EcoChem, INC.
Environmental Data Quality

# DATA QUALITY EVALUATION

## PORTLAND HARBOR

**Surface Water – High Flow: XAD Resin Sampling – Spring 2006**

**Polycyclic Aromatic Hydrocarbons (PAH) – Axys Method MLA-021**
**Chlorinated Pesticides - Axys Method MLA-028**
**Polychlorinated Biphenyl (PCB) Congeners – EPA Method 1668A**
**Dioxin/Furan Compounds – EPA Method 1613 Ver. B**

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# DATA QUALITY EVALUATION

## BASIS OF DATA EVALUATION

The data were validated using guidance and quality control (QC) criteria documented in the analytical methods; *Guidance on Environmental Data Verification and Validation* (EPA 2002c); *Portland Harbor RI/FS, Round 2, Quality Assurance Project Plan* (QAPP) *Addendum 1: Surface Water;* and *National Functional Guidelines for Organic and/or Inorganic Data Review* (USEPA 1994, 1999 & 2002). Additional guidance for polychlorinated biphenyl (PCB) congener data validation were *Axys Standard Operating Procedure MLA-10, Revision 5; EPA Region 10 SOP for the Validation of Method 1668 Toxic, Dioxin-like PCB Data* (USEPA 1995).

All samples were analyzed by Axys Analytical Services, Ltd. Sidney, British Columbia, Canada.

Data qualifier definitions, reason codes, and validation criteria are included as **Appendix A**. Data validation reports, which discuss individual findings for each quality control element [by sample delivery group (SDG)], are provided in **Appendix B**. Data validation worksheets and all communication records are organized by SDG and will be kept on file at EcoChem.

## PROCESS FOR DATA VALIDATION

All electronic data deliverable files (EDD) were verified by comparing 100% of the field sample results and 10% of the QC sample results to the hardcopy data package (10% for PCB congener EDD files).

The XAD resin data received a Level III validation, which included evaluation (as appropriate for each method) of:

- Package completeness
- Sample chain-of-custody and sample preservation
- Analytical holding times
- Blank contamination
- Precision (replicate analyses)
- Accuracy (compound recovery)
- Detection limits
- Instrument performance (initial calibration, continuing calibration, tuning, sensitivity and degradation)

Ten percent (10%) of all XAD resin data packages received full (Level IV) data validation, which includes evaluation of compound identification and quantitation (transcription and calculation checks).

A dual-tier system of primary and secondary reviewers is utilized to ensure technical correctness and QC of the validation process; and all data validation is documented using standardized and controlled validation worksheets and spreadsheets. These worksheets are completed for each SDG, documenting all deficiencies, outliers and subsequent qualifiers.

After qualifiers are entered into the EcoChem database, a second party verifies 100% of the qualifier entry. Interpretive qualifiers are then applied to the field samples and qualified data is exported to the Project Database (Integral).

## SUMMARY OF DATA VALIDATION: POLYCYCLIC AROMATIC HYDROCARBONS

Six XAD resin samples were analyzed for polycyclic aromatic hydrocarbon (PAH) compounds for the Portland Harbor Surface Water – High Flow Sampling Event. Two rinsate blanks were collected to monitor the field collection process.

The PAH data for the XAD resin samples were acceptable. No data were rejected for any reason.

The laboratory data were evaluated in terms of completeness, holding times, instrument performance, bias, and precision. The results of the QC procedures used during sample analyses are discussed below.

### Completeness of Data Set

Completeness is defined as the total number of usable results (results that were not rejected during data validation) divided by the total results reported by the laboratory. The results reported by the laboratory were 100% complete for the XAD resin PAH analyses. Four data points were qualified as estimated. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

### Holding Times and Sample Preservation

No technical holding time requirements are established for extraction of XAD resins and filters. All analytical holding time requirements were met for PAH analyses of the XAD resin samples and associated field QC samples.

### Instrument Performance

Initial and continuing calibrations were completed for all target analytes and met the criteria for frequency of analysis. All initial calibrations and continuing calibration verifications met all acceptance criteria.

### Method Blank Analyses

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blanks are used to evaluate all associated samples.

Various target analytes were detected in the method blanks. Seventeen PAH results – four results each for acenaphthylene, anthracene, and 2-methylnaphthalene, two results for naphthalene, and one result each for benz(a)anthracene, dibenz(a,h)anthracene, and indeno(1,2,3-cd)pyrene - (12.5% of all XAD resin PAH results) were qualified as not detected (U) based on method blank contamination.

## Accuracy

The accuracy of the analytical results is evaluated in the following sections in terms of analytical bias (labeled compound and ongoing precision and recovery samples [OPR]) and precision (field duplicate analyses).

