EcoChem, INC.
Environmental Data Quality

DATA QUALITY EVALUATION

PORTLAND HARBOR

Round 3A Surface Water – High Flow Event, 2007

Peristaltic Pump Sampling

Semivolatile Organic Compounds (SVOC) - Method SW8270C
Polycyclic Aromatic Hydrocarbons (PAH) - Method SW8270-SIM
Phthalate Esters – Method 525.2
Chlorinated Herbicides - Method SW8151A
Butyltins - Krone Method
Metals - Methods SW6020 & SW7470A
Conventionals - Methods 160.1, 160.2, 415.1 & 6010B
Hexavalent Chromium – Method SW7196A

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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 and/or Inorganic Data Review* (USEPA 1994, 1999 & 2002).

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 sample delivery group (SDG) and will be kept on file at EcoChem.

PROCESS FOR DATA VALIDATION

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

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

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

Ten percent (10%) of all surface water 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: SAMPLE IDENTIFICATION

Two samples were collected and originally reported using identifications that previously existed in this sampling event. All records for both were renamed as follows to distinguish them in the client database.

Sample LW3-W4023-M was originally collected on 1/15, and recollected on 3/02. The original sample data was validated in SDG K0700440. The recollected sample was validated in SDG K0701738 and renamed in the EDD (sys_sample_code field) as LW3-W4023-M2 to differentiate from the original.

Sample LW3-W4025-M was originally collected on 1/15, and recollected on 3/10. The original sample data was validated in SDG K0700440. The recollected sample was validated in SDG K0702017 and renamed in the EDD (sys_sample_code field) as LW3-W4025-M2 to differentiate from the original.

SUMMARY OF DATA VALIDATION: SEMIVOLATILE ORGANIC COMPOUNDS

A total of 40 peristaltic pump samples were analyzed for semivolatile organic compounds (SVOC) for this Portland Harbor Surface Water High Flow sampling event. Two field duplicate samples and one rinsate blank were collected to monitor the field collection process and three system blanks were also analyzed. Columbia Analytical Services, Kelso, Washington completed the SVOC analyses.

The SVOC data for the High Flow samples were generally acceptable. Two data points (0.1% of SVOC results) were rejected. Rejected data must not be used for any purpose. A total of 110 data points (5.2% of SVOC results) were qualified as estimated because control limits were exceeded in one or more laboratory QC samples or 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 High Flow SVOC analyses.

Holding Times and Sample Preservation

The holding time requirements were met for all surface water samples and associated field QC samples.

The sample preservation requirements (cooler temperature of 4°C ±2°) were not met for many samples. Some of the coolers were received at the laboratory at temperatures below the control limits. These temperature outliers did not impact data quality and no qualifiers were required.

Instrument Performance

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

Method Blank Analyses

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

Various target analytes were detected in the method blanks. A total of seven results (0.3% of all SVOC 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 (surrogate compound, laboratory control sample [LCS] and matrix spike [MS] recoveries) and precision (duplicate LCS and MS analyses).

Surrogate Compound Recovery

Surrogate compounds were added to all field and QC samples. The surrogate recovery values from one sample were below control limits. All 46 results and reporting limits (2.2% of all SVOC results) were qualified as estimated (J/UJ) with potential low bias.

Matrix Spike Recovery

MS analyses were not performed with some data sets. For these sets, laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses were used to assess accuracy and precision. The recovery values of aniline and 3,3'-dichlorobenzidine from four MS/MSD sets were below the control limits. Reporting limits for these compounds (0.4% of all SVOC results) in the parent sample were qualified as estimated (UJ) with potential low bias.

Laboratory Control Sample Recovery

LCS/LCSD analyses met the criteria for frequency of analysis. The recovery of 2,4-dinitrophenol from one LCS/LCSD set was less than 10%. Reporting limits for this compound were rejected for potential very low bias in the two associated samples. A total of two results (0.1% of all High Flow SVOC results) were rejected for accuracy. The recovery values of numerous other analytes from LCS analyses were below the control limits. Reporting limits for these compounds (2.7% of all SVOC results) in the associated samples were qualified as estimated (UJ) with potential low bias.

Precision

LCS/LCSD and MS/MSD analyses were evaluated for laboratory precision. Relative percent difference (RPD) values for numerous analytes were greater than the control limits. One result for benzoic acid (0.05% of all SVOC results) was qualified as estimated (J) for precision.

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 0.0074 to 1.9 µg/L for the non-detected results. The laboratory reporting limits met the target MRL stated in *Round 2 QAPP, Addendum 1: Surface Water*, with the following exception.

