EcoChem, INC.
Environmental Data Quality

DATA QUALITY EVALUATION

PORTLAND HARBOR

Round 3A Surface Water – High Flow Event, 2007

XAD Sampling

Polycyclic Aromatic Hydrocarbons (PAH) – Axys Method MLA-021
Chlorinated Pesticides - Axys Method MLA-028
Polychlorinated Biphenyl (PCB) Congeners – Axys Method MLA-010 (EPA 1668A)
Dioxin/Furan Compounds – Axys Method MLA-017 (EPA 1613B)

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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* (Integral, 10/4/04); *Supplement 1 to QAPP Addendum 1: Round 3A Surface Water Sampling* (Integral, 8/11/06); and *National Functional Guidelines for Organic Data Review* (USEPA 1999). 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**. Findings for each QC element are discussed in the data validation reports, which 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 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 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 Integral project database.

SUMMARY OF DATA VALIDATION: POLYCYCLIC AROMATIC HYDROCARBONS

Twenty-two (22) XAD resin samples and 22 filter samples were analyzed for polycyclic aromatic hydrocarbon (PAH) compounds for the Portland Harbor Surface Water High Flow sampling event. Four rinsate blanks were collected to monitor the field collection and filtration processes.

The PAH data for the XAD samples were generally acceptable. No data points were rejected. Six data points (0.7% of all PAH results) were qualified as estimated because control limits were exceeded in one or more laboratory quality control (QC) samples or procedures.

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 PAH 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 PAH analyses of the XAD 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.

Laboratory Blank Analyses

Laboratory (method) blanks were analyzed at the appropriate frequency. Seventy-three results (8.9% of all XAD PAH results) were qualified as not detected (U) based on laboratory blank contamination. Four further results (0.5%) were qualified as estimated (J) based on potential contamination from a compromised laboratory blank.

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).

Labeled Compound Recovery

Labeled compounds were added to all field and QC samples. All recovery values met the criteria for acceptable performance.

Matrix Spike Recovery

Matrix spike and matrix spike duplicate analyses were not performed. Accuracy was assessed using labeled compound and OPR analyses.

Ongoing Precision and Recovery Sample Recovery

OPR analyses met the criteria for frequency of analysis. Six PAH results and reporting limits (0.7% of all XAD PAH results) were qualified as estimated (J or UJ) for potential low bias due to low OPR recovery values.

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

The reported detection limits are based on the method detection limit (MDL), adjusted for sample size and dilution. These detection limits ranged from 0.0123 ng/L to 0.783 ng/L for the non-detected results. The laboratory reporting limits generally met the target MRL stated in *Round 2 QAPP, Addendum 1: Surface Water*, except when dilutions were required or limits were raised to due interferences.

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. A total of 131 results (16.1% of all XAD 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 Surface Water High Flow Sampling included field blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Four field blanks (LW3-W4900-C and LW3-W4901-C, LW3-W4900-F and LW3-W4901-F) were associated with the XAD samples. After qualifiers based on laboratory blank contamination were issued, 73 results (8.9% of all XAD PAH results) were qualified as not detected (U) based on field blank contamination.

SUMMARY OF DATA VALIDATION: PESTICIDE COMPOUNDS

Twenty-six (26) XAD resin samples and 26 filter samples were analyzed for pesticide compounds for the Portland Harbor Surface Water High Flow sampling event. Four rinsate blanks were collected to monitor the field collection and filtration processes.

The pesticide data for the XAD samples were generally acceptable. Three pesticide data points (0.2% of all pesticide results) 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. Another 74 data points (4.7% of all pesticide results) 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 greater than 99% complete for the XAD pesticide analyses.

No results were reported for toxaphene, although it was originally designated as a target analyte in the QAPP for this project. This was documented in previous surface water validation reports.

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 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.

A total of 53 hexachlorobutadiene results (3.4% of all XAD 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. The recoveries from these mixtures met all acceptance criteria.

Laboratory Blank Analyses

Laboratory (method) blanks were analyzed at the appropriate frequency. Eleven pesticide results were qualified as not detected (U) based on method blank contamination (0.7% of all XAD pesticide results).

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).

Labeled Compound Recovery

Labeled compounds were added to all field and QC samples. Three reporting limits for hexachlorobutadiene (0.2% of all XAD pesticide results) were rejected due to extremely low labeled compound recovery. Seventeen (17) more pesticide results and reporting limits (1.1% of all XAD pesticide results) were qualified as estimated (J or UJ) for potential low bias due to low labeled compound recovery values.

Matrix Spike Recovery

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 Recovery

OPR analyses met the criteria for frequency of analysis. A total of 24 pesticide results and reporting limits (1.5% of all XAD pesticide results) were qualified as estimated (J or UJ) for OPR outliers.

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

The reported detection limits are based on the method detection limit (MDL), adjusted for sample size and dilution. These detection limits ranged from 0.0935 ng/L to 0.142 ng/L for the non-detected results.

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. A total of 258 results (16.5% of all XAD 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 Surface Water High Flow Sampling included field blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Four field blanks (LW3-W4900-C and LW3-W4901-C, LW3-W4900-F and LW3-W4901-F) were associated with the XAD samples. After qualifiers based on laboratory blank contamination were issued, 18 results (1.1% of all XAD pesticide results) were qualified as not detected (U) based on field blank contamination.

SUMMARY OF DATA VALIDATION: PCB CONGENERS

Forty (40) XAD resin samples and 40 filter samples were analyzed for PCB congener compounds for the Portland Harbor Surface Water High Flow sampling event. Four rinsate blanks were collected to monitor the field collection and filtration processes.

The PCB data for the XAD samples were generally acceptable. Thirty-seven (37) PCB data points (0.27% of all PCB results) 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. Sixty-nine (69) data points (0.50% of all PCB results) were qualified as estimated and 228 data points (1.6% of all PCB results) were qualified as tentatively identified because requirements were not met in one or more laboratory procedures. 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 greater than 99% complete for the XAD 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 PCB analyses of the XAD 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.

Laboratory Blank Analyses

Laboratory (method) blanks were analyzed at the appropriate frequency. A total of 83 results (0.60% of all XAD 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]).

Labeled Compound Recovery

Labeled compounds were added to all field and QC samples. Thirty-five (35) results (0.25% of all XAD PCB results) were rejected due to extremely low labeled compound recovery. A further 24 results (0.17% of all XAD PCB results) were qualified as estimated (J/UJ) due to low labeled compound recovery.

Matrix Spike Recovery

Matrix and duplicate matrix spike analyses were not performed. Accuracy was assessed using the labeled compound and OPR analyses.