### *Labeled Compound Recoveries*

Labeled compounds were added to all field and QC samples. Four acenaphthene results and reporting limits (2.9% of all XAD resin PAH results) were estimated (J or UJ) for potential low bias due to low labeled compound recoveries.

### *Matrix Spike Recoveries*

Matrix and duplicate matrix spike (MS/MSD) analyses were not performed. Accuracy was assessed using the labeled compound and OPR analyses.

### *Ongoing Precision and Recovery Sample Recoveries*

OPR analyses met the criteria for frequency of analysis. The recoveries reported by the laboratory met the criteria for acceptable performance.

## Precision

Laboratory duplicate analyses were not performed. Precision was assessed using field duplicate analyses.

## Method Detection Limits and Method Reporting Limits

To meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the method detection limits (MDLs), adjusted for sample size, percent moisture, and any dilution factor. These method reporting limits (MRLs) ranged from 0.4 ng/L to 1 ng/L. The MRLs met project criteria for all analytes

## Compound Identification

Several different flags were used by the laboratory to provide information about the reported results. A “K” flag indicates that a peak was detected at the correct retention time for the target analyte; however, the ion abundance ratio criteria were not met. The reported result is an estimated maximum possible concentration value, which is essentially an elevated detection limit. Data flagged “K” by the laboratory were qualified as not detected (U) to make this relationship clear to the data user. Nineteen data points (13.9% of all XAD resin PAH data points) were qualified as not detected because the ion abundance ratio criteria were not met.

## Field Quality Control Samples

Field QC samples collected for the High Flow Sampling included field blank samples. The results for the field QC samples are discussed in the following sections.

### *Field Blanks*

Two field blanks (LW2-W1902 C and LW2-W1902 F) were associated with the XAD resin samples. After qualifiers based on method blank contamination were issued, no results were qualified based on field blank contamination.

### *Field Replicate Samples*

No field replicate sample pairs were associated with these XAD resin samples. Precision could not be assessed.

## SUMMARY OF DATA VALIDATION: PESTICIDE COMPOUNDS

Six XAD resin samples were analyzed for pesticide compounds for the Portland Harbor Surface Water – High Flow Sampling Event. Two (2) rinsate blanks were collected to monitor the field collection process.

The pesticide data for the XAD resin samples were generally acceptable. Fifteen (15) pesticide data points were rejected because control limits were significantly exceeded in one or more laboratory quality control (QC) samples or procedures. These rejected data points are not suitable for use. Other pesticide data points were qualified as estimated. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

The laboratory data were evaluated in terms of completeness, holding times, instrument performance, bias, and precision. The results of the QC procedures used during sample analyses are discussed below.

### Completeness of Data Set

Completeness is defined as the total number of usable results (results that were not rejected during data validation) divided by the total results reported by the laboratory. The results reported by the laboratory were 93% complete for the XAD resin pesticide analyses.

No results were reported for toxaphene, although it was originally designated as a target analyte for this project. This was noted in an email to Laura Jones at Integral and Georgina Brooks at Axys Analytical on June 1, 2005.

### Holding Times and Sample Preservation

No technical holding time requirements are established for extraction of XAD resins and filters. All analytical holding time requirements were met for pesticide analysis of the XAD resin samples and associated field QC samples.

### Instrument Performance

#### *Calibrations*

Initial and continuing calibrations were completed for all reported analytes except hexachlorobutadiene. Results for this analyte were calculated from a ratio of its response to that of another analyte, 1,2,4-trichlorobenzene. This calibration method does not meet project requirements; therefore, all hexachlorobutadiene results and detection limits were estimated (J or UJ). All other calibrations met the criteria for frequency of analysis and all initial calibrations met all acceptance criteria.

The continuing calibration percent difference (%D) values were used to evaluate instrument stability. Hexachlorobutadiene was not included in the continuing calibration checks, so all hexachlorobutadiene results and detection limits were estimated (J or UJ). All other calibrations met the criteria for frequency of analysis and all continuing calibrations met all acceptance criteria.

A total of seven hexachlorobutadiene results (3.1% of all XAD resin pesticide results) were qualified as estimated (J or UJ) based on calibration outliers.

### *Endrin/DDT Breakdown*

Breakdown evaluation mixtures were analyzed at the proper frequency to measure percent breakdown of 4,4'-DDT and endrin. All acceptance criteria were met.

## Method Blank Analyses

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blanks are used to evaluate all associated samples.

Method blanks were analyzed at the appropriate frequency. Two results each for hexachlorobenzene and hexachlorobutadiene (1.8% of all XAD resin pesticide results) were qualified as not detected (U) based on method blank contamination.