The MRL for n-nitrosodimethylamine is 0.002 µg/L; however the laboratory reporting limit was 2.0 µg/L.

Field Quality Control Samples

Field QC samples collected for the SVOC analysis included field duplicate, field blank and system blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

One field blank (LW3-W4902) was associated with all samples. A positive result for 2-methylphenol was reported in this field blank, but not detected in the associated samples. No qualifiers were required.

System Blanks

Three system blanks (Decon Blank, Lab Blank, and LW3-W4-FLAB-RB) were generated and analyzed with the peristaltic pump samples. A positive result for phenol was reported in LW3-W4-FLAB-RB. There was no direct association between these blanks and other field samples. No data were qualified based on system blank contamination.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB) were submitted. All RPD values met the criteria for acceptable precision. The field duplicates are discussed in more detail in the data validation reports.

SUMMARY OF DATA VALIDATION: POLYCYCLIC AROMATIC HYDROCARBONS

A total of 40 peristaltic pump samples were analyzed for semivolatile polycyclic aromatic hydrocarbon compounds (PAH) for the Portland Harbor Surface Water High flow sampling event. Two field duplicate samples and one rinsate blank were collected to monitor the field collection process and three system blanks were also analyzed. Columbia Analytical Services, Kelso, Washington completed the PAH analyses.

The PAH data for the High Flow samples were acceptable. No data points were rejected. Two data points (0.2% 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 surface water PAH analyses.

Holding Times and Sample Preservation

The holding time requirements were met for all surface water samples and associated field QC samples.

The sample preservation requirements (cooler temperature of 4°C ±2°) were not met for many samples. Some of the coolers were received at the laboratory at temperatures below the control limits. These temperature outliers did not impact data quality and no qualifiers were required.

Instrument Performance

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

Method Blank Analyses

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. No target analytes were detected in the method blanks.

Accuracy

The accuracy of the analytical results is evaluated in the following sections in terms of analytical bias (surrogate compound, laboratory control sample [LCS] and matrix spike [MS] recoveries) and precision (duplicate LCS and MS analyses).

Surrogate Compound Recovery

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

Matrix Spike Recovery

MS analyses were not performed with some data sets. For these sets, laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses were used to assess accuracy and precision. All reported recoveries met the criteria for acceptable performance.

Laboratory Control Sample Recovery

LCS/LCSD analyses met the criteria for frequency of analysis. The recovery values reported by the laboratory met the criteria for acceptable performance.

Precision

LCS/LCSD and MS/MSD analyses were evaluated for laboratory precision. Relative percent difference (RPD) values for naphthalene and 2-methylnaphthalene were greater than the control limits. One result for each (0.2% of all PAH results) were qualified as estimated (J) for precision.

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 0.0023 to 0.013 µg/L for the non-detected results. The laboratory reporting limits met the target MRL stated in *Round 2 QAPP, Addendum 1: Surface Water*.

Field Quality Control Samples

Field QC samples collected for the PAH analysis included field duplicate, field blank and system blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

One field blank (LW3-W4902) was associated with all samples. No target analytes were detected in the field blank.

System Blanks

Three system blanks (Decon Blank, Lab Blank, and LW3-W4-FLAB-RB) were generated and analyzed with the peristaltic pump samples. There was no direct association between these blanks and other field samples. No target analytes were detected in the system blanks.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB) were submitted. All RPD values met the criteria for acceptable precision. The field duplicates are discussed in more detail in the data validation reports.

SUMMARY OF DATA VALIDATION: PHTHALATE ESTERS

A total of 40 peristaltic pump samples were analyzed for semivolatile phthalate ester compounds (phthalates) for the Portland Harbor Surface Water High flow sampling event. Two field duplicate samples and one rinsate blank were collected to monitor the field collection process and three system blanks were also analyzed. Columbia Analytical Services, Kelso, Washington completed the analyses.

The phthalate data for the High Flow samples were generally acceptable. No data points were rejected. Seventy-five data points (27% of all phthalate results) were qualified as estimated because control limits were exceeded in one or more laboratory quality control (QC) samples or 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 100% complete for the surface water phthalate analyses.

Holding Times and Sample Preservation

The holding time requirements were exceeded for four surface water samples. A total of 24 results (8.7% of all phthalate results) were qualified as estimated (J/UJ) based on holding time outliers.

The sample preservation requirements (cooler temperature of 4°C ±2°) were not met for many samples. Some of the coolers were received at the laboratory at temperatures below the control limits. These temperature outliers did not impact data quality and no qualifiers were required.

