EcoChem, Inc.

Environmental Science and Chemistry

DATA QUALITY EVALUATION

PORTLAND HARBOR RI/FS

BENTHIC INVERTEBRATE MULTIPLATE TISSUES – FALL 2005

Dioxin/Furan Compounds – EPA Method 1613 ver. B
Polychlorinated Biphenyl (PCB) Congeners – EPA Method 1668A
Chlorinated Pesticides - Axys Method MLA-028
Metals – EPA Methods 6010, 6020, 7742

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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 5: Multiplate Tissue Collection;* 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).

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 communication records are organized by SDG and retained 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 (ten percent for PCB congener EDD files).

The Multiplate tissue data received Level III data 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)

A minimum ten percent (10%) of all Multiplate tissue 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: METALS

Two (2) Multiplate tissue samples were analyzed for metals for the Portland Harbor RI/FS. One rinse proof blank was generated to monitor the sample homogenization process. All metals analyses were completed by Columbia Analytical Services, Kelso, Washington.

The metals data for the Multiplate tissue samples were generally acceptable. A total of nine (9) data points (19% of all Multiplate tissue metals results) were 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, 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 Multiplate tissue metals analyses.

Holding Times and Sample Preservation

All analytical holding time requirements were met for metals analysis of the Multiplate tissue samples and associated QC samples.

Instrument Performance

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

Method Blank Analyses

Method and instrument 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 and instrument blanks were analyzed at the appropriate frequency. Various target analytes were detected in the method and/or instrument blanks. A summary of contaminant levels, associated samples, and action levels is provided in the data validation worksheets. Two antimony results (4.3% of all Multiplate tissue metals results) were qualified as not detected (U) based on blank contamination.

Accuracy

The accuracy of the analytical results is evaluated in the following sections in terms of analytical bias (matrix spike [MS], laboratory control sample [LCS], contract required detection limit [CRDL] standard recovery values, interference check samples [ICS], and serial dilution percent difference [%D] values) and precision (laboratory duplicate analyses).

Matrix Spike Recoveries

There was insufficient tissue sample available to analyze a matrix spike; therefore the laboratory analyzed duplicate standard reference material (SRM) samples. Analysis frequency and recoveries met the criteria for acceptable performance.

Laboratory Control Sample Recoveries

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

Contract Required Detection Limit Standard Analyses

CRDL standards were analyzed at the beginning of each analytical sequence. For recoveries greater than the 130% upper control limit, the associated positive results less than two times the CRDL are estimated (J) to indicate a potential high bias. For recoveries less than the 70% lower control limit, positive results less than twice the CRDL and non-detects are estimated (J/UJ) to indicate a potential low bias. One result for selenium (2.1% of all Multiplate tissue metals results) was qualified as estimated (J) for potential low bias based on CRDL standard recovery outliers.

Interference Check Samples

ICP interference check samples were analyzed at the beginning of each analytical sequence. All ICP interference check sample results were within the acceptance criteria.

Serial Dilution Analyses

Serial dilution analyses were performed at the proper frequency. Serial dilution %D values greater than 10% may indicate the presence of matrix interference, resulting in potential bias. Eight (8) results (17% of all Multiplate tissue metals results) were qualified as estimated (J) based on serial dilution outliers. The qualifiers were applied to copper, lead, silver, and zinc (2 results each).

Precision

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

Method Detection Limits and Method Reporting Limits

The laboratory reported non-detects at the MDLs, adjusted for sample size and any dilution factor. The method reporting limit (MRL) for antimony was 0.011 mg/kg for the only non-detected tissue result.

Other Quality Control Samples

QC samples analyzed with the Multiplate tissue study included a homogenization proof blank. The results are discussed in the following sections.

Homogenization Proof Blank

A homogenization proof blank was associated with the Multiplate tissue samples. After qualifiers based on method blank contamination were issued, positive results for six metals remained in this proof blank. Results from the associated tissue samples were above the blank action levels, so no qualifiers were required.

Field Replicate Samples

No field replicate sample pairs were associated with the Multiplate tissue samples.

SUMMARY OF DATA VALIDATION: PESTICIDE COMPOUNDS

Seven (7) Multiplate tissue samples were analyzed for chlorinated pesticide compounds for the Portland Harbor RI/FS. One rinse proof blank was generated to monitor the sample homogenization process. Axys Analytical, Sidney, British Columbia, completed all pesticide analyses.

The pesticide data for the Multiplate tissue samples were generally acceptable. A total of 11 data points (4.9% of all Multiplate tissue pesticide results) were estimated because control limits were exceeded in one or more laboratory quality control 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, 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 Multiplate tissue pesticide analyses.

No results were reported for toxaphene, although it was designated as a target analyte for this project. This was noted in previous data quality evaluation reports for earlier phases of this project. No further action was taken.