## Accuracy

The accuracy of the analytical results is evaluated in the following sections in terms of analytical bias (labeled compound and ongoing precision and recovery samples [OPR] recoveries) and precision (field duplicate analyses).

### *Labeled Compound Recoveries*

Labeled compounds were added to all field and QC samples. One reporting limit for hexachlorobutadiene (0.4% of all XAD resin pesticide results) was rejected due to extremely low labeled compound recovery. Seven other hexachlorobutadiene results and reporting limits (3.1% of all XAD resin pesticide results) were estimated (J or UJ) for potential low bias due to low labeled compound recoveries.

### *Matrix Spike Recoveries*

Matrix spike and matrix spike duplicate (MS/MSD) analyses were not performed. Accuracy was assessed using the labeled compound and OPR analyses.

### *Ongoing Precision and Recovery Sample Recoveries*

OPR analyses met the criteria for frequency of analysis. Hexachlorobutadiene was not included in the OPR samples. Seven hexachlorobutadiene results and reporting limits were estimated (J or UJ). Ten results were estimated and 14 reporting limits were rejected for beta-endosulfan, endrin, endrin aldehyde, endrin ketone, endosulfan sulfate and methoxychlor data points from four samples associated with extremely low OPR recoveries. A total of 31 results (13.8% of all XAD resin pesticide results) were qualified as estimated (J or UJ) based on OPR outliers.

## Precision

Laboratory duplicate analyses were not performed. Precision was assessed using field duplicate analyses.

## Method Detection Limits and Method Reporting Limits

To meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the method detection limits (MDLs), adjusted for sample size, percent moisture, and any dilution factor. These method reporting limits (MRLs) ranged from 0.01 pg/L to 0.06 pg/L for the non-detected results. The MRLs met project criteria for all analytes except toxaphene, which was not analyzed for the XAD resin samples.

## Compound Identification

Several different flags were used by the laboratory to provide information about the reported results. A "K" flag indicates that a peak was detected at the correct retention time for the target analyte; however, the ion abundance ratio criteria were not met. The reported result is an estimated maximum possible concentration value, which is essentially an elevated detection limit. Data flagged "K" by the laboratory were qualified as not detected (U) to make this relationship clear to the data user. Fifty-four data points (24.1% of all XAD resin pesticide data points) were qualified as not detected because the ion abundance ratio criteria were not met.

## Field Quality Control Samples

Field QC samples collected for the High Flow Sampling included field blank samples. The results for the field QC samples are discussed in the following sections.

### *Field Blanks*

Two field blanks (LW2-W1902 C and LW2-W1902 F) were associated with the XAD resin samples. After qualifiers based on method blank contamination were issued, no results were qualified based on field blank contamination.

### *Field Replicate Samples*

No field replicate sample pairs were associated with these XAD resin samples. Precision could not be assessed.

## SUMMARY OF DATA VALIDATION: PCB CONGENERS

Six XAD resin samples were analyzed for PCB congener compounds for the Portland Harbor Surface Water – High Flow Sampling Event. Two (2) rinsate blanks were collected to monitor the field collection process.

The laboratory data were evaluated in terms of completeness, holding times, instrument performance, bias, and precision. The results of the QC procedures used during sample analyses are discussed below.

### Completeness of Data Set

Completeness is defined as the total number of usable results (results that were not rejected during data validation) divided by the total results reported by the laboratory. The results reported by the laboratory were 100% complete for the XAD resin PCB analyses.

### Holding Times and Sample Preservation

No technical holding time requirements are established for extraction of XAD resins and filters. All analytical holding time requirements were met for the dioxin/furan analyses of the XAD resin samples and associated field QC samples.

### Instrument Performance

Initial and continuing calibrations were completed for all target analytes and met the criteria for frequency of analysis. All calibrations met all acceptance criteria.

All other instrument performance criteria were met by the laboratory.

### Method Blank Analyses

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blanks are used to evaluate all associated samples.

Method blanks were analyzed at the appropriate frequency. A total of 35 results (2.5% of all XAD resin PCB results) were qualified as not detected (U) based on method blank contamination.

### Accuracy

The accuracy of the analytical results is evaluated in the following sections in terms of analytical bias (labeled compound and ongoing precision and recovery samples [OPR]) and precision (field duplicate analyses).

### *Labeled Compound Recoveries*

Labeled compounds were added to all field and QC samples. One result (0.02% of all XAD resin PCB results) was qualified as estimated with potential low bias due to low recovery of a labeled compound.

### *Matrix Spike Recoveries*

Matrix and duplicate matrix spike (MS/MSD) analyses were not performed. Accuracy was assessed using the labeled compound and OPR analyses.