Instrument Performance

Initial and continuing calibrations were completed for all target analytes and met the criteria for frequency of analysis. All initial calibration analyses met linearity and recovery acceptance criteria. No qualifiers were required based on continuing calibration outliers.

Method Blank Analyses

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blanks are used to evaluate all associated samples. Various target analytes were detected in the method blanks. A total of 25 results (9.1% of all phthalate results) were qualified as estimated (J) and 37 results (13% of all phthalate 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 (surrogate compound, laboratory control sample [LCS] and matrix spike [MS] recoveries) and precision (duplicate LCS and MS analyses).

Surrogate Compound Recovery

Surrogate compounds were added to all field and QC samples. All sample surrogate recovery values reported by the laboratory met the criteria for acceptable performance.

Matrix Spike Recovery

MS analyses met the criteria for frequency of analysis. The recovery of bis (2-ethylhexyl) phthalate from one MS was below the control limit. One result for this compound (0.4% of all phthalate results) in the parent sample was qualified as estimated (J) with potential low bias.

Laboratory Control Sample Recovery

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

Precision

Duplicate analyses were not performed. There was no routine measurement of laboratory precision.

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 0.005 to 0.098 µg/L for the non-detected results. The laboratory reporting limits (MRL) met the target MRL stated in *Round 2 QAPP, Supplement 1 to Addendum 1: Surface Water*, with the exception of the limit for di-n-octylphthalate (0.2 ug/L), which exceeded the target MRL of 0.1 ug/L.

Field Quality Control Samples

Field QC samples collected for the phthalate analysis included field duplicate, field blank and system blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

One field blank (LW3-W4902) was associated with all samples. Numerous target analytes were reported in these field blanks. A total of 60 results (22% of all phthalate results) were qualified as estimated (J) and 81 results (29% of all phthalate results) were qualified as not detected (U)based on field blank contamination.

System Blanks

Three system blanks (Decon Blank, Lab Blank, and LW3-W4-FLAB-RB) were generated and analyzed with the peristaltic pump samples. Target analytes were reported in the all three system blanks. There was no direct association between these blanks and other field samples. No data were qualified based on system blank contamination.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB) were submitted. The RPD value for bis(2-ethylhexyl)phthalate from the former pair exceeded the criteria for acceptable precision. The field duplicates are discussed in more detail in the data validation reports.

SUMMARY OF DATA VALIDATION: CHLORINATED HERBICIDES

A total of 40 peristaltic pump samples were analyzed for chlorinated herbicide compounds for the Portland Harbor Surface Water High flow sampling event. Two field duplicate samples and one rinsate blank were collected to monitor the field collection process and three system blanks were also analyzed. Columbia Analytical Services, Kelso, Washington performed the herbicide analyses.

The herbicide data for the High flow samples were generally acceptable. No data points were rejected. Four data points (0.9% of all herbicide results) were qualified as estimated or tentatively identified because control limits were exceeded in one or more laboratory quality control (QC) samples or 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 100% complete for the surface water herbicide analyses.

Holding Times and Sample Preservation

The holding time requirements were met for all surface water samples and associated field QC samples.

The sample preservation requirements (cooler temperature of 4°C ±2°) were not met for many samples. Some of the coolers were received at the laboratory at temperatures below the control limits. These temperature outliers did not impact data quality and no qualifiers were required.

Instrument Performance

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

Method Blank Analyses

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. No target analytes were detected in any method blank.

Accuracy

The accuracy of the analytical results is evaluated in the following sections in terms of analytical bias (surrogate compound, laboratory control sample [LCS] and matrix spike [MS] recoveries) and precision (duplicate LCS and MS analyses).

Surrogate Compound Recovery

A surrogate compound was added to all field and QC samples. The recoveries reported by the laboratory met the criteria for acceptable performance.

Matrix Spike Recovery

MS/MSD analyses were not performed with some data sets. For these sets, laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses were used to assess accuracy and precision. All reported recovery values met the criteria for acceptable performance.

Laboratory Control Sample Recovery

LCS/LCSD analyses met the criteria for frequency of analysis. The recovery value reported by the laboratory met the criteria for acceptable performance.

Precision

LCS/LCSD and MS/MSD analyses were evaluated for laboratory precision. All of the relative percent difference (RPD) values were acceptable.

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 0.034 to 43 µg/L for the non-detected results. The laboratory reporting limits met the target MRL stated in *Round 2 QAPP, Addendum 1: Surface Water*, except four samples which were analyzed at dilution (factor 5), resulting in elevated MDL and MRL values.

