30          | 150         |
|                  | 13C-4,4'-DDT       | 15.3       | 40          | 150         |
|                  | 13C-2,4'-DDT       | 20.7       | 40          | 150         |

**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  |
| 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  |