Holding Times and Sample Preservation

All extraction holding time requirements were met for pesticide analysis of the Multiplate tissue samples and associated QC samples. The analytical holding time of 40 days from the date of extraction was exceeded for re-analysis of Sample LW2-MIT004. The three (3) results reported from this re-analysis (1.3% of all Multiplate tissue pesticide results) were qualified as estimated (J) due to holding time outliers.

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, 1,2,3-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 acceptance criteria.

A total of eight (8) hexachlorobutadiene results (3.6% of all Multiplate pesticide results) were qualified as estimated (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.

Method blanks were analyzed at the appropriate frequency. A total of three (3) results (1.3% of all Multiplate tissue 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).

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 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 and field duplicate analyses were not performed. Precision was assessed using OPR analyses only.

Method Detection Limits and Method Reporting Limits

To meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the detection limits, adjusted for sample size and any dilution factor. These method reporting limits (MRLs) ranged from 0.00185 pg/g to 0.063 pg/g for the non-detected results. The MRLs met project criteria for all analytes except toxaphene, which was not analyzed for the Multiplate tissue 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. Forty (40) data points (18% of all Multiplate tissue pesticide data points) were qualified as not detected because the ion abundance ratio criteria were not met.

Other Quality Control Samples

QC samples analyzed with the Multiplate tissue study included a homogenization proof blank. The results are discussed in the following sections.

Homogenization Proof Blank

A homogenization proof blank was associated with the Multiplate tissue samples. After qualifiers based on method blank contamination and compound identification were issued, no positive results for dioxins or furans were detected in this proof blank.

Field Replicate Samples

No field replicate sample pairs were associated with the Multiplate tissue samples.

SUMMARY OF DATA VALIDATION: PCB CONGENERS

Seven (7) Multiplate tissue samples were analyzed for polychlorinated biphenyl (PCB) congeners for the Portland Harbor RI/FS. One rinse proof blank was generated to monitor the sample homogenization process. Samples were analyzed by Axys Analytical Services, Ltd. Sidney, British Columbia.

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 Multiplate tissue PCB analyses.

Holding Times and Sample Preservation

All extraction and analytical holding time requirements were met for the PCB congener analyses of Multiplate tissue samples and associated 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.

Method blanks were analyzed at the appropriate frequency. A total of 23 results (1.6% of all Multiplate tissue 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 OPR samples).

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

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 and field duplicate analyses were not performed. Precision was assessed using OPR analyses only.

Method Detection Limits and Method Reporting Limits

To meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the detection limits, adjusted for sample size and any dilution factor. These method reporting limits (MRLs) range from 0.0369 pg/g to 15.9 pg/g 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 83 data points (5.9% of all Multiplate tissue PCB data points) were qualified as not detected because the ion abundance ratio criteria were not met.

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 17 positive Aroclor results (1.2% of all Multiplate tissue PCB data points) were qualified (NJ) to indicate that they should be considered as tentatively identified at estimated concentrations.

Other Quality Control Samples

QC samples analyzed with the Multiplate tissue study included a homogenization proof blank. The results are discussed in the following sections.

Homogenization Proof Blank

A homogenization proof blank was associated with the Multiplate tissue samples. After qualifiers based on method blank contamination and compound identification were issued, positive results for PCB congeners remained in this proof blank. Results from the associated tissue samples were above the blank action levels, so no qualifiers were required.

Field Replicate Samples

No field replicate sample pairs were associated with the Multiplate tissue samples.

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

Seven (7) Multiplate tissue samples were analyzed for dioxin and furan compounds for the Portland Harbor RI/FS. One rinse proof blank was generated to monitor the sample homogenization process. Axys Analytical, Sidney, British Columbia, completed all dioxin/furan analyses.

The dioxin/furan data for the Multiplate tissue samples were acceptable. No data were estimated or 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 Multiplate tissue dioxin/furan analyses.

Holding Times and Sample Preservation

All extraction and analytical holding time requirements were met for the dioxin/furan analyses of the Multiplate tissue samples and associated 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.

Method blanks were analyzed at the appropriate frequency. A total of seven results (3.4% of all Multiplate tissue 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 OPR samples).

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

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 and field duplicate analyses were not performed. Precision was assessed using OPR analyses only.

Method Detection Limits and Method Reporting Limits

To meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the detection limits, adjusted for sample size and any dilution factor. These method reporting limits (MRLs) range from 0.0489 pg/g to 0.95 pg/g 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 estimated maximum possible concentration (EMPC) value, which is 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. Twenty-two (22) data points (11% of all Multiplate tissue dioxin/furan data points) were qualified as not detected because the ion abundance ratio criteria were not met.