Compound Identification

The results from the two analytical columns were compared for agreement. In cases where the percent difference (%D) value between the two columns was greater than 40% the reported result was "P" flagged by the laboratory. As an elevated %D value may indicate the presence of an interferent resulting in a high bias, the associated results were estimated (J). If the %D value was greater than 60%, the result was qualified as a tentative identification (NJ). Three data points (0.7% of herbicide results) were qualified as estimated (J) and one data point (0.2%) was qualified as tentative identification (NJ).

Field Quality Control Samples

Field QC samples collected for the herbicide analysis included field duplicate, field blank and system blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

One field blank (LW3-W4902) was associated with the samples. No target analytes were detected in any field blank.

System Blanks

Three system blanks (Decon Blank, Lab Blank, and LW3-W4-FLAB-RB) were generated and analyzed with the peristaltic pump samples. No target analytes were detected in any system blank.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB) were submitted. All RPD values met the criteria for acceptable precision. The field duplicates are discussed in more detail in the data validation reports.

SUMMARY OF DATA VALIDATION: BUTYLTINS

A total of 40 peristaltic pump samples were analyzed for butyltin compounds for the Portland Harbor Surface Water High flow sampling event. Two field duplicate samples and one rinsate blank were collected to monitor the field collection process and three system blanks were also analyzed. Columbia Analytical Services, Kelso, Washington performed the butyltin analyses.

The butyltin data for the surface water samples were generally acceptable. No data points were rejected. Two data points (1.1% of all butyltin results) were qualified as estimated because control limits were exceeded in one or more laboratory quality control (QC) samples or 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 100% complete for the surface water butyltin analyses.

Holding Times and Sample Preservation

The holding time requirements were met for all surface water samples and associated field QC samples.

The sample preservation requirements (cooler temperature of 4°C ±2°) were not met for many samples. Some of the coolers were received at the laboratory at temperatures below the control limits. These temperature outliers did not impact data quality and no qualifiers were required.

Instrument Performance

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

Method Blank Analyses

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.

The target analytes n-butyltin and di-n-butyltin were detected in the method blank. A total of 88 di-n-butyltin results (48% of all butyltin 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 (surrogate compound, laboratory control sample [LCS] and matrix spike [MS] recoveries) and precision (duplicate LCS and MS analyses).

Surrogate Compound Recovery

A surrogate compound was added to all field and QC samples. The surrogate recovery from one sample was above the control limit. No qualifiers were required since no target compounds were detected in the sample despite the potential high bias.

Matrix Spike Recovery

MS/MSD analyses met the criteria for frequency of analysis. Several recovery values exceeded the control limit. No qualifiers were required since the affected compounds were not detected in the sample despite the potential high bias, or were detected at levels greater than four times the spiking amount.

Laboratory Control Sample Recovery

LCS/LCSD analyses met the criteria for frequency of analysis. Several recovery values exceeded the control limit. Two results for tri-n-butyltin (1.1% of all butyltin results) were qualified as estimated (J-10) due to potential high bias.

Precision

LCS/LCSD and MS/MSD analyses were evaluated for laboratory precision. The relative percent differences (RPD) for three compounds were above the control limits. No qualifiers were required since these compounds were not detected in the associated samples.

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 0.0006 to 0.0017 µg/L for the non-detected results. The laboratory reporting limits met the target MRL stated in *Round 2 QAPP, Addendum 1: Surface Water*.

Field Quality Control Samples

Field QC samples collected for the butyltin analysis included field duplicate, field blank and system blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

One field blank (LW3-W4902) was associated with the samples. No target analytes were detected in the field blank.

System Blanks

Three system blanks (Decon Blank, Lab Blank, and LW3-W4-FLAB-RB) were generated and analyzed with the peristaltic pump samples. The target analyte n-butyltin was detected in both Decon Blank and Lab Blank. There was no direct association between these blanks and other field samples. No data were qualified based on system blank contamination.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB) were submitted. All RPD values met the criteria for acceptable precision. The field duplicates are discussed in more detail in the data validation reports.

SUMMARY OF DATA VALIDATION: METALS

A total of 40 peristaltic pump samples were analyzed for total and dissolved metals for the Portland Harbor Surface Water High flow sampling event. Two field duplicate samples and one rinsate blank were collected to monitor the field collection process and three system blanks were also analyzed. Columbia Analytical Services, Kelso, Washington, performed all analyses. The following analytical methods were used:

Parameter	Method
ICP-MS Metals	SW6020
Mercury	SW7470A

The metals data for the surface water samples were generally acceptable. No data points were rejected. A total of 106 data points (8.9% of all metals results) were qualified as estimated because control limits were exceeded in one or more laboratory quality control (QC) samples or 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 100% complete for the surface water metals analyses.