### *Ongoing Precision and Recovery Sample Recoveries*

OPR analyses met the criteria for frequency of analysis. The recoveries reported by the laboratory met the criteria for acceptable performance.

## Precision

Laboratory duplicate analyses were not performed. Precision was assessed using field duplicate analyses.

## Method Detection Limits and Method Reporting Limits

To try to meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the method detection limits (MDLs), adjusted for sample size, percent moisture, and any dilution factor. These MDLs ranged from 0.03 pg/L to 0.6 pg/L for the non-detected results.

## Compound Identification

Different flags were used by the laboratory to provide information about the reported results. A “K” flag indicates that a peak was detected at the correct retention time for the target analyte; however, the ion abundance ratio criteria were not met. The reported result is an estimated maximum possible concentration value, which is essentially an elevated detection limit. Data flagged “K” by the laboratory were qualified as not detected (U) to make this relationship clear to the data user. A total of 232 data points (16.5% of all XAD resin PCB data points) were qualified as not detected because the ion abundance ratio criteria were not met.

A "G" flag indicates a disturbance in the mass spectrometer detector lock mass associated with the target analyte. This lock mass disturbance may affect the quantitation of the analyte. Data flagged “G” by the laboratory were qualified as estimated (J) for potential interference. A total of three data points (0.2% of all XAD resin PCB data points) were qualified as estimated because of lock mass disturbance.

No Aroclor patterns were observed in the samples, but the laboratory reported results for Aroclors by summing the concentrations of certain PCB congeners and applying a laboratory-derived correction factor. All nine positive Aroclor results (0.6% of all XAD resin PCB data points) were qualified to indicate that they should be considered as tentatively identified at estimated concentrations.

## Field Quality Control Samples

Field QC samples collected for the High Flow Sampling included field blank and field replicate samples. The results for the field QC samples are discussed in the following sections.

### *Field Blanks*

Two field blanks (LW3-W1902 C and LW3-W1902 F) were associated with the XAD resin samples. After qualifiers based on method blank contamination were issued, 55 results (3.8% of all XAD resin PCB results) were qualified as not detected (U) based on field blank contamination.

### *Field Replicate Samples*

No field replicate sample pairs were associated with these XAD resin samples. Precision could not be assessed.

## SUMMARY OF DATA VALIDATION: DIOXIN/FURAN COMPOUNDS

### Basis of Data Evaluation

Dioxin/furan data were validated using guidance and quality control (QC) criteria documented in the analytical method (E1613); *Guidance on Environmental Data Verification and Validation* (EPA 2002c); *EPA Region 10 SOP for the Validation of Polychlorinated Dibenzodioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data* (EPA 1996), in addition to the sources listed above.

### Summary of Data Validation

Six XAD resin samples were analyzed for dioxin and furan compounds for the Portland Harbor Surface Water – High Flow Sampling Event. Two (2) rinsate blanks were collected to monitor the field collection process.

The dioxin/furan data for the XAD resin samples were generally acceptable. No data were rejected or estimated for any reason.

The laboratory data were evaluated in terms of completeness, holding times, instrument performance and bias. The results of the QC procedures used during sample analyses are discussed below.

### Completeness of Data Set

Completeness is defined as the total number of usable results (results that were not rejected during data validation) divided by the total results reported by the laboratory. The results reported by the laboratory were 100% complete for the XAD resin dioxin/furan analyses.

### Holding Times and Sample Preservation

No technical holding time requirements are established for extraction of XAD resins and filters. All analytical holding time requirements were met for the dioxin/furan analyses of the XAD resin samples and associated field QC samples.

### Instrument Performance

Initial and continuing calibrations were completed for all target analytes and met the criteria for frequency of analysis. All calibrations met all acceptance criteria.

### Method Blank Analyses

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blanks are used to evaluate all associated samples.

Method blanks were analyzed at the appropriate frequency. A total of eight results (3.8% of all XAD resin dioxin/furan results) were qualified as not detected (U) based on method blank contamination. The qualifiers were issued to three results for 1,2,3,7,8,9-HxCDF, and one result each for 2,3,4,7,8-PeCDF, 2,3,4,6,7,8-HxCDF, OCDD, total hexafurans and total heptadioxins.

## Accuracy

The accuracy of the analytical results is evaluated in the following sections in terms of analytical bias (labeled compound and ongoing precision and recovery samples [OPR]) and precision (field duplicate analyses).

### *Labeled Compound Recoveries*

Labeled compounds were added to all field and QC samples. The recoveries reported by the laboratory met the criteria for acceptable performance.

### *Matrix Spike Recoveries*

Matrix spike and matrix spike duplicate (MS/MSD) analyses were not performed. Accuracy was assessed using the labeled compound and OPR analyses.