Other Quality Control Samples

QC samples analyzed with the Multiplate tissue study included a homogenization proof blank. The results are discussed in the following sections.

Homogenization Proof Blank

A homogenization proof blank was associated with the Multiplate tissue samples. After qualifiers based on method blank contamination and compound identification were issued, no positive results for dioxins or furans were detected in this proof blank.

Field Replicate Samples

No field replicate sample pairs were associated with the Multiplate tissue samples.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Multiplate Tissue Study
Dioxin/Furan Compounds
Method: EPA 1613B
Axys Analytical Services, Ltd.

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

SDG	Number of Samples	Validation Level
WG17772	7 TISSUES	FULL
WG17828	1 HOMOGENIZATION PROOF BLANK	COMPLIANCE

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 quality control (QC)results were also verified (10%).

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

	Holding Times and Sample Receipt	1	Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
	Initial Calibration (ICAL)	1	Ongoing Precision and Recovery
	GC/MS Tuning	1	Laboratory Duplicate
	Continuing Calibration (CCAL)	1	Field Duplicates
2	Laboratory Blanks	2	Compound Identification
1	Field Blanks		Reporting Limits (MDL and MRL)
	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.***

Laboratory Blanks

SDG WG17772: Positive values for OCDD, 2,3,4,6,7,8-HpCDF, and OCDF were reported in the laboratory blank. Action levels of five times the blank concentrations were established and the sample results were compared to this action level. Values less than the action level were qualified as not detected (U-7) at the reported concentration.

SDG WG17828: This SDG consisted of a laboratory preparation blank and a homogenization proof blank. Positive values for 1,2,3,4,6,7,8-HpCDD and OCDD were reported in the laboratory preparation blank. Action levels of five times the blank concentrations were established and the sample results were compared to this action level. Values less than the action level were qualified as not detected (U-7) at the reported concentration. After qualifiers based on the laboratory preparation blank were assigned, no positive values remained in the homogenization proof blank.

Field Blanks

No field blanks were submitted with these SDG.

Matrix Spikes/Matrix Spike Duplicates (MS/MSD)

No MS/MSD sets were performed with these SDGs. Accuracy and precision were assessed using labeled compound recoveries and ongoing precision and recovery samples.

Ongoing Precision and Recovery

SDG WG17828: No ongoing precision and recovery (OPR) was analyzed with this SDG.

Laboratory Duplicate

No laboratory duplicate were reported with these SDGs. Precision was assessed using the ongoing precision and recovery 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 value may be a false positive. These values were qualified as not detected (U-21).

Calculation Verification

SDG WG17772: Full (Level IV) validation 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 percent recovery values. Precision was acceptable as demonstrated by the OPR recovery values.

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

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Multiplate Tissue Study
PCB Congeners by EPA Method 1668
Axys Analytical Services, Ltd.

This report documents the review of analytical data from the analyses of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Axys Analytical Services, Ltd. Sidney, British Columbia, Canada.

SDG	Number of Samples	Validation Level
WG17772	7 TISSUES	FULL
WG17828	1 HOMOGENIZATION PROOF BLANK	COMPLIANCE

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.

III. 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 quality control (QC) results were also verified (10%). Numerous reporting limits and method detection limits (MDL) did not match between the data packages and EDD files. The laboratory re-submitted all EDD files and replaced several data package pages to resolve the inconsistencies.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

	Holding Times and Sample Receipt	1	Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
	GC/MS Tuning	1	Ongoing Precision and Recovery
	Initial Calibration (ICAL)	1	Laboratory Duplicate
	Calibration Verification (CVER)	1	Field Duplicates
	Isomer Specificity	2	Compound Identification
2	Laboratory Blanks	1	Calculation Verification (full validation only)
1	Field Blanks	1	Reporting Limits (MDL and MRL)
1	Labeled Compound Recovery		

[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.***

Laboratory Blanks

In order to assess the impact of laboratory blank contamination on the reported sample results, action levels at five times the blank concentrations are established. If the concentrations in the associated field samples are less than the action levels, the results are qualified as not detected (U-7). If the result is also less than the reporting limit, the result is elevated to the reporting limit.

SDG WG17772: Positive results for several PCB congeners were reported in the laboratory preparation blank. All sample concentrations were either greater than the established action levels, or the PCB was not detected in the sample. The samples were then compared to the homogenization proof blank from SDG WG17828. Again, all sample concentrations were greater than the established action levels. No qualifies were required.

SDG WG17828: This SDG consisted of one laboratory preparation blank and a homogenization proof blank. Positive results for several PCB congeners were reported in the laboratory preparation blank. After qualifiers based on the laboratory preparation blank were assigned, positive results for 11 congeners remained in the homogenization proof blank. Results from the associated tissue samples were greater than the action levels so no qualifiers were required.