Ongoing Precision and Recovery Sample Recovery

OPR analyses met the criteria for frequency of analysis, except for two samples which were analyzed using remaining pesticide extract fractions, without PCB spikes.

The recovery values 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

The reported detection limits are based on the method detection limit (MDL), adjusted for sample size and dilution. These detection limits ranged from 0.0010 to 0.783 pg/L for the non-detected results. The laboratory reporting limits generally met the target MRL stated in *Round 2 QAPP, Addendum 1: Surface Water*.

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. Results flagged with “K” by the laboratory were qualified as not detected (U) to make this relationship clear to the data user. A total of 1,823 results (13% of all XAD PCB results) were qualified as not detected because the ion abundance ratio criteria were not met.

A "G" flag indicates that samples contained interferences that affected the quantitation of some compounds. The results for these compounds were qualified as estimated (J/UJ). A total of 45 data points (0.32% of all XAD PCB results) were qualified as estimated because of these interferences.

Two data points (0.01% of all XAD PCB results) were rejected because the laboratory was not able to provide data due to interferences.

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 228 positive Aroclor results (1.6% of all XAD 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 Surface Water High Flow Sampling included field blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Four field blanks (LW3-W4900-C and LW3-W4901-C, LW3-W4900-F and LW3-W4901-F) were associated with the XAD samples. After qualifiers based on laboratory blank contamination were issued, 355 results (2.5% of all XAD PCB results) were qualified as not detected (U) based on field blank contamination.

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 (EPA1613); *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

Twenty-two (22) XAD resin samples and 22 filter samples were analyzed for dioxin and furan compounds for the Portland Harbor Surface Water High Flow sampling event. Four rinsate blanks were collected to monitor the field collection and filtration processes.

The dioxin/furan data for the XAD samples were generally acceptable. No data points were rejected. Eleven data points (0.9% of all XAD dioxin/furan data points) were qualified as estimated because control limits were exceeded in one or more laboratory QC samples or procedures. 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 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 dioxin/furan analyses.

A total of 48 results for 2,3,7,8-TCDF quantitated from the DB-5 column were marked as do-not-report (DNR) in favor of results quantitated from the DB-225 column. These are considered as duplicate results, and do not affect the completeness of the data set.

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 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.

Laboratory Blank Analyses

Laboratory (method) blanks were analyzed at the appropriate frequency. A total of 175 results (14.0% of all XAD dioxin/furan 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].

Labeled Compound Recovery

Labeled compounds were added to all field and QC samples. One result (0.1% of all XAD dioxin results) was qualified as estimated (J) due to high labeled compound recovery.

Matrix Spike Recovery

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 Recovery

OPR analyses met the criteria for frequency of analysis. The recovery values 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

The reported detection limits are based on the method detection limit (MDL), adjusted for sample size and dilution. These detection limits ranged from 0.0010 pg/L to 0.0257 pg/L for the non-detected results, and generally met the target MDL stated in *Round 2 QAPP, Addendum 1: Surface Water*.

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. A total of 169 data points (13.5% of all XAD dioxin/furan data points) were qualified as not detected because the ion abundance ratio criteria were not met.

The laboratory also noted that samples contained chemical or lock mass interferences that affected the quantitation of some compounds. The results for these compounds were qualified as estimated (J/UJ). Ten data points (0.8% of all XAD dioxin/furan results) were qualified as estimated because of these interferences.

Field Quality Control Samples

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

Field Blanks

Four field blanks (LW3-W4900-C and LW3-W4901-C, LW3-W4900-F and LW3-W4901-F) were associated with the XAD samples. After qualifiers based on laboratory blank contamination were issued, 165 results (13.2% of all XAD dioxin/furan data points) were qualified as not detected for field blank contamination.

EcoChem, INC.
Environmental Data Quality

APPENDIX A

DATA QUALIFIER DEFINITIONS, REASON CODES, AND CRITERIA TABLES

DATA VALIDATION QUALIFIER CODES
National Functional Guidelines

The following definitions provide brief explanations of the qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents the approximate concentration.
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.
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.

The following is an EcoChem qualifier that may also be assigned during the data review process:

DNR	Do not report; a more appropriate result is reported from another analysis or dilution.

DATA QUALIFIER REASON CODES

1	Holding Time/Sample Preservation
2	Chromatographic pattern in sample does not match pattern of calibration standard.
3	Compound Confirmation
4	Tentatively Identified Compound (TIC) (associated with NJ only)
5A	Calibration (initial)
5B	Calibration (continuing)
6	Field Blank Contamination
7	Lab Blank Contamination (e.g., method blank, instrument, etc.)
8	Matrix Spike(MS & MSD) Recoveries
9	Precision (all replicates)
10	Laboratory Control Sample Recoveries
11	A more appropriate result is reported (associated with “R” and “DNR” only)
12	Reference Material
13	Surrogate Spike Recoveries (a.k.a., labeled compounds & recovery standards)
14	Other (define in validation report)
15	GFAA Post Digestion Spike Recoveries
16	ICP Serial Dilution % Difference
17	ICP Interference Check Standard Recovery
18	Trip Blank Contamination
19	Internal Standard Performance (e.g., area, retention time, recovery)
20	Linear Range Exceeded
21	Potential False Positives

Integral - Portland Harbor Site
Pesticides/PCBs/Herbicides/Phenols by GC/ECD (Based on Organic NFG 1999)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler/Storage Temperature	Aqueous/Soil/Sediment <4°C Tissues <-10°C	J(+)/UJ(-) if greater than 6 deg. C (EcoChem PJ)	1
Holding Time	Water: 7 days from collection Soil: 14 days from collection Analysis: 40 days from extraction	J(+)/UJ(-) if ext/analyzed > HT J(+)/R(-) if ext/analyzed > 3X HT (EcoChem PJ)	1
Resolution Check	Beginning of ICAL Sequence Within RTW Resolution >90%	Narrate (Use Professional Judgement to qualify)	14
Instrument Performance (Breakdown)	DDT Breakdown: < 20% Endrin Breakdown: <20% Combined Breakdown: <30% Compounds within RTW	J(+) DDT NJ(+) DDD and/or DDE R(-) DDT - If (+) for either DDE or DDD J(+) Endrin NJ(+) EK and/or EA R(-) Endrin - If (+) for either EK or EA	5A
Retention Times	Surrogates: TCX (+/- 0.05); DCB (+/- 0.10) Target compounds: elute before heptachlor epoxide (+/- 0.05) elute after heptachlor epoxide (+/- 0.07)	NJ(+)/R(-) results for analytes with RT shifts For full DV, use PJ based on examination of raw data	5B
Initial Calibration	Pesticides: Low=QL, Mid=4X, High=16X Multiresponse - one point Calibration %RSD<20% %RSD<30% for surr; two comp. may exceed if <30% Resolution in Mix A and Mix B >90%	J(+)/UJ(-)	5A
Continuing Calibration	Alternating PEM standard and INDA/INDB standards every 12 hours (each preceeded by an inst. Blank) %D < 25% Resolution >90% in IND mixes; 100% for PEM	J(+)/UJ(-) J(+)R(-) if %D > 90% PJ for resolution	5B
Method Blank	One per matrix per batch No results > QL	U(+) if sample result is < QL and < 5X rule (raise sample value to QL)	7
		U(+) if sample result is > or equal to QL and < 5X rule (at reported sample value)	7
Instrument Blanks	Analyzed at the beginning of every 12 hour sequence No analyte > 1/2 QL	Same as Method Blank	7
Field Blanks	No results > QL	Apply 5X rule; U(+) < action level	6