Holding Times and Sample Preservation

The holding time requirements were met for all surface water samples and associated field QC samples.

The sample preservation requirements (cooler temperature of 4°C ±2°) were not met for many samples. Some of the coolers were received at the laboratory at temperatures below the control limits. These temperature outliers did not impact data quality and no qualifiers were required.

Instrument Performance

Initial and continuing calibrations were completed for all target analytes and met the criteria for frequency of analysis. All initial and continuing calibration analyses met linearity and recovery 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 total of 74 metals results (6.2% of all

metals results) were qualified as not detected (U) based on method blank contamination. The qualifiers were applied to aluminum (3 results), antimony (14 results), chromium (8 results), lead (3 results), nickel (7 results), silver (37 results) and zinc (2 results). A total of 21 metals results (1.8% of all metals results) were qualified as estimated (J/UJ) based on potential low bias exhibited in method blanks. The qualifiers were applied to mercury (3 results), chromium and nickel (5 results each), lead (12 results), and selenium (8 results).

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 (duplicate MS and laboratory duplicate analyses).

Matrix Spike Recovery

MS analyses met the criteria for frequency of analysis. The recovery of arsenic from one MS was below the control limit. Thirteen results for this compound (1.1% of all metals results) in associated samples were qualified as estimated (J) with potential low bias.

Laboratory Control Sample Recovery

LCS analyses met the criteria for frequency of analysis. All recovery values met the criteria for acceptable performance.

Contract Required Detection Limit Standard Analyses

CRDL standards were analyzed at the beginning of each analytical sequence. For recovery values 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 recovery values 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. CRDL standard outliers were reported, with high bias for chromium, copper, and zinc and low bias for silver. Eleven reporting limits for silver, two results each for chromium and zinc, and one result for copper (1.3% of all metals results) were qualified as estimated (J/UJ) for CRDL 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 exceeding 10% may indicate the presence of matrix interference, resulting in potential bias. Recoveries of aluminum and arsenic exceeded the control limit. Nine results and reporting limits for aluminum and 46 results and reporting limits for arsenic (4.6% of all metals results) were qualified as estimated (J/UJ) for serial dilution outliers.

Precision

MS/MSD analyses were evaluated for laboratory precision. For mercury analyses, laboratory duplicate analyses were evaluated instead of MS/MSD analyses. All relative percent difference (RPD) values were acceptable.

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 0.003 µg/L to 0.7 µg/L for the non-detected results (0.02 µg/L for mercury). The laboratory reporting limits met the target MRL stated in *Round 2 QAPP, Addendum 1: Surface Water*, with the exception of several reporting limits for total aluminum, which exceeded the target MRL by a factor of 2.

Field Quality Control Samples

Field QC samples collected for the metals analysis included field duplicate, field blank and system blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

One field blank (LW3-W4902) was associated with the High Flow samples. Numerous target compounds were reported in this field blank. A total of 66 dissolved results - 23 for chromium and 43 for zinc - were qualified as not detected (U) based on field blank contamination (5.6% of all metals results).

System Blanks

Three system blanks (Decon Blank, Lab Blank, and LW3-W4-FLAB-RB) were generated and analyzed with the peristaltic pump samples. Positive results for aluminum, chromium, copper, lead, and zinc were detected in these blanks. There was no direct association between these blanks and other field samples. No data were qualified based on system blank contamination.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB) were submitted. The RPD and absolute difference values for dissolved aluminum and lead from the latter pair exceeded the criteria for acceptable precision. The field duplicates are discussed in more detail in the data validation reports.

SUMMARY OF DATA VALIDATION: GENERAL CHEMISTRY

A total of 40 peristaltic pump samples were analyzed for some or all of the following general chemistry parameters for the Portland Harbor Surface Water High flow sampling event. Two field duplicate samples and one rinsate blank were collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington, performed all analyses. The following analytical methods were used:

Parameter	Method
Total Dissolved Solids (TDS)	EPA 160.1
Total Suspended Solids (TSS)	EPA 160.2
Total Organic Carbon (TOC)	EPA 415.1
Dissolved Organic Carbon (DOC)	EPA 415.1
Hardness as $CaCO_3$	SW6010B / SM2340B
Perchlorate	EPA 314.0

Data for the general chemistry parameters analysis for the surface water samples were acceptable. No data were rejected. A total of 21 data points (6.9% of all general chemistry results) were qualified as estimated because control limits were exceeded in one or more laboratory quality control (QC) samples or 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 100% complete for the surface water general chemistry parameters analyses.