Field Blanks

No field blanks were submitted with these SDGs.

Labeled Compound Recovery

SDG WG17772: The percent recovery (%R) values for the labeled compounds 13C-PCB4 and 13C-PCB19 were less than their lower control limits in the laboratory preparation blank. The %R values for the labeled compounds 13C-PCB1, 13C-PCB4, and 13C-PCB19 were less their lower control limits in the duplicate ongoing precision and recovery (OPR) sample. Qualifiers are not assigned to QC samples.

SDG WG17828: The %R values for the labeled compounds 13C-PCB4 and 13C-PCB19 were less than their lower control limits in the laboratory preparation blank. Qualifiers are not assigned to QC samples.

Matrix Spikes/Matrix Spike Duplicates (MS/MSD)

No MS/MSD sets were performed with these SDGs. Accuracy and precision were assessed using labeled compound recoveries and OPR samples.

Ongoing Precision and Recovery

SDG WG17828: No OPR was analyzed with this SDG.

Laboratory Duplicate

No laboratory duplicate were reported with these SDG. Precision was assessed using the ongoing precision and recovery samples.

Field Duplicates

No field duplicates were submitted with these SDGs.

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; therefore these results were qualified as not detected (U-21).

SDG WG17772: No Aroclor patterns were observed in the samples and the laboratory reported default Aroclor identifications. Additionally, results 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).

Calculation Verification

SDG WG17772: Full (Level IV) validation was performed on this SDG. No transcription or calculation errors were found.

Reporting Limits (MDL and MRL)

SDG WG17828: The laboratory determined sample detection limits by converting the minimum detectable signal to a concentration. The minimum detectable area is determined as three times the height of the detector noise, converted to a peak area using the area/height ratio of the corresponding surrogate peak. These limits are generally supported by regular method detection limit (MDL) studies using low level spikes, following the statistical procedures outlined in the US Code of Federal Regulations (40 CFR Part 136, Appendix B). However, for this SDG, the statistical MDL values exceeded the quoted detection limits by **up to ten times**. The laboratory explained that the sample detection limit is a more realistic measure of sensitivity than the statistical MDL, so this value should be considered the actual detection limit of this analysis. However, when no target analyte is detected, the higher value of the sample detection limit and the MDL is reported as the detection limit.

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 acceptable as demonstrated by the RPD values for the duplicate OPR analyses.

Data were qualified as not detected based on contamination in the associated blanks and potential false positive results. Aroclor results were also qualified as tentatively identified at estimated concentrations.

All data, as estimated, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Multiplate Tissue Study
Method: MLA013
Axys Analytical Services, Ltd.

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

SDG	Number of Samples	Validation Level
WG17772	7 TISSUES	FULL
WG17828	1 HOMOGENIZATION PROOF BLANK	COMPLIANCE

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables, with exceptions noted below. 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 quality control (QC) results were also verified (10%).

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

2	Holding Times and Sample Receipt	1	Ongoing Precision & Recovery (OPR)
	GC/MS Tuning	1	Laboratory Duplicates
2	Initial Calibration (ICAL)	1	Field Duplicates
2	Continuing Calibration (CCAL)		DDT/Endrin Breakdown
2	Laboratory Blanks	2	Compound Identification
	Field Blanks	1	Reporting Limits (MDL and MRL)
	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

SDG WG17772: Positive results for 2,4'-DDD, 2,4'-DDE and 4,4'-DDD from Sample LW2-MIT004 required re-analysis at dilution to bring them within the calibration range. This re-analysis was

performed 45 days after the date of extraction, which exceeds the 40-day holding time. These results were estimated (J-1).

Initial Calibration (ICAL)

The analyte hexachlorobutadiene was not included in the standards used for the initial calibration. No positive results for this compound were reported in the samples and all reporting limits were estimated (UJ-5A).

Continuing Calibration (CCAL)

The analyte hexachlorobutadiene was not included in the continuing calibration verification standards. This compound was not reported in the samples and all reporting limits were estimated (UJ-5B).

Laboratory Blanks

In order to assess the impact of blank contamination on the reported sample results, action levels at five times the blank concentrations are established. If the concentrations in the associated field samples are less than the action levels, the results are qualified as not detected (U-7).

SDG WG17772: Positive results for hexachlorobenzene, oxychlordane, gamma-chlordane, and alpha-chlordane were reported in the laboratory preparation blank. Results for these analytes were reported in all samples at concentrations greater than the established action levels and no qualifiers were required.

SDG WG17828: This SDG consisted of a laboratory preparation blank and a homogenization proof blank. Positive results for hexachlorobenzene, aldrin, alpha-chlordane, and trans-nonachlor were reported in the one laboratory preparation blank. After qualifiers based on the laboratory preparation blank were assigned no positive results remained in the homogenization proof blank.