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Integral - Portland Harbor Site
Pesticides/PCBs/Herbicides/Phenols by GC/ECD (Based on Organic NFG 1999)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
MS/MSD (recovery)	One set per matrix per batch Method Acceptance Criteria	Qualify parent only unless other QC indicates systematic problems: J(+) if both %R > UCL J(+)/UJ(-) if both %R < LCL J(+)/R(-) if both %R < 10% PJ if only one %R outlier	8
MS/MSD (RPD)	One set per matrix per batch Method Acceptance Criteria	J(+) if RPD > CL	9
Lab Duplicate	Results >5X reporting limit (RL): RPD criteria: use QAPP limits, all matrices Results <5X RL: Solid: Absolute difference < 2X RL Aqueous: Absolute difference < 1X RL	J(+)/UJ(-) if outside limits	9
LCS/LCSD *(if required)*	One set per matrix and batch of 20 samples RPD < 35%	J(+) assoc. cmpd. in all samples	9
Surrogates	TCX and DCB added to every sample %R = 30-150%	J(+)/UJ(-) if both %R = 10 - 60% J(+) if both >150% J(+)/R(-) if any %R <10%	13
Quantitation/ Identification	Analyte within RTW on both columns Quantitated using CCV or ICAL CF Lowest value from either column reported RPD between columns (25%)	J(+) if RPD = 25-60% (Pest/Aroclor); 40-60% (Herb/Phenol) NJ(+) using PJ if RPD > 60%	3
Two analyses for one sample	Report only one result per analyte	"DNR" results that should not be used to avoid reporting two results for one sample	11
Sample Clean-up	GPC required for soil samples Florisil required for all samples Sulfur is optional Clean-up standard check %R within CLP limits	J(+)/UJ(-) if %R < LCL J(+) if %R > UCL	14
Field Duplicates	Results >5X reporting limit (RL): RPD < 50% (all matrices, QAPP specified) Results <5X RL: Solid: Absolute difference < 2X RL Aqueous: Absolute difference < 1X RL	Narrate; do not qualify	na

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Integral - Portland Harbor Site
Compounds Analyzed By HRMS (Methods 1613B or SW846 - 8290)
Polycyclic Aromatic Hydrocarbons (PAH), PCB Congeners and Dioxins/Furans

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler/Storage Temperature	Aqueous/Soil/Sediment <4°C Tissues <-10°C	EcoChem PJ, see TM-05	1
Holding Time	Water: 30 days from collection Soil: 30 days from collection Analysis: 40 days from extraction Note: Under CWA, SDWA, and RCRA the HT for H2O is 7 days	J(+)/UJ(-) if ext > 30 days J(+)/UJ(-) if analysis > 40 Days EcoChem PJ, see TM-05	1
Mass Resolution	>=10,000 resolving power at m/z 304.9824 Exact mass of m/z 380.9760 w/in 5 ppm of theoretical value (380.97410 to 380.97790) . Analyzed prior to ICAL and at the start and end of each 12 hr. shift	R(+/-) if not met	14
Window Defining Mix and Column Performance Mix	Window defining mixture/Isomer specificity std run before ICAL and CCAL Valley < 25% (valley = (x/y)*100%) x = ht. of TCDD y = baseline to bottom of valley For all isomers eluting near 2378-TCDD/TCDF isomers (TCDD only for 8290)	J(+) if valley > 25%	5A (ICAL) 5B (CCAL
Initial Calibration	ICAL: Minimum of five standards %RSD < 20% for native compounds %RSD <30% for labeled compounds (%RSD <35% for labeled compounds under 1613b)	J(+) natives if %RSD > 20%	5A
	Abs. RT of $^{13}C_{12}$-1234-TCDD >25 min on DB5 >15 min on DB-225	EcoChem PJ, see TM-05	
	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	EcoChem PJ, see TM-05	
	S/N ratio > 10 for all native and labeled compounds in CS1 std.	If <10, elevate Det. Limit or R(-)	

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Integral - Portland Harbor Site
Compounds Analyzed By HRMS (Methods 1613B or SW846 - 8290)
Polycyclic Aromatic Hydrocarbons (PAH), PCB Congeners and Dioxins/Furans

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Continuing Calibration	Analyzed at the start and end of each 12 hour shift. %D+/-20% for native compounds %D +/-30% for labeled compounds (Must meet limits in Table 6 for 1613B) (If %D in the closing CCAL are w/in 25%/35%, the avg RF from the 2 CCAL may be used to calculate samples per Section 8.3.2.4 of 8290)	Do not qualify labeled compounds. Narrate in report for labeled compound %D outliers. For native compound %D outliers: *Method 8290:* J(+)/UJ(-) if %D = 20% - 75% J(+)/R(-) if %D > 75% *Method 1613:* J(+)/UJ(-) if %D is outside Table 6 limits J(+)/R(-) if %D is +/- 75% of Table 6 limit	5B
	Abs. RT of $^{13}C_{12}$-1234-TCDD and ^{13}C12-123789-HxCDD +/- 15 sec of ICAL.	EcoChem PJ, see ICAL section of TM-05	
	RRT of all other compounds must meet table 2 of 1613B.	EcoChem PJ, see TM-05	
	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	EcoChem PJ, see TM-05	
	S/N ratio > 10	If <10, elevate Det. Limit or R(-)	
Method Blank	One per matrix per batch No positive results	If sample result <5X action level, qualify U at reported value. (<10X for phthalates)	7
Field Blanks	No results > QL	Apply 5X rule; U(+) < action level	6
LCS / OPR	Concentrations must meet limits in Table 6 of method 1613B or lab limits.	J(+) if %R > UCL J(+)/UJ(-) if %R < LCL J(+)/R(-) using PJ if %R <<LCL (< 10%)	10
MS/MSD (recovery)	May not analyze MS/MSD %R should meet lab limits.	Qualify parent only unless other QC indicates systematic problems: J(+) if both %R > UCL J(+)/UJ(-) if both %R < LCL J(+)/R(-) if both %R < 10% PJ if only one %R outlier	8