Holding Times and Sample Preservation

Analyses for total dissolved solids (TDS) and total suspended solids (TSS) were performed outside the QAPP specified 7 day holding time. Seven TDS results and 14 TSS results (6.9% of all general chemistry results) were qualified as estimated (J).

The sample preservation requirements (cooler temperature of 4°C ±2°) were not met for many samples. Some of the coolers were received at the laboratory at temperatures below the control limits. These temperature outliers did not impact data quality and no qualifiers were required.

Instrument Performance

Initial and continuing calibrations were completed for the TOC, DOC, hardness, and perchlorate analyses and met the criteria for frequency of analysis. The initial calibrations met the linearity (percent relative standard deviation or correlation coefficient) control limits.

Method Blank Analyses

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. No target analytes were detected in any method blank.

Accuracy

The accuracy of the analytical results is evaluated in the following sections in terms of analytical bias (matrix spike [MS] and laboratory control sample [LCS] recoveries) and precision (sample duplicate analyses).

Matrix Spike Recovery

MS analyses were evaluated for accuracy of the TOC and DOC analyses. All MS recovery values were acceptable.

Laboratory Control Sample Recovery

LCS analyses met the frequency criteria for all analyses. All of the LCS recovery values were acceptable.

Precision

Duplicate sample analyses were evaluated for laboratory precision. All precision measurements were acceptable.

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. The laboratory reporting limits met the target MRL for general chemistry parameters stated in *Round 2 QAPP, Addendum 1: Surface Water*.

Field Quality Control Samples

Field QC samples collected for the general chemistry analyses included field duplicate and field blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

One field blank (LW3-W4902) was associated with the High Flow samples. No target analytes were detected in the field blank.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB) were submitted. All RPD values met the criteria for acceptable precision. The field duplicates are discussed in more detail in the data validation reports.

SUMMARY OF DATA VALIDATION: HEXAVALENT CHROMIUM

Six peristaltic pump samples were analyzed for hexavalent chromium for the Portland Harbor Surface Water High flow sampling event. One rinsate blank was collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington, performed the analyses.

Data for the hexavalent chromium analysis for the surface water samples were generally acceptable. No data were rejected. Two data points (1.1% of all butyltin results) were qualified as estimated because control limits were exceeded in one or more laboratory quality control (QC) samples or 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 quality control (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 surface water hexavalent chromium analysis.

Holding Times and Sample Preservation

All analyses were performed outside the QAPP specified 24 hour holding time. All nine results and reporting limits (100% of hexavalent chromium data) were qualified as estimated (UJ).

The sample preservation requirements (cooler temperature of 4°C ±2°) were not met for many samples. Some of the coolers were received at the laboratory at temperatures below the control limits. These temperature outliers did not impact data quality and no qualifiers were required.

Instrument Performance

Initial and continuing calibrations were completed and met the criteria for frequency of analysis. The initial calibrations met the linearity (percent relative standard deviation or correlation coefficient) control limits.

Method Blank Analyses

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. Hexavalent chromium was detected in the method blank. Three results (33% of hexavalent chromium 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 (matrix spike [MS] and laboratory control sample [LCS] recoveries) and precision (sample duplicate analyses).

Matrix Spike Recovery

MS analyses were evaluated for accuracy. All MS recovery values were acceptable.

Laboratory Control Sample Recovery

LCS analyses met the frequency criteria. All of the LCS recovery values were acceptable.

Precision

Duplicate sample analyses were evaluated for laboratory precision. All precision measurements were acceptable.

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. The laboratory reporting limit of 2 µg/L for the non-detected results exceeded the target MRL stated in *Round 2 QAPP, Addendum 1: Surface Water*.

Field Quality Control Samples

Field QC samples collected for the hexavalent chromium analysis included a field blank sample. The results for the field QC sample are discussed in the following section.

Field Blanks

One field blank (LW3-W4902) was associated with the samples. No target analyte was detected in the field blank.