### *Ongoing Precision and Recovery Sample Recoveries*

OPR analyses met the criteria for frequency of analysis. The recoveries reported by the laboratory met the criteria for acceptable performance.

## Precision

Field duplicate samples were not collected and laboratory duplicate analyses were not performed. Precision was not assessed.

## Method Detection Limits and Method Reporting Limits

To try to meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the method detection limits (MDLs), adjusted for sample size, percent moisture, and any dilution factor. These MDLs ranged from 0.001 pg/L to 0.01 pg/L for the non-detected results.

## Compound Identification

Flags were used by the laboratory to provide information about the reported results. A “K” flag indicates that a peak was detected at the correct retention time for the target analyte; however, the ion abundance ratio criteria were not met. The reported result is an EMPC (estimated maximum possible concentration) value, which is essentially an elevated detection limit. Data flagged with “K” by the laboratory were qualified as not detected (U) to make this relationship clear to the data user. Twenty-one (21) data points (9.9% of all XAD resin dioxin/furan data points) were qualified as not detected because the ion abundance ratio criteria were not met.

## Field Quality Control Samples

Field QC samples collected for the High Flow Sampling included field blank samples. The results for the field QC samples are discussed in the following sections.

### *Field Blanks*

Two field blanks (LW2-W1902 C and LW2-W1902 F) were associated with the XAD resin samples. After qualifiers based on method blank contamination were issued, four results (1.9% of all XAD resin dioxin/furan results) were qualified as not detected (U) based on field blank contamination. The qualifiers were issued to two results for total tetrafurans and one result each for 1,2,3,4,7,8-HxCDD and 2,3,4,7,8-PeCDF.

### *Field Replicate Samples*

No field replicate sample pairs were associated with these XAD resin samples. Precision could not be assessed.

# DATA VALIDATION REPORT
# Portland Harbor RI/FS
# Surface Water High Flow – XAD Samples
# Polycyclic Aromatic Hydrocarbons by Axys Method MLA-021
# Axys Analytical Services, Ltd.

This report documents the review of analytical data from the analyses of filter and XAD resin samples and the associated laboratory and field quality control (QC) samples. Axys Analytical Services, Ltd. of Sidney, British Columbia, Canada, analyzed the samples.

| SDG | No. Samples | Validation Level |
|---|---|---|
| WG18512 | 3 XAD Resins & 1 Field blank,<br>3 Filters & 1 Field blank | Full |

## I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

## II. EDD TO HARDCOPY VERIFICATION

A complete (100%) verification of the electronic data deliverable (EDD) results was performed by comparison to the hardcopy laboratory data package. Laboratory QC results were also verified (10%).

## III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

1 Holding Times and Sample Receipt
Initial Calibration (ICAL)
Continuing Calibration (CCAL)
2 Laboratory Blanks
Field Blanks
2 Labeled Compounds
1 Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
Ongoing Precision and Recovery (OPR)
Field Duplicates
2 Compound Identification
Reporting Limits (MDL and MRL)
1 Calculation Verification (full validation only)

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[1] ***Quality control results are discussed below, but no data were qualified.***
[2] ***Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.***

### Holding Times and Sample Receipt

No technical holding time requirements are established for these XAD extracts and filters. All were extracted within 47 days of collection and analyzed within 40 days of extraction.

## Laboratory Blanks

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times the concentration detected in the blank. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U-7). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Method blanks were analyzed at the appropriate frequency. A summary of contaminant levels, associated samples, and action levels is provided in the data validation worksheets. Various target analytes were detected in the method blanks. However, only the following analytes were qualified as not detected in one or more samples in the associated laboratory data sets: Naphthalene (3 results), 2-methylnaphthalene (4 results), acenaphthylene (4 results), anthracene (4 results), and benz(a)anthracene (1 result)

## Labeled Compounds

Acenaphthene was calculated in the filter samples using phenanthrene d-8 rather than acenaphthylene d-8 due to an apparent "over-recovery" caused by low recovery of the labeled acenaphthylene. Acenaphthene results and reporting limits were estimated (J/UJ-13) in all filter samples.

## Matrix Spikes/Matrix Spike Duplicates

No matrix spike/matrix spike duplicate (MS/MSD) sets were performed with this SDG. Accuracy was assessed using labeled compound recoveries and ongoing precision and recovery (OPR) samples. Precision could not be assessed.

## Compound Identification

The laboratory assigned a "K" qualifier to one or more analytes in all samples to indicate the ion ratio criterion were not met. An outlier indicates that the reported result may be a false positive. Due to this, the results were qualified as not detected (U-21).

## Calculation Verification

A full (Level IV) validation was performed this SDG. No anomalies were identified.

# IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the labeled compound and OPR percent recovery

values, with the exception noted above. No replicate analyses were performed so precision could not be assessed.

Data were qualified as not detected due to ion ratio criteria outliers and contamination in the associated blanks. Data were qualified as estimated due to labeled compound outliers.

All data, as qualified, are acceptable for use.

# DATA VALIDATION REPORT
# Portland Harbor RI/FS
# Surface Water High Flow – XAD Samples
# PCB Congeners
# Method: EPA 1668A
# Axys Analytical Services, Ltd.

This report documents the review of analytical data from the analyses of XAD resin cartridge and filter samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Axys Analytical Services, Ltd. Sidney, British Columbia, Canada.

| SDG | No. Samples | Validation Level |
|---|---|---|
| WG18479 | 3 XAD Resins & 1 Field blank | Summary |
| WG18480 | 3 XAD Filters & 1 Field blank | Full |

## I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

## II. EDD TO HARDCOPY VERIFICATION

A 10% verification of the PCB congener electronic data deliverable (EDD) results was performed by comparison to the hardcopy laboratory data package. Laboratory QC results were also verified (10%).

## III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

1 Holding Times and Sample Receipt
Initial Calibration (ICAL)
Continuing Calibration (CCAL)
2 Laboratory Blanks
2 Field Blanks
Labeled Compounds
1 Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
Ongoing Precision and Recovery (OPR)
1 Field Duplicates
2 Compound Identification
1 Reporting Limits (MDL and MRL)
1 Calculation Verification (full validation only)

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[1] ***Quality control results are discussed below, but no data were qualified.***
[2] ***Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.***

## Holding Times and Sample Receipt

No technical holding time requirements are established for these XAD extracts and filters. All were extracted and analyzed within 1 year of collection.

## Laboratory Blanks

***SDG WG18479:*** Positive values for several analytes were reported in the method blank. Action levels of five times the blank concentrations were established and the sample results were compared to these action levels. Positive results less than the action levels were qualified as not detected (U-7) at the reported concentrations in all samples.

***SDG WG18480:*** Positive values for several analytes were reported in the method blank. Action levels of five times the blank concentrations were established and the sample results were compared to these action levels. Positive results less than the action levels were qualified as not detected (U-7) at the reported concentrations in Sample LW3-W1902 F.

## Field Blanks

***SDG WG18479:*** Sample LW3-W1902 C was submitted for analysis as an equipment blank. Positive values for multiple analytes were remained in this sample after laboratory blank qualifiers were assigned. An action level of five times the blank concentration was established and the sample results were compared to these action level. Positive values less than the action levels were qualified as not detected (U-6) at the reported concentrations in all samples.

***SDG WG18480:*** Sample LW3-W1902 F was submitted for analysis as an equipment blank. Positive values for PCB60, PCB112, and PCB114 remained in this sample after laboratory blank qualifiers were assigned. An action level of five times the blank concentration was established and the sample results were compared to this action level. All sample concentrations were greater than the action levels and no qualifiers were assigned.

## Matrix Spikes/Matrix Spike Duplicates

No matrix spike/matrix spike duplicate (MS/MSD) sets were performed with these SDGs.

## Field Duplicates

No field duplicates were submitted with these SDG.

## Compound Identification

The laboratory assigned a "K" qualifier to one or more analytes in all samples to indicate the ion abundance ratio criteria were not met. Since the ion abundance ratio is the primary identification

criterion for high resolution mass spectroscopy, an outlier indicates that the reported result may be a false positive. Due to this, these results were qualified as not detected (U-21).

No Aroclor patterns were observed in the samples and the laboratory reported default Aroclor identifications. Additionally, values reported for Aroclors were calculated from the concentrations of PCB congeners using a laboratory-derived correction factor, instead of an Aroclor specific response factor. For these reasons all positive Aroclor results were qualified as estimated and tentatively identified (NJ-14).

***SDG WG18479:*** The laboratory noted a lock mass disturbance affecting PCB82 in Sample LW3-W1023 C. The result for this analyte was estimated (J-14) in this sample.

***SDG WG18480:*** The laboratory noted lock mass disturbances affecting PCB82 and PCB148 in Sample LW3-W1024 F. The results and reporting limits for these analytes were estimated (J/UJ-14) in this sample.

### Reporting Limits (MDL and MRL)

***SDG WG18479:*** Some analytes in Samples LW3-W1005 C and LW3-W1024 C were reported from dilution analyses (10x). Reporting limits were adjusted accordingly.

***SDG WG18480:*** All samples were reported from dilutions (3x) and some analyte results from samples LW3-W1005-F, LW3-W1023F, and LW3-W1902 F were reported from further dilutions (10x). Reporting limits were adjusted accordingly.