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Integral - Portland Harbor Site
Compounds Analyzed By HRMS (Methods 1613B or SW846 - 8290)
Polycyclic Aromatic Hydrocarbons (PAH), PCB Congeners and Dioxins/Furans

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
MS/MSD (RPD)	May not analyze MS/MSD RPD < 20%	J(+) if RPD > CL	9
Lab Duplicate	RPD <25% if present.	J(+) if outside limts	9
Labeled Compounds / Internal Standards	*Method 8290:* %R = 40% - 135% in all samples	J(+)/UJ(-) if %R = 10% to LCL J(+) if %R > UCL J(+)/R(-) if %R < 10%	13
	Method 1613B: %R must meet limits specified inTable 7		
Quantitation/ Identification	Ions for analyte, IS, and rec. std. must max w/in 2 sec. S/N >2.5 IA ratios meet limits in Table 9 of 1613B or Table 8 of 8290 RRTs w/in limits in table 2 of 1613B	If RT criteria not met, use PJ (see TM-05) If S/N criteria not met, J(+). if unlabelled ion abundance not met, change to EMPC If labelled ion abundance not met, J(+).	21
EMPC (estimated maximum possible concentration)	If quantitation identification criteria are not met, laboratory should report an EMPC value.	If laboratory correctly reported an EMPC value, qualify with U to indicate that the value is a detection limit.	14
Interferences	PCDF interferences from PCDPE	If both detected, change PCDF result to EMPC	14
Second Column Confirmation	All 2378-TCDF hits must be confirmed on a DB-225 column (or equiv). All QC specs in this table must be met for the confirmation analysis.	Report lower of the two values. If not performed use PJ (see TM-05).	3
Field Duplicates	QAPP specified RPD < 50% (all matrices)	Narrate; do not qualify	na
Two analyses for one sample	Report only one result per analyte	"DNR" results that should not be used	11

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Environmental Data Quality

APPENDIX B
DATA VALIDATION REPORTS

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3A Surface Water – High Flow Event (57C)
Polycyclic Aromatic Hydrocarbons by Axys Method MLA-021
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. Axys Analytical Services, Ltd. of Sidney, British Columbia, Canada, analyzed the samples.

SDG	Number of Samples	Validation Level
WG21532	4 XAD Filters & 2 Field Blanks	Summary
WG21558	4 XAD Cartridges & 2 Field Blanks	Full
WG21777	12 XAD Filters	Summary
WG21909	9 XAD Cartridges	Summary
WG21918	6 XAD Filters	Summary
WG21921	9 XAD Cartridges	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%).

SDG WG21558: The (co-eluting) analytes benzo(j+k)fluoranthenes were listed in the EDD file as benzo(k)fluoroanthene for this SDG only. The EDD file was corrected to match all other SDG.

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
2 Labeled Compounds
1 Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
Ongoing Precision and Recovery (OPR)
2 Compound Identification
Reporting Limits (MDL and MRL)
1 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 were extracted within 40 days of collection and analyzed within 25 days of extraction. No holding time qualifiers were applied.

SDG WG21532 & WG21558: The sample cooler was received above the recommended temperature range of 4°C ±2°, at 7°C. It was determined that this outlier did not impact data quality and no qualifiers were required. The chain of custody (COC) form was not signed by the sampler to relinquish custody of the samples. It was correctly signed by the laboratory to indicate sample receipt.

SDG WG21777, WG21909, WG21918, WG21921: The sample coolers were received between 3°C and 8°C, some were above the recommended temperature range of 4°C ±2°. Specific cooler temperatures were not listed. It was determined that the outliers did not impact data quality and no qualifiers were required.

SDG WG21777: Sample LW3-W4023-W-F was mislabeled as LW3-W023-W-F (omitting the '4'). The laboratory corrected this sample identification prior to analysis.

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. The following analyte results were qualified as not detected in one or more samples in the associated SDG.

SDG WG21532: Phenanthrene (5 results), anthracene (4 results), chrysene (1 result), 2-methylnaphthalene (5 results).

SDG WG21777: Acenaphthylene (12 results), anthracene (12 results), fluorene (12 results), 2-methylnaphthalene (2 results).

SDG WG21909: Benz(a)anthracene (3 results).

SDG WG21918: Naphthalene (1 result), acenaphthylene (6 results), fluorene (4 results), anthracene (4 results), 2-methylnaphthalene (1 result).

SDG WG21921: Naphthalene (1 result).

Field Blanks

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. If a contaminant is reported in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U-6).

SDG WG21532: Two field blanks (LW3-W4900-F and LW3-W4901-F) were reported, and associated with all filter samples. Positive results for fluoroanthene, naphthalene, and pyrene were reported in Samples LW3-W4900-F, and for fluoranthene and naphthalene in Sample LW3-W4901-F. Four results each for naphthalene, fluoroanthene, and pyrene were qualified as not detected (U-6) in this SDG based on field blank contamination.

The following results were also qualified as not detected due to field blank contamination in one or more filter samples.

SDG WG21777: Twelve results each for naphthalene and pyrene, and three results for fluoroanthene were qualified as not detected (U-6) based on field blank contamination.

SDG WG21918: Six results for naphthalene and four results for pyrene were qualified as not detected (U-6) based on field blank contamination.

SDG WG21558: Two field blanks (LW3-W4900-C and LW3-W4901-C) were reported, and associated with all cartridge samples. Positive results were detected for naphthalene, phenanthrene, and 2-methylnaphthalene in Sample LW3-W4900-C, and for naphthalene, pyrene, and 2-methylnaphthalene in Sample LW3-W4901-C. Four results for naphthalene and two results for pyrene were qualified as not detected (U-6) in this SDG based on field blank contamination.

The following results were also qualified as not detected due to field blank contamination in one or more cartridge samples.

SDG WG21909, WG21921: Eighteen results for naphthalene were qualified as not detected (U-6) based on field blank contamination.