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
Semivolatile Compounds by GC/MS (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	Water: J(+)/UJ(-) if ext. > 7 and < 21 days J(+)/R(-) if ext > 21 days (EcoChem PJ) Solids/Wastes: J(+)/UJ(-) if ext. > 14 and < 42 days J(+)/R(-) if ext. > 42 days (EcoChem PJ) J(+)/UJ(-) if analysis >40 days	1
Tuning	DFTPP Beginning of each 12 hour period Method acceptance criteria	R(+/-) all analytes in all samples associated with the tune	5A
Initial Calibration (Minimum 5 stds.)	RRF > 0.05	(EcoChem PJ, see TM-06) If MDL= reporting limit: J(+)/R(-) if RRF < 0.05 If reporting limit > MDL: note in worksheet if RRF <0.05	5A
	%RSD < 30%	(EcoChem PJ, see TM-06) J(+) if %RSD > 30%	5A
Continuing Calibration (Prior to each 12 hr. shift)	RRF > 0.05	(EcoChem PJ, see TM-06) If MDL= reporting limit: J(+)/R(-) if RRF < 0.05 If reporting limit > MDL: note in worksheet if RRF <0.05	5B
	%D <25%	(EcoChem PJ, see TM-06) If > +/-90%: J+/R- If -90% to -26%: J+ (high bias) If 26% to 90%: J+/UJ- (low bias)	5B
Method Blank	One per matrix per batch No results > QL	U(+) if sample (+) result is less than QL and less than appropriate 5X or 10X rule (raise sample value to QL)	7
		U(+) if sample (+) result is greater than or equal to QL and less than appropriate 5X and 10X rule (at reported sample value)	7
	No TICs present	R(+) TICs using 10X rule	7
Field Blanks	No results > QL	Apply 5X/10X rule; U(+) < action level	6

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Integral - Portland Harbor Site
Semivolatile Compounds by GC/MS (Based on Organic NFG 1999)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
MS/MSD (recovery)	One per matrix per batch Use 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 per matrix per batch Use method acceptance criteria	J(+) if RPD > CL	9
LCS low conc. H2O SVOA	One per lab batch Within method control limits	J(+) assoc. cmpd if > UCL J(+)/R(-) assoc. cmpd if < LCL J(+)/R(-) all cmpds if half are < LCL	10
LCS regular SVOA (H2O & solid)	One per lab batch Lab or method control limits	J(+) if %R > UCL J(+)/UJ(-) if %R <LCL J(+)/R(-) if %R < 10% (EcoChem PJ)	10
LCS/LCSD *(if required)*	One set per matrix and batch of 20 samples RPD < 35%	J(+) assoc. cmpd. in all samples	9
Surrogates	Minimum of 3 acid and 3 base/neutral compounds Use method acceptance criteria	Do not qualify if only 1 acid and/or 1 B/N surrogate is out unless <10% J(+) if %R > UCL J(+)/UJ(-) if %R < LCL J(+)/R(-) if %R < 10%	13
Internal Standards	Added to all samples Acceptable Range: IS area 50% to 200% of CCAL area RT within 30 seconds of CC RT	J(+) if > 200% J(+)/UJ(-) if < 50% J(+)/R(-) if < 25% R T>30 seconds, narrate and Notify PM	19
Field Duplicates	QAPP specified RPD < 50% (sediment & water)	Narrate; do not qualify.	na
TICs	Major ions (>10%) in reference must be present in sample; intensities agree within 20%; check identification	R(+) common laboratory contaminants R(+) target compouds from other fractions See Technical Director for ID issues	4
Quantitation/ Identification	RRT within 0.06 of standard RRT Ion relative intensity within 20% of standard All ions in std. at > 10% intensity must be present in sample	See Technical Director if outliers	14 21 (false +)

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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 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	QAPP specified RPD < 50% (sediment & water)	Narrate; do not qualify.	na

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Butyltins by GC/FPD

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
Initial Calibration	%RSD<30% or correlation co-efficient >0.99	J(high bias), J/UJ(low bias)	5A
Continuing Calibration	%D < 25%	J(high bias), J/UJ(low bias)	5B
Method Blank	One per matrix per batch	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 > MRL	Same as Method Blank	7
Field Blanks	No results > QL	Apply 5X rule; U(+) < action level	6
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	tri-n-propyltin added to every sample %R = Laboratory control limits	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 ICAL CF Higher value from either column reported RPD between columns (40%)	J(+) if RPD = 40 - 60% NJ(+) if RPD >60% EcoChem PJ - See TM-08	3