### Calculation Verification

***SDG WG18480:*** A full (Level IV) validation was performed this SDG. No anomalies were identified.

## IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the labeled compound and OPR percent recovery values, with the exceptions noted above. Precision was not measured by the laboratory and could not be assessed.

Data were qualified as estimated due to lock mass disturbances. Data were qualified as not detected due to ion ratio criteria outliers and contamination from the associated laboratory and field blanks. Aroclor data were qualified as tentatively identified at estimated concentrations.

All data, as qualified, are acceptable for use.

# DATA VALIDATION REPORT
# Portland Harbor RI/FS
# Surface Water High Flow – XAD Samples
# Chlorinated Pesticides by Axys Method MLA-028
# Axys Analytical Services, Ltd.

This report documents the review of analytical data from the analyses of filter and XAD resin samples and the associated laboratory and field quality control (QC) samples. Axys Analytical Services, Ltd., Sidney, British Columbia, Canada, analyzed the samples.

| SDG | No. Samples | Validation Level |
|---|---|---|
| WG18510 | 3 XAD Resins & 1 Field blank,<br>3 Filters & 1 Field blank | Full |

## I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables, with the exception noted below. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

The summaries and raw data for the calculation of endrin breakdown were not included in the data package. The laboratory was contacted and the necessary information was provided.

## II. EDD TO HARDCOPY VERIFICATION

A complete (100%) verification of the electronic data deliverable (EDD) results was performed by comparison to the hardcopy laboratory data package. Laboratory QC results were also verified (10%).

## III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

1 Holding Times and Sample Receipt
2 Initial Calibration (ICAL)
2 Continuing Calibration (CCAL)
2 Laboratory Blanks
Field Blanks
2 Labeled Compounds
1 Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
2 Ongoing Precision and Recovery (OPR)
1 Field Duplicates
DDT/Endrin Breakdown
2 Compound Identification
1 Reporting Limits (MDL and MRL)
1 Calculation Verification (full validation only)

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[1] ***Quality control results are discussed below, but no data were qualified.***
[2] ***Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.***

## Holding Times and Sample Receipt

No technical holding time requirements are established for extraction of these XAD resins and filters. All were extracted within 47 days of collection and analyzed within 40 days of extraction.

## Initial Calibration

All percent relative standard deviation (%RSD) values were within the control limits.

The analyte hexachlorobutadiene was not included in the standards used for calibration. The values reported of this analyte were based on the response of 1,2,4-trichlorobenzene and an experimentally determined ratio of the response of hexachlorobutadiene to that of 1,2,4-trichlorobenzene. All values and/or reporting limits for hexachlorobutadiene were estimated (U/UJ-5A).

## Continuing Calibration

All percent difference (%D) values were within the control limit of ±25%.

The analyte hexachlorobutadiene was not included in the continuing calibration standards. All values and/or reporting limits for hexachlorobutadiene were estimated (U/UJ-5B).

## Laboratory Blanks

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times the concentration detected in the blank. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U-7). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Method blanks were analyzed at the appropriate frequency. A summary of contaminant levels, associated samples, and action levels is provided in the data validation worksheets. Various target analytes were detected in the method blanks. However, only the following analytes were qualified as not detected in one or more samples in the associated laboratory data set: Hexachlorobutadiene (2 results) & hexachlorobenzene (2 results)

## Labeled Compounds

The percent recovery (%R) values for 13C-1,2,3-trichlorobenzene were less than the lower control limit in seven samples, and less than 10% in Sample LW3-W1023C. The quantitation of hexachlorobutadiene is associated with this labeled compound. When the %R value was less than the lower control limit but greater than 10%, positive values and reporting limits for hexachlorobutadiene were estimated (J/UJ-13). When the %R value was below 10%, the reporting limit for hexachlorobutadiene was rejected (R-10).

## Matrix Spikes/Matrix Spike Duplicates

No matrix spike/matrix spike duplicate (MS/MSD) sets were performed with this SDG. Accuracy was assessed using labeled compound recoveries and ongoing precision and recovery (OPR) samples. Precision could not be assessed.

## Ongoing Precision and Recovery

The following labeled compounds and their associated native analytes were not quantifiable by the laboratory in the OPR sample associated with the four filter samples: beta-endosulfan, endrin, endrin aldehyde, endrin ketone, endosulfan sulfate and methoxychlor. Positive values for these analytes were estimated (J-10) and reporting limits were rejected (R-10) in all four filter samples. Hexachlorobutadiene was not included in the OPR sample. Positive values and/or reporting limits for this analyte were estimated (J/UJ-10) in all samples.