Labeled Compounds

SDG WG21918: The percent recovery (%R) of labeled benzo(a)pyrene from laboratory blank WG21918-101 was less than the amount necessary to quantify benzo(a)pyrene. Since this analyte was flagged as non-quantifiable, there is no way to establish possible contamination of benzo(a)pyrene in the blank. Therefore, associated results for benzo(a)pyrene which were less than five times the quantitation limit were qualified as estimated (J-14) in case of potential contamination.

Matrix Spikes/Matrix Spike Duplicates

No matrix spike/matrix spike duplicate or laboratory duplicate analyses were performed. Accuracy was assessed using labeled compound recoveries and ongoing precision and recovery samples (OPR). Precision could not be assessed.

Ongoing Precision and Recovery

SDG WG21918: The %R value for anthracene was less than lower control limits from the OPR. Results and reporting limits for anthracene were qualified as estimated (J/UJ-10) in the associated 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 mass spectroscopy, an outlier indicates that the reported result may be a false positive. Therefore, these results were qualified as not detected (U-21) at the reported concentration.

It was noted by the laboratory that chrysene may co-elute with triphenylene and that dibenz(a,h)anthracene may co-elute with dibenz(a,c)anthracene.

Calculation Verification

SDG WG21558: Calculation verifications were performed on this SDG. No calculation errors were found.

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. No replicate analyses were performed so precision could not be assessed.

Data were qualified as not detected due to ion ratio criteria outliers, contamination in the associated laboratory and field blanks, and potential benzo(a)pyrene contamination from the laboratory blank. Data were qualified as estimated because of an OPR %R outlier.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3A Surface Water – High Flow Event (57C)
Chlorinated Pesticides by Axys Method MLA-028
Axys Analytical Services, Ltd.

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

SDG	Number of Samples	Validation Level
WG21544	4 XAD Filters & 2 Field Blanks	Full
WG21586	4 XAD Columns & 2 Field Blanks	Summary
WG21776	12 XAD Filters	Summary
WG21824	9 XAD Columns	Summary
WG21917	6 XAD Filters	Summary
WG21920	9 XAD Columns	Summary
WG22031	4 XAD Filters	Summary
WG22063	4 XAD Columns	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%).

SDG WG22031: Sample LW3-W4031-NB-F was labeled LW3-W4031-F-NB and Sample LW3-W4031-NS-F was labeled LW3-W4031-F-NS on the Chain of Custody (COC). These discrepancies were noted in the laboratory case narrative and corrected in the EDD.

SDG WG22063: Sample LW3-W4031-NB-C was labeled LW3-W4031-C-NB and Sample LW3-W4031-NS-C was labeled LW3-W4031-C-NS on the COC. This sample did not have the collection time listed. The COC listed the sample collection time as 12:08. These discrepancies were noted in the laboratory case narrative and corrected in the EDD.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

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

[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 56 days of collection and analyzed within 33 days of extraction.

SDG WG21544: The sample cooler was received above the recommended temperature range of 4°C ±2°, at 7°C. It was determined that this outlier did not impact data quality and no qualifiers were required. The chain of custody (COC) form was not signed by the sampler to relinquish custody of the samples. It was correctly signed by the laboratory to indicate sample receipt.

SDG WG21776, WG21824, WG21917, WG21920, WG22031, WG22063: The sample coolers were received between 3°C and 8°C, some were above the recommended temperature range of 4°C ±2°. Specific cooler temperatures were not listed. It was determined that the outliers did not impact data quality and no qualifiers were required.

Initial Calibration

The analyte hexachlorobutadiene was not included in the standards used for calibration. The results for this analyte were calculated using the response of 1,2,4-trichlorobenzene and an experimentally determined ratio of the response of hexachlorobutadiene to 1,2,4-trichlorobenzene. All results and reporting limits for hexachlorobutadiene were qualified as estimated (U/UJ-5A).

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

SDG WG21920: The initial calibration curve for endrin aldehyde exceeded the %RSD criterion of 20%. No qualifiers were required since this analytes was not detected in the associated samples.

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 results 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 SDG.

SDG WG21544: hexachlorobenzene (4 results), hexachlorobutadiene (1 result)

SDG WG21586: hexachlorobenzene (2 results)

SDG WG21917: beta-HCH (4 results)

Field Blanks

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 samples. If a contaminant is reported in any field sample and the concentration is less than the action level, the result is qualified as not detected (U-6).

SDG WG21544, WG21776, WG21917, WG22031: Two field blanks (LW3-W4900-F and LW3-W4901-F) were reported, and associated with all filter samples. A positive result was detected for 4,4´-DDE. One result for 4,4'-DDE was qualified as not detected (U-6) based on field blank contamination.

SDG WG21586, WG21824, WG21920, WG22063: Two field blanks (LW3-W4900-C and LW3-W4901-C) were reported, and associated with all cartridge samples. Positive results for cis-chlordane, heptachlor, and 4,4'-DDE were reported in Sample LW3-W4900-C. Positive results for dieldrin and methoxychlor were reported in Sample LW3-W4901-C. Eleven results for heptachlor and six results for methoxychlor were qualified as not detected (U-6) based on field blank contamination.

Labeled Compounds

SDG WG21544: The percent recovery (%R) values for 13C-1,2,3-trichlorobenzene were less than the lower control limit in Samples LW3-W4023-M-F, LW3-W4901-F, LW3-W4025-M-F, and LW3-W4024-NS-F. The quantitation of hexachlorobutadiene is associated with this labeled compound. The hexachlorobutadiene results and reporting limits were qualified as estimated (J/UJ-13) in these samples. 13C-1,2,3-trichlorobenzene was not recovered in Sample LW3-W4900-F. Hexachlorobutadiene was not reported in this sample and the result field was

qualified as rejected (R-13). The %R value for 13C-methoxychlor was less than the lower control limit in the laboratory blank. Qualifiers are not assigned to QC samples.

SDG WG21586: The %R values for 13C-1,2,3-trichlorobenzene were less than the lower control limit in Samples LW3-W4900-C, LW3-W4901-C, LW3-W4025-M-C, LW3-W4024-NS-C, and the laboratory blank. The quantitation of hexachlorobutadiene is associated with this labeled compound. The hexachlorobutadiene results and reporting limits were qualified as estimated (J/UJ-13) in these samples. Qualifiers are not assigned to QC samples. The %R value for 13C-endrin was greater than the upper control limit in Samples LW3-W4024-NB-C, LW3-W4023-M-C, and LW3-W4025-M-C. Positive results for the associated analytes (endrin, endrin aldehyde, and endrin ketone) were qualified as estimated (J-13) in these samples.