Matrix Spikes/Matrix Spike Duplicates (MS/MSD)

No MS/MSD sets were performed with these SDG. Accuracy and precision were assessed using labeled compound recoveries and ongoing precision and recovery samples.

Ongoing Precision & Recovery (OPR)

SDG WG17828: No ongoing precision and recovery (OPR) was analyzed with this SDG.

Laboratory Duplicates

No laboratory duplicate was reported with these SDGs.

Field Duplicates

No field duplicates were submitted with these SDGs.

Compound Identification & Quantitation

The laboratory applies a “K” flag to pesticide results when a peak is detected but does not meet quantitation criteria, and therefore the reported value cannot be considered a positive identification for this analyte, these results are considered potential false positives and qualified as not detected (U-21).

Reporting Limits (MDL and MRL)

Toxaphene is listed as a target analyte in the quality assurance program plan (QAPP) but was not reported by the laboratory.

Calculation Verification

SDG WG17772: Full (Level IV) validation 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 surrogate and OPR, %R values. Precision was acceptable as demonstrated by the RPD values for the duplicate OPR analyses.

Data were qualified as estimated due to a target analyte being omitted from the calibration standards. Data were also qualified as not detected based on contamination in the associated blanks and potential false positive results.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Mutltiplate Tissue Study, Fall 2005
Metals
Columbia Analytical Laboratories—Kelso

This report documents the review of analytical data from the analyses of archived core samples and the associated laboratory and field quality control samples. Columbia Analytical Laboratories, Inc., Kelso, Washington, analyzed the samples.

SDG	Number of Samples	Validation Level
K0506539	2 TISSUE SAMPLES, 1 RINSE 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 quality control (QC)results were also verified (10%). No errors were found.

III. TECHNICAL DATA VALIDATION

The QC requirements for review are listed below.

	Holding Times and Sample Preservation	1	Laboratory Duplicates
	Initial Calibration (ICAL)	1	ICP Interference Check Samples
	Calibration Verification (CVER)	2	ICP Serial Dilution
2	CRDL Standard		ICPMS Internal Standards
2	Laboratory Blanks		Field Replicates
	Laboratory Control Samples		Reporting Limits (MDL and MRL)
1	Matrix Spike Samples	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.***

CRDL Standard

Contract required detection limit (CRDL) standards were analyzed at the beginning of each analytical sequence. The recovery for selenium (45%) was less than the lower control limit of 70%. The selenium result for Sample MIT009 was less than twice the standard concentration and was qualified as estimated (J) to indicate a potential low bias.

Laboratory Blanks

Aluminum, antimony, cadmium, chromium, copper, lead, silver and zinc were detected in the method and/or instrument blanks at levels greater than the method detection limit (MDL). To evaluate the effect on the sample data, action levels of five times the blank concentrations were established. The antimony result for Sample LW2-MITPROOF was less than the action level and was qualified as not detected (U).

A homogenization blank, LW2-MITPROOF, was prepared and analyzed. After qualification for laboratory blank contamination, positive results remained for aluminum, chromium, copper, lead, nickel and zinc. Results for these analytes in the associated samples were greater than the action level of five times the blank concentrations; no qualification of data was necessary.

Matrix Spike Samples

There was insufficient tissue sample to analyze a matrix spike; therefore the laboratory analyzed duplicate standard reference material (SRM) samples. All recoveries were within the control limits of 75% to 125%; laboratory accuracy was acceptable.

Laboratory Duplicates

There was insufficient tissue sample to analyze a replicate aliquot. The results from the analysis of duplicate SRMs were used to evaluate laboratory precision. The relative percent difference (RPD) values were within control limit of 20% for sample results greater than five times the reporting limit (for results less than five times the reporting limit, the absolute difference was less than twice the reporting limit).

ICP Interference Check Samples

The concentration of the interfering element calcium was greater than the level in the interference check samples (ICSA/ICSAB) in Sample MIT001. The ICSA results were carefully evaluated to determine if there was a potential high or low bias caused by iron interference. Calcium does not interfere with the analytes of interest; no further action was taken.

ICP Serial Dilution

For QC sample LW2-MIT001, the serial dilution percent difference (%D) values for copper (14%), lead (18%), silver (14%), and zinc (13%) were greater than the control limit of 10%. All associated results were positive and were estimated (J).

Calculation Verification

Several results were verified by recalculation from the raw data. No calculation or transcription errors were found.

IV. OVERALL ASSESSMENT

As determined by this evaluation, the laboratory followed the specified analytical methods. The duplicate SRM RPD values indicated acceptable precision. Accuracy was also acceptable, as demonstrated by the SRM %R values.

Data were qualified as not detected based on laboratory blank contamination. Data were also qualified as estimated based on CRDL %R and serial dilution %D outliers.