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Butyltins by GC/FPD

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
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
Field Duplicates	QAPP specified RPD < 50% (sediment & water)	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	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 > 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 %Ds 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: 8290: J(+)/UJ(-) if %D = 20% - 75% J(+)/R(-) if %D > 75% 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 (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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Phthalates Analysis by GC/MS Method 525.2 (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	Water: J(+)/UJ(-) if ext. > 7 and < 21 days J(+)/R(-) if ext > 21 days (EcoChem PJ) Solids/Wastes: J(+)/UJ(-) if ext. > 14 and < 42 days J(+)/R(-) if ext. > 42 days (EcoChem PJ) J(+)/UJ(-) if analysis >40 days	1
Tuning	DFTPP Beginning of each 12 hour period Method acceptance criteria	R(+/-) all analytes in all samples associated with the tune	5A
Initial Calibration (Minimum 5 stds.)	RRF > 0.05	(EcoChem PJ, see TM-06) If MDL= reporting limit: J(+)/R(-) if RRF < 0.05 If reporting limit > MDL: note in worksheet if RRF <0.05	5A
	%RSD < 30%	(EcoChem PJ, see TM-06) J(+) if %RSD > 30%	5A
Continuing Calibration (Prior to each 12 hr. shift)	RRF > 0.05	(EcoChem PJ, see TM-06) If MDL= reporting limit: J(+)/R(-) if RRF < 0.05 If reporting limit > MDL: note in worksheet if RRF <0.05	5B
	%D <25%	(EcoChem PJ, see TM-06) If > +/-90%: J+/R- If -90% to -26%: J+ (high bias) If 26% to 90%: J+/UJ- (low bias)	5B
Method Blank	One per matrix per batch No results > QL	U (+) if result is < adjusted blank concentration. Use 2X for bis(2-ethylhexyl)phthalate J (+) if result is < 5X adjusted blank concentration. Use < 10X for bis(2-ethylhexyl)phthalate	7
Field Blank	No results > QL	Same as Method Blank	6

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Phthalates Analysis by GC/MS Method 525.2 (Based on Organic NFG 1999)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
MS/MSD (recovery)	One per matrix per batch Use 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 per matrix per batch Use method acceptance criteria	J(+) if RPD > CL	9
LCS low conc. H2O SVOA	One per lab batch Within method control limits	J(+) assoc. cmpd if > UCL J(+)/R(-) assoc. cmpd if < LCL J(+)/R(-) all cmpds if half are < LCL	10
LCS regular SVOA (H2O & solid)	One per lab batch Lab or method control limits	J(+) if %R > UCL J(+)/UJ(-) if %R <LCL J(+)/R(-) if %R < 10% (EcoChem PJ)	10
LCS/LCSD *(if required)*	One set per matrix and batch of 20 samples RPD < 35%	J(+) assoc. cmpd. in all samples	9
Surrogates	Minimum of 3 acid and 3 base/neutral compounds Use method acceptance criteria	Do not qualify if only 1 acid and/or 1 B/N surrogate is out unless <10% J(+) if %R > UCL J(+)/UJ(-) if %R < LCL J(+)/R(-) if %R < 10%	13
Internal Standards	Added to all samples Acceptable Range: IS area 50% to 200% of CCAL area RT within 30 seconds of CC RT	J(+) if > 200% J(+)/UJ(-) if < 50% J(+)/R(-) if < 25% R T>30 seconds, narrate and Notify PM	19
Field Duplicates	QAPP specified RPD < 50% (sediment & water)	Narrate; do not qualify.	na
TICs	Major ions (>10%) in reference must be present in sample; intensities agree within 20%; check identification	R(+) common laboratory contaminants R(+) target compouds from other fractions See Technical Director for ID issues	4
Quantitation/ Identification	RRT within 0.06 of standard RRT Ion relative intensity within 20% of standard All ions in std. at > 10% intensity must be present in sample	See Technical Director if outliers	14 21 (false +)

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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	
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

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

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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EcoChem Validation Guidelines for Conventional Chemistry Analysis
(Based on EPA Standard Methods)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler Temperature and Preservation	4°C ±2° Water: NaOH to pH > 12 (for CN)	J(+)/UJ(-) if preservation requirements not met EcoChem PJ	1
Holding Time	Method Specific	Professional Judgment J(+)/UJ(-) if holding time exceeded J(+)/R(-) if HT exceeded by > 3X	1
Initial Calibration	Method specific once every 24 hours One at CRDL r>0.995	Professional judgment J(+)/UJ(-) for r < 0.995	5A
Initial Calibration Verification (ICV)	Independent source analyzed immediately after cal. %R method specific	R(+/-) if %R sig < LCL J(+)/UJ(-) if %R < LCL J(+) if %R > UCL R(+) if %R sig > UCL	5