SDG WG21824: The %R value for 13C-1,2,3-trichlorobenzene was less than the lower control limit in Sample LW3-W4025-E-C. The quantitation of hexachlorobutadiene is associated with this labeled compound. The hexachlorobutadiene result was qualified as estimated (J-13) in this sample.

SDG WG21917: The %R values for 13C-1,2,3-trichlorobenzene was less than the lower control limit in Samples LW3-W4035-NB-F, LW3-W4035-NS-F, the Method Blank, and the ongoing precision and recovery (OPR) sample. The quantitation of hexachlorobutadiene is associated with this labeled compound. The hexachlorobutadiene result was qualified as estimated (J-13) in Samples LW3-W4035-NB-F and LW3-W4035-NS-F; no qualifiers were applied to the QC samples. The %R values for 13C-1,2,3-trichlorobenzene was less than 10% in Sample LW3-W4032-NB-F, the hexachlorobutadiene result was rejected (R-13) in this sample.

SDG WG21920: The %R values for 13C-1,2,3-trichlorobenzene was less than the lower control limit in five samples. The quantitation of hexachlorobutadiene is associated with this labeled compound. The hexachlorobutadiene results were qualified as estimated (J-13) in these samples. The %R values for 13C-1,2,3-trichlorobenzene was less than 10% in Sample LW3-W4025-W-C. The hexachlorobutadiene result was rejected (R-13) in this sample.

Matrix Spikes/Matrix Spike Duplicates

No matrix spike/matrix spike duplicate or laboratory duplicate analyses were performed. Accuracy was assessed using labeled compound recoveries and OPR samples. Precision could not be assessed.

Ongoing Precision and Recovery

SDG WG21544: The %R values for endrin aldehyde and endrin ketone were less than the lower control limits. Results and reporting limits were qualified as estimated (J/UJ-10) in the associated samples.

SDG WG21917: The %R values for 2,4'-DDD and 4,4'-DDD were greater than the upper control limits. Positive results were qualified as estimated (J-10) in the associated samples.

SDG WG21920: The %R value for endrin aldehyde was greater than the upper control limit. No positive results for this analyte were reported, no qualifiers were applied.

Field Duplicates

Duplicate sample pairs are listed below. The following acceptance criteria were applied: the relative percent difference (RPD) control limit is 50% for results greater than 5x the reporting limit. For results less than 5x the reporting limit, the absolute difference between the sample and duplicate must be less than the reporting limit. No data were qualified based on field duplicate outliers; however, data users should take field precision into account when interpreting sample data.

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. These results were qualified as not detected (U-21) at the reported concentration.

SDG WG22063: The laboratory noted an interference that affected the quantitation of heptachlor in Sample LW3-W4031-NB-C. This result was previously qualified as not detected (U-21); no further qualification was necessary.

Reporting Limits (Method Detection Limit and Method Reporting Limit)

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

Calculation Verification

SDG WG21544: A full (Level IV) validation, including calculation verification, was performed for this SDG. No calculation or transcription errors were found.

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. Precision was not evaluated.

Data were qualified as estimated due to labeled compound and OPR %R outliers, and due to an analyte not being included in the calibration standards. Data were qualified as not detected due to ion ratio criteria outliers and contamination in the associated laboratory and field blanks.

Results which were associated with very low or zero recoveries of labeled compound were rejected. Rejected data is not usable.

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

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3A Surface Water – High Flow Event (57C)
PCB Congeners by Axys Method MLA-010 (EPA 1668A)
Axys Analytical Services, Ltd.

This report documents the review of analytical data from the analyses of XAD resin 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	Number of Samples	Validation Level
WG21461	4 XAD Resins & 2 Equipment Blanks	Summary
WG21531	4 XAD Filters & 2 Equipment Blanks	Full
WG21775	12 XAD Filters	Summary
WG21766	9 XAD Filters	Full
WG21768	9 XAD Resins	Summary
WG21824	1 XAD Resin	Summary
WG21913	8 XAD Resins	Summary
WG21916	6 XAD Filters	Summary
WG21919	8 XAD Resins	Summary
WG21920	1 XAD Resin	Summary
WG22030	9 XAD Filters	Summary
WG22064	9 XAD Resins	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 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%).

SDG WG21531: Aroclor results were not provided in the EDD file.

SDG WG21768: Sample LW3-W4029-NS-C was mislabeled as LW3-W4-SW029-NS. This discrepancy was noted in the laboratory case narrative and corrected in the EDD.

SDG WG22030: Sample LW3-W4031-NB-F was mislabeled as LW3-W4031-F-NB and Sample LW3-W4031-NS-F was mislabeled as LW3-W4031-F-NS. These discrepancies were noted in the laboratory case narrative and corrected in the EDD.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

1	Holding Times and Sample Receipt	1	Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
	Initial Calibration (ICAL)		Ongoing Precision and Recovery (OPR)
	Continuing Calibration (CCAL)		Field Duplicates
2	Laboratory Blanks	2	Compound Identification
2	Field Blanks		Reporting Limits (MDL and MRL)
2	Labeled Compounds	1	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. All were extracted within 55 days of collection and analyzed within 55 days of extraction.

SDG WG21531: Samples were received at a temperature of 7ºC, greater than the recommended range of 4ºC ±2º. This outlier did not impact data quality and no qualifiers were required.

SDG WG21775, WG21766, WG21768, WG21824, WG21913, WG21916, WG21919, WG21920, WG22030, WG22064: The sample coolers were received between 3°C and 8°C, some exceeding the recommended temperature range of 4°C ±2°. Specific cooler temperatures were not listed. It was determined that the temperature outliers did not impact data quality and no qualifiers were required.

SDG WG21913: The PCB extract from Sample LW3-W4023-M-C was lost during a laboratory mishap. The remaining extract after pesticide analysis was analyzed for PCB congeners and reported as SDG WG21824.

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 for all SDG. Positive results less than the action levels were qualified as not detected (U-7) in several samples from each associated SDG.

Results for several target analytes were reported in the associated method blanks. All results less than these action levels were qualified as not detected (U-7) in all associated samples.

SDG WG21913: The laboratory noted that a second blank (a "media field blank") was associated with Samples LW3-W4025-E-C and LW3-W4025-M-C2. This blank monitored the contamination from a different batch of extraction media, which was used with these samples. All results less than this blank's action levels were qualified as not detected (U-7) in both samples.

Field Blanks

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 samples. If a contaminant is reported in any field sample and the concentration is less than the action level, the result is qualified as not detected (U-6).