All data, as qualified, are acceptable for use.

EcoChem, Inc.

Environmental Science and Chemistry

APPENDIX A

DATA QUALIFIER DEFINITIONS AND VALIDATION CRITERIA

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

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler Temperature	Waters/Solids <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 > 30days 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)
Phthalates, 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 %Ds in the closing CCAL are w/in 25%/35% the avg RF from the 2 CCAL may be used to calculate sam	J(+)/UJ(-) natives if %D = 30% - 75% R(+/-) if %D > 75%	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)
Phthalates, 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% - LCL J(+) if %R > UCL J(+)/R(-) if %R < 10%	13
	Method 1613B: %R must meet limits specified inTable 7		
Quantitation/ Identification	SIM ions for analyte, Istd, rec. std. 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 (or equiv) column. 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% (sediment & water)	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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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 Temperature	4°C ±2°	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=CRQL, 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 > CRQL	U(+) if sample result is < CRQL and < 5X rule (raise sample value to CRQL)	7
		U(+) if sample result is > or equal to CRQL and < 5X rule (at reported sample value)	7
Instrument Blanks	Analyzed at the beginning of every 12 hour sequence No analyte > 1/2 CRQL	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
LCS	One per SDG Method Acceptance Criteria	J(+) if %R > UCL J(+)/UJ(-) if %R < LCL J(+)/R(-) using PJ if %R <<LCL (< 10%)	10
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 %D between columns (25%)	J(+) if RPD = 25-60% (Pest/Aroclor); 40-60% (Herb/Phenol) NJ(+) using PJ if RPD > 60% R(+) using PJ if RPD >90%	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	QAPP specified RPD < 50% (sediment & water)	Narrate; do not qualify.	na

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Integral - Portland Harbor Site
Metals by ICP (Based on Inorganic NFG 1994 & 2002)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler Temperature and Preservation	4°C ±2° Water Only: Nitric Acid to pH < 2 For Dissolved metals, 0.45 um filter preserve after filtration	Professional Judgment J(+)/UJ(-) if preservation requirements are not met	1
Holding Time	180 days	Professional Judgment J(+)/UJ(-)	1
Initial Calibration	Blank + minimum 1 standard once every 24 hours if more than 1 standard r>0.995	Professional Judgment J(+)/UJ(-) if r<0.995 (multi point cal)	5A
Initial Calibration Verification (ICV)	Independent source analyzed immed. after cal. %R within +/- 10% of true value	Professional Judgment J(+)/UJ(-) if %R 75%-89% J(+) if %R = 111-125% R(+) if %R > 125% R(+/-) if %R < 75%	5A
Continuing Cal Verification (CCV)	Every ten samples, immed. Before samples+ and end of run %R within +/- 10% of true value	Professional Judgment J(+)/UJ(-) if %R = 75%-89% J(+) if %R 111-125% R(+) if %R > 125% R(+/-) if %R < 75%	5B
CRI Standard (to check CRDL)	2X CRDL (or 2X IDL if greater) analyzed beginning and end of run (at least 8 hrs) Not required for Al, Ba, Ca, Fe, Mg, Na, K %R = 70%-130% (50%-150% Sb, Pb, Tl)	Professional Judgment R(-),(+)<2XCRDL if %R <50% (< 30% Sb, Pb, Tl) J(+)<2XCRDL, UJ(-) if %R 50-69% (30%-49% Sb, Pb,Tl) J(+) <2X CRDL if %R 130%-180% (150%-200%Sb, Pb, Tl) R(+)<2X CRDL if %R>180%(200% Sb, Pb, Tl)	14

Initial and Continuing Cal Blanks (ICB/CCB)	after each ICV and CCV
	every ten samples and end of run
	blank < +/- IDL
		Action level is 5x abs. value of blk conc.
		For (+) blk value, U(+) values < action level
		For (-) blk value, J(+)/UJ(-) values < action level
			7

Prep Blank	One per matrix per batch
	(not to exceed 20 samples)
		Action level is 5x abs. value of blk conc.
		For (+) blk value, U(+) values < action level
		For (-) blk value, J(+)/UJ(-) values < action level
			7

Interference Check Samples ICSA/ICSAB	Beginning and end of each run or every eight hours ICSAB +/- 20% ICSA < +/- IDL	For samp with Al,Ca,Fe,Mg > ICS levels R(+/-) if %R<50% J(+) if %R >120% J(+)/UJ(-) if %R= 50% to 79% Professional Judgment ICSA	17
Post Digestion Spike	If ICP Matrix Spike is outside 75-125%, spike at twice the sample conc.	No Quals assigned based on this element	