## Compound Identification

The laboratory assigned a "K" flag to one or more analytes in all samples to indicate the ion ratio criterion were not met. Since the ion abundance ratio is the primary identification criterion for high resolution mass spectroscopy, an outlier indicates that the reported result may be a false positive. Due to this, these results were qualified as not detected (U-21) at the reported concentration.

## Reporting Limits (MDL and MRL)

The analytical concentration goals (ACG) of 0.6 pg/L to 3.0 pg/L were met for all analytes except endosulfan sulfate and endrin aldehyde. No action was taken on this basis.

Additionally, toxaphene was listed as a target analyte in the QAPP, but no toxaphene analysis was performed.

## Calculation Verification

A full (Level IV) validation was performed this SDG. No anomalies were identified.

# IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the labeled compound and OPR %R values, with the exceptions noted above. No replicate analyses were performed so precision could not be assessed.

Data were qualified as estimated due to an analyte not being included in the calibration standards and OPR. Data were qualified as not detected due to ion ratio criteria outliers and contamination in the associated blanks.

Data were rejected due to non-recovery of several analytes from the OPR. Rejected data is not usable.

All other data, as qualified, are acceptable for use.

# DATA VALIDATION REPORT
# Portland Harbor RI/FS
# Surface Water High Flow – XAD Samples
# Dioxin & Furan Compounds
# Method: EPA 1613B
# Axys Analytical Services, Ltd.

This report documents the review of analytical data from the analyses of XAD filter and resin samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Axys Analytical Services, Ltd. of Sidney, British Columbia, Canada.

| SDG | No. Samples | Validation Level |
|---|---|---|
| WG18511 | 3 Filters, 3 XAD Resins & 2 Field blanks | Summary |

## I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

## II. EDD TO HARDCOPY VERIFICATION

A complete (100%) verification of the electronic data deliverable (EDD) results was performed by comparison to the hardcopy laboratory data package. Laboratory QC results were also verified (10%).

## III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

1 Holding Times and Sample Receipt
Initial Calibration (ICAL)
Continuing Calibration (CCAL)
2 Laboratory Blanks
2 Field Blanks
Labeled Compounds
1 Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
Ongoing Precision and Recovery (OPR)
1 Field Duplicates
2 Compound Identification
Reporting Limits (MDL and MRL)
Calculation Verification (full validation only)

[1] ***Quality control results are discussed below, but no data were qualified.***
[2] ***Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.***

### Holding Times and Sample Receipt

No technical holding time requirements are established for extraction of these XAD resins and filters, although the QAPP notes contractual holding times of 30 days from collection to extraction

and 30 days from extraction to analysis. All samples were extracted within 50 days of collection and analyzed within 40 days of extraction.

## Laboratory Blanks

***SDG WG18511:*** Positive results for several analytes were reported in the method blanks. Action levels of five times these blank concentrations were established and the sample results were compared to these action levels. Sample results less than the action levels were qualified as not detected (U-7) at the reported concentrations.

## Field Blanks

***SDG WG18511:*** Two field blanks were submitted. Positive values for 1,2,3,4,7,8-HxCDD, 2,3,4,7,8-PeCDF, total hexadioxins, total tetrafurans, and total pentafurans remained in Sample LW2-W1902 C after laboratory blank qualifiers were assigned. Action levels of five times the blank concentration were established and the associated sample results were compared to these action levels. Associated sample results less than the action levels were qualified as not detected (U-6) at the reported concentrations.

No positive results were reported in Sample LW2-W1902 F.

## Matrix Spikes/Matrix Spike Duplicates

No matrix spike/matrix spike duplicate (MS/MSD) sets were performed with these SDGs. Accuracy and precision were assessed using labeled compound recoveries, ongoing precision and recovery (OPR) samples, and field duplicate samples.

## Field Duplicates

No field duplicates were submitted.

## Compound Identification

The laboratory assigned a" K" qualifier to one or more analytes in all samples to indicate the ion ratio criterion were not met. Since the ion abundance ratio is the primary identification criterion for high resolution mass spectroscopy, an outlier indicates that the reported result may be a false positive. Due to this, the results were qualified as not detected (U-21).

All results for 2,3,7,8-TCDF were confirmed on a DB-225 column as required by the method. The 2,3,7,8-TCDF results on the DB-5 column were qualified as do-not-report (DNR-11), and results from the DB-225 column were reported instead.

## IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the labeled compound and OPR percent recovery values. Precision was not evaluated.

Data were qualified as not detected due to ion ratio criteria outliers and contamination from the associated blanks. Data were qualified as do-not-report to indicate which result (of duplicate results) should be used.

Data qualified as do-not-report should not be used for any purpose. All other data, as qualified, are acceptable for use.