SDG WG21461, WG21768, WG21913, WG21919, WG22064, WG21824, WG21920: Two field blanks (LW3-W4900-C and LW3-W4901-C) were reported. Many positive results were detected in these field blanks. A complete list of these results is included with the validation worksheets. Numerous congener and Aroclor results were qualified as not detected (U-6).

SDG WG21531, WG21766, WG21775, WG21916, WG22030: Two field blanks (LW3-W4900-F and LW3-W4901-F) were reported. Many positive results were detected in these field blanks. A complete list of these results is included with the validation worksheets. Results for PCB congeners 1, 3, and 52 were qualified as not detected (U-6).

Labeled Compounds

SDG WG21766: The recovery of 13C-PCB28, a clean-up standard, was less than lower control limits in Sample LW3-W4030-NS-F. This compound is not used to calculate target analyte results; no qualifiers were applied.

SDG WG21768: The labeled compounds 13C-PCB1 and 13C-PCB3 were not recovered from any of the nine samples or the method blank. Results for the associated native PCB congeners (1,2,3) were not reported; these data points were rejected (R-13) in all samples. No qualifiers were applied to the method blank.

SDG WG21920: The labeled compounds 13C-PCB1 and 13C-PCB4 were recovered at less than lower control limits from Sample LW3-W4032-NB-C. Results and reporting limits for the associated native PCB congeners (1,2,4,5,6,7,8,9,10,11,12,13,14) were qualified as estimated (J/UJ-13). The labeled compounds 13C-PCB1, 13C-PCB3, 13C-PCB4, and 13C-PCB19 were not recovered from the method blank and the labeled compounds 13C-PCB15 and 13C-PCB77 were recovered at less than lower control limits and greater than upper control limits, respectively. Target compounds associated with zero or low %R values from this blank were already qualified as estimated, or were greater than five times the detection limit. Therefore, no further qualifiers were applied for potential contamination.

SDG WG22030: The labeled compounds 13C-PCB1 and 13C-PCB3 were not recovered from Samples LW3-W4031-NS-C and LW3-W4038-NS-C. Results for the associated native PCB congeners (1,2,3) were not reported and these data points were rejected (R-13) in these samples. The labeled compound 13C-PCB3 was not recovered from Samples LW3-W4036-NS-C and

LW3-W4037-NS-C. Results for PCB3 were rejected (R-13) and results for PCB2 were qualified as estimated (J-13) in these samples.

SDG WG22064: The recovery (%R) of labeled compound 13C-PCB4 was below control limits in Sample LW3-W4034-NB-F. Results and reporting limits for the associated native PCB congeners were qualified as estimated (J/UJ-13) in this sample.

Matrix Spikes/Matrix Spike Duplicates

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

Ongoing Precision and Recovery

SDG WG21824, WG21920: No OPR samples were submitted, since these analyses were performed on remaining pesticide extract fractions, without PCB spikes. Accuracy was assessed using labeled compound recovery values only.

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. These results were qualified as not detected (U-21) at the reported concentration.

The laboratory did not include Aroclor identification standards to assess pattern matching. Additionally, values reported for Aroclors were calculated from PCB congener results using a laboratory-derived calculation factor, instead of an Aroclor-specific response factor. Therefore, all positive Aroclor results were qualified as estimated and tentatively identified (NJ-14).

SDG WG21461: Interferences were noted that affected the quantitation of PCB001, PCB002, PCB003 in Samples LW3-W4023-M-C and LW3-W4901-C. The results for these PCB were qualified as estimated (J-14) in these samples, with the exceptions of PCB002 and PCB003 from Sample LW3-W4901-C, where the laboratory was unable to provide data. These data points were rejected (R-14).

SDG WG21531: Interferences were noted that affected the quantitation of PCB082 and PCB197/200 in Sample LW3-W4024-NS-F and PCB027 in Sample LW3-W4025-M-F. These results were qualified as estimated (J-14) in these samples.

SDG WG21768: Interferences were noted that affected the quantitation of PCB082 in five samples and PCB083/099 in three samples. Interferences were noted that affected the quantitation of PCB066 in Samples LW3-W4026-NB-C and LW3-W4026-NS-C. The results for these PCB were qualified as estimated (J-14) in these samples.

SDG WG21775: Interferences were noted that affected the quantitation of PCB197/200 in seven samples. Interferences were noted that affected the quantitation of PCB027 in Sample LW3-W4011-NS-F and PCB198 in Sample LW3-W4023-M-F. The results for these PCB were qualified as estimated (J-14) in these samples.

SDG WG21919: Interferences were noted that affected the quantitation of PCB001 in four samples and the quantitation of PCB032 in three samples. Interferences were noted that affected the quantitation of PCB004 in Sample LW3-W4035-NS-C and PCB024 in Sample LW3-W4025-W-C. The results for these PCB were qualified as estimated (J-14) in these samples.

SDG WG21920: Interferences were noted that affected the quantitation of PCB032 in Sample LW3-W4032-NB-C. The result for this PCB was qualified as estimated (J-14) in this sample.

SDG WG22030: Interferences were noted that affected the quantitation of PCB203 in Samples LW3-W4031-NS-F and LW3-W4034-NB-F, and PCB0321 & 197/200 in Sample LW3-W4031-NB-F. These results were qualified as estimated (J-14) in these samples.

SDG WG22064: Interferences were noted that affected the quantitation of PCB082 in Samples LW3-W4031-NB-C, LW3-W4036-NS-C, and LW3-W4038-NS-C, and PCB001 & 002 in Sample LW3-W4031-NB-C. These results were qualified as estimated (J-14) in these samples.

Calculation Verification

SDG WG21531, WG21766: A full (Level IV) validation, including calculation verification, was performed for these SDG. No calculation or transcription errors were found.

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 exception noted above. Precision was acceptable as demonstrated by the field duplicates, with the exceptions noted above.

Data were qualified as estimated due to matrix or spectral interferences. Data were qualified as not detected due to ion ratio criteria outliers and contamination from the associated laboratory and field blanks. Aroclor results were qualified as tentatively identified at estimated concentrations.

Data were rejected due to labeled compound outliers and severe spectral interferences.

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

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3A Surface Water – High Flow Event (57C)
Dioxin & Furan Compounds by Axys Method MLA-017 (EPA 1613B)
Axys Analytical Services, Ltd.

This report documents the review of analytical data from the analyses of XAD resin and filter 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	Number of Samples	Validation Level
WG21529	4 XAD Filters & 2 Field Blanks	Full
WG21542	4 XAD Resin & 2 Field Blanks	Summary
WG21774	12 XAD Filters	Summary
WG21789	9 XAD Resins	Summary
WG21823	6 XAD Filters	Summary
WG21868	9 XAD Resins	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%).