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Integral - Portland Harbor Site
Metals by ICP (Based on Inorganic NFG 1994 & 2002)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Matrix Spike	One per matrix per batch 75-125% for samples less than 4 x spike level	J(+) if %R>125% J(+)/UJ(-) if %R <75% J(+)/R(-) if %R<30%	8
Laboratory Duplicate	One per matrix per batch RPD <20% for samples > 5x CRDL Diff <CRDL for samples >CRDL and <5 x CRDL (may use RPD < 35%, Diff < 2X CRDL for solids)	J(+)/UJ(-) if RPD > 20% or diff > CRDL	9
Serial Dilution	5x dilution one per matrix %D <10% for values > 50x IDL	J(+)/UJ(-) if %D >10%	16
Laboratory Control Sample	Waters: One per matrix per batch %R (80-120%)	R(+/-) if %R < 50% J(+)/UJ(-) if %R = 50-79% J(+) if %R >120%	10
	Soils: One per matrix per batch Result within manufacturer's certified acceptance range	J(+)/UJ(-) if < LCL, J(+) if > UCL	10
Field Blanks	No results > QL	Apply 5X rule; U(+) < action level	6
Field Duplicates	QAPP specified RPD < 50% (sediment & water)	Narrate; do not qualify.	na
Instrument Detection Limit	determined every 3 months	Professional Judgment	14
Linear Range	determined yearly samples diluted to fall within range	J(+) values over range	20

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Integral - Portland Harbor Site
Metals by ICP-MS (Based on Inorganic NFG 1994 & 2002)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler Temperature and Preservation	4°C ±2° Water Only: Nitric Acid to pH < 2 For Dissolved metals, 0.45 um filter preserve after filtration	Professional Judgment J(+)/UJ(-) if preservation requirements are not met	1
Holding Time	180 days	Professional Judgment J(+)/UJ(-) if holding time exceeded J(+)/R(-) if HT exceeded by 3x	1
Tune	Prior to ICAL Analyzed 5 times wih Std Dev. ≤ 5% mass calibration <0.1 amu from True Value Resolution < 0.9 AMU @ 10% peak height or <0.75 amu @ 5% peak height	Professional Judgment No Tune - R all results criteria not met - J(+)/UJ(-)	5A
Initial Calibration	Mininum Blank+1 Standard every 24 hours	Professional Judgment J(+)/UJ(-) >24 hours J(+)/UJ(-) if r<0.995 (for multi point cal)	5A
Initial Calibration Verification (ICV)	Independent source; analyzed post ICAL and prior to samples +/-10% of the True value	Professional Judgment J(+)/UJ(-) if %R 75%-89% J(+) if %R = 111-125% R(+) if %R > 125% R(+/-) if %R < 75%	5A
Continuing Cal Verification (CCV)	Every 10 samples,post ICV/ICB and end of run +/- 10% of True value	professional judgment J(+)/UJ(-) if %R 75%-89% J(+) if %R = 111-125% R(+) if %R > 125% R(+/-) if %R < 75%	5B
CRDL Standard (CRI)	2X CRDL (or 2X IDL if greater) analyzed beginning and end of run (at least 8 hrs) Not required for Al, Ba, Ca, Fe, Mg, Na, K %R = 70%-130% (50%-150% Co,Mn, Zn)	Professional judgment R(-),(+)<2XCRDL if %R <50% (< 30% Co,Mn, Zn) J(+)<2XCRDL, UJ(-) if %R 50-69% (30%-49% Co,Mn, Zn) J(+) <2X CRDL if %R 130%-180% (150%-200% Co,Mn, Zn) R(+)<2X CRDL if %R>180%(200% Co,Mn, Zn)	14

Initial and Continuing Cal Blanks (ICB/CCB)	after each ICV and CCV every ten samples and end of run blank < +/- IDL	Action level is 5x abs. value of blk conc.
		For (+) blk value, U(+) values < AL
		For (-) blk value, J(+)/UJ(-) values < AL
			7

Prep Blank	One per matrix per batch (not to exceed 20 samples)	Action level is 5x abs. value of blk conc.
		For (+) blk value, U(+) values < AL
		For (-) blk value, J(+)/UJ(-) values < AL
			7