SDG WG21774 & WG21789: Samples LW3-W4023-M (-C and –F) were originally collected on 1/15, and recollected on 3/02. The recollected samples were renamed in the EDD (sys_sample_code field) as LW3-W4023-M2 (-C and –F) to differentiate from the originals.

Samples LW3-W4025-M (-C and –F) were originally collected on 1/15, and recollected on 3/10. The recollected samples were renamed in the EDD (sys_sample_code field) as LW3-W4025-M2 (-C and –F) to differentiate from the originals.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

1	Holding Times and Sample Receipt	1	Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
	Initial Calibration (ICAL)		Ongoing Precision and Recovery (OPR)
	Continuing Calibration (CCAL)		Field Duplicates
2	Laboratory Blanks	2	Compound Identification
2	Field Blanks		Reporting Limits (MDL and MRL)
2	Labeled Compounds	1	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 were extracted within 46 days of collection and analyzed within 36 days of extraction.

SDG WG21529 & WG21542: The sample cooler was received at 7°C, outside the recommended temperature range of 4°C ±2°. This temperature outlier did not impact data quality and no qualifiers were required. In addition, the chain of custody (COC) was not signed by the sampler to relinquish custody. The COC was correctly signed by the laboratory to indicate sample receipt.

SDG WG21774, WG21789, WG21823, & WG28168: The sample coolers were received at temperatures ranging from 3° to 8°C, outside the recommended temperature range of 4°C ±2°. These temperature outliers did not impact data quality and no qualifiers were required.

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. Contaminant levels, associated samples, and action levels are documented 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.

SDG WG21529: OCDD (2 results)

SDG WG21542: OCDD (2 results); 2,3,4,7,8-PeCDF (1 result); 2,3,4,6,7,8-HxCDF (1 result); total penta-furans (1 result)

SDG WG21774: 1,2,3,7,8-PeCDD (5 results), 1,2,3,7,8-PeCDF (7 results), 1,2,3,7,8,9-HxCDF (7 results)

SDG WG21789: 1,2,3,7,8-PeCDD (3 results), 1,2,3,4,7,8-HxCDD (7 results), 1,2,3,4,7,8-HxCDF (4 results), 1,2,3,6,7,8-HxCDF (2 results), 1,2,3,4,6,7,8-HpCDF, 1,2,3,4,7,8,9-HpCDF, OCDF, total hexa-dioxins, total hepta-furans (1 result each)

SDG WG21823: 1,2,3,7,8,9-HxCDF (2 results), 1,2,3,4,7,8,9-HpCDF (1 result)

SDG WG21868: 1,2,3,7,8-PeCDD (5 results), 1,2,3,6,7,8-HxCDD (8 results), 1,2,3,7,8,9-HxCDD (8 results), 1,2,3,7,8-PeCDF (7 results), 2,3,4,7,8-PeCDF (7 results), 1,2,3,4,7,8-HxCDF (9 results), 1,2,3,6,7,8-HxCDF (9 results), 1,2,3,7,8,9-HxCDF (3 results), 2,3,4,6,7,8-HxCDF (5 results), 1,2,3,4,6,7,8-HpCDF (8 results), 1,2,3,4,7,8,9-HpCDF (9 results), OCDF (9 results), total penta-dioxins (1 result), total hexa-dioxins (1 result), total tetra-furans (9 results), total penta-furans (9 results), total hexa-furans (9 results), total hepta-furans (9 results)

Field Blanks

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 samples. If a contaminant is reported in any field sample and the concentration is less than the action level, the result is qualified as not detected (U-6).

WG21529, WG21774, WG21823: Two field blanks (LW3-W4900-F and LW3-W4901-F) were reported. Positive results for several target analytes were detected in these blanks after qualifiers based on the laboratory blanks were assigned. The following filter sample results were qualified as not detected (U-6) based on field blank contamination.

Analyte	Number of Results
1,2,3,7,8-PeCDF	12
2,3,4,7,8-PeCDF	15
1,2,3,6,7,8-HxCDF	4
1,2,3,7,8,9-HxCDF	7
2,3,4,6,7,8-HxCDF	8
1,2,3,4,7,8,9-HpCDF	3
Total Tetra-Furans	1
Total Penta-Furans	1
Total Hexa-Furans	1

WG21542, WG21789, WG21868: Two field blanks (LW3-W4900-C and LW3-W4901-C) were reported. Positive results for several target analytes were detected in these blanks after qualifiers based on the laboratory blanks were assigned. The following column sample results were qualified as not detected (U-6) based on field blank contamination.

Analyte	Number of Results
1,2,3,7,8-PeCDD	12
1,2,3,4,7,8-HxCDD	19
1,2,3,7,8-PeCDF	9
1,2,3,4,7,8-HxCDF	22
1,2,3,6,7,8-HxCDF	9
1,2,3,4,7,8,9-HpCDF	2
Total Hexa-Dioxins	4
Total Tetra-Furans	12
Total Penta-Furans	16
Total Hexa-Furans	8

Labeled Compounds

SDG WG21823: The percent recovery (%R) value for 13C-1,2,3,6,7,8-HxCDF was greater than the upper control limit of 123%, at 129%, in Sample LW3-W4033-NB-F. The result for the associated native compound was estimated (J-13).

Matrix Spikes/Matrix Spike Duplicates

No matrix spike/matrix spike duplicate or duplicate analyses were performed. Accuracy was assessed using labeled compound recovery values and ongoing precision and recovery (OPR) samples. Precision could not be assessed.

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. These results were qualified as not detected (U-21) at the reported concentration.

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.

SDG WG21823: The laboratory noted diphenyl ether interferences affecting quantitation of 2,3,4,6,7,8-HxCDF, total tetra-furans, and total penta-furans in Samples LW3-W4033-NB-F and LW3-W4035-NS-F. The results for these analytes were qualified as estimated (J-14) in these samples.

SDG WG21868: The laboratory noted lock mass interferences affecting total penta-dioxins and total penta-furans in Sample LW3-W4027-NS-C and 1,2,3,7,8-PeCDF and total penta-furans in Sample LW3-W4035-NB-C. Positive results and reporting limits for these analytes in these samples were estimated (J/UJ-14).

Calculation Verification

SDG WG21529: Calculation verification was performed on this SDG. No transcription or calculation errors were found.

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. Precision could not be evaluated.

Data were qualified as estimated due to a labeled compound %R outlier and due to diphenyl ether and lock mass interferences. Data were qualified as not detected due to ion ratio criteria outliers and contamination from the associated laboratory and field 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.