Field Blanks	No results > QL	Apply 5X rule; U(+) < action level	6

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Integral - Portland Harbor Site
Metals by ICP-MS (Based on Inorganic NFG 1994 & 2002)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Interference Check Samples ICSA/ICSAB	ICSAB +/- 20% of true value ICSA < +/- IDL	Where Al,Ca,Fe,Mg = ICS levels J(+) if %R >120% J(+)/UJ(-) if %R = 50% to 79% R(+/-) if %R<50% Professional Judgment for ICSA > +/- IDL	17
Post Digestion Spike	If ICP Matrix Spike is outside 75-125% Spike parent sample at 2X the sample conc.	Use Professional Judgment - usually no action	14
Matrix Spike	One per matrix, batch and SDG 75-125% for samples where results do not exceed 4x spike level	J (+) if %R > 125% J(+)/UJ(-) if %R < 75% J(+)/R(-) if %R < 30% UJ(-) if %R = 30-74%	8
Laboratory Duplicate	One per matrix per batch RPD <20% for samples > 5x CRDL Diff<CRDL for samples >CRDL and <5 x CRDL (may use RPD < 35%, Diff < 2X CRDL for solids)	J(+)/UJ(-) associated samples if RPD > 20% or diff > CRDL	9
Laboratory Control Sample	Waters: One per matrix per batch %R (80-120%)	R(+/-) if %R < 50% J(+)/UJ(-) if %R = 50-79% J(+) if %R >120%	10
	Soils: One per matrix per batch result within manufaturer's certified acceptance range	J(+)/UJ(-) if < LCL, J(+) if > UCL	10
Serial Dilution	5x dilution one per matrix (or SDG) %D <10% of the undiluted value for values > 50x IDL	J(+)/UJ(-) if %D >10%	16
Field Duplicates	QAPP specified RPD < 50% (sediment & water)	Narrate; do not qualify.	na
Internal Standards	Every Sample 60%-125% of ICAL IS	J (+)/UJ (-) analytes associated with IS outlier	19
Instrument Detection Limit	Determined every 3 months	Professional Judgment	14
Linear Range	determined yearly samples diluted to fall within range	J(+) values over range	20

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Integral - Portland Harbor Site
Mercury by CVAA (Based on Inorganic NFG 1994 & 2002)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler Temperature and Preservation	4°C ±2° Water Only: Nitric Acid to pH < 2 For Dissolved metals, 0.45 um filter preserve after filtration	Professional Judgment J(+)/UJ(-) if preservation requirements are not met	1
Holding Time	28 days from date sampled	Professional Judgment J(+)/UJ(-) if holding time exceeded	1
Initial Calibration	Blank + 4 standards r > 0.995 once every 24 hours	Professional Judgment J(+)/UJ(-) if r<0.995	5A
Initial Calibration Verification (ICV)	Independent source analyzed immediately after cal. %R within +/- 20% of true value	Professional Judgment R(+/-) if %R < 65% R(+) if %R > 135% J(+)/UJ(-) if %R = 65%-79% J(+) if %R = 121-135%	5A
Continuing Cal Verification (CCV)	Every ten samples, immed. following ICV/ICB and end of run %R within +/- 20% of true value	R(+/-) if %R < 65% R(+) if %R > 135% J(+)/UJ(-) if %R = 65%-79% J(+) if %R = 121-135%	5B
CRDL Standard (CRA)	Beginning of run after ICV/ICB CCV/CCB Conc = CRDL 70% - 130%	Professional Judgment R(-),(+)<2XCRDL if %R <50% J(+)<2XCRDL, UJ(-) if %R 50-69% J(+) <2X CRDL if %R 130%-180% R(+)<2X CRDL if %R>180%	14

Initial and Continuing Cal Blanks (ICB/CCB)	After each ICV and CCV
	every ten samples and end of run
	blank < +/- IDL
		Action level is 5x abs. value of blk conc.
		For (+) blk value, U(+) sample values < AL
		For (-) blk value, J(+)/UJ(-) sample values < AL
			7

Prep Blank	One per matrix per batch
	(not to exceed 20 samples)
		Action level is 5x abs. value of blk conc.
		For (+) blk value, U(+) sample values < AL
		For (-) blk value, J(+)/UJ(-) sample values < AL
			7

Matrix Spike	One per matrix per batch 5% frequency 75-125% for samples less than 4x spike level	J(+) if %R > 125% J(+)/UJ(-) if %R < 75% J(+)/R(-) if %R < 30%	8
Laboratory Duplicate	One per matrix per batch RPD <20% for samples > 5x CRDL (+/-)CRDL for samples >CRDL and <5 x CRDL (may use RPD < 35%, Diff < 2X CRDL for solids)	J(+)/UJ(-) if RPD > 20% or diff > CRDL	9

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VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Laboratory Control Sample	Waters: One per matrix per batch %R (80-120%)	R(+/-) if %R < 50%; J(+) if %R >120% J(+)/UJ(-) if %R = 50-79%	10
	Soils: One per matrix per batch Result within manufacturer's certified acceptance range	J(+)/UJ(-) if < LCL, J(+) if > UCL	10
Field Duplicates	No specific QAPP limits Use RPD < 35% (water) or < 50% (soil)	Narrate; do not qualify.	na
Field Duplicates	QAPP specified RPD < 50% (sediment & water)	J(+)/UJ(-) in parent samples only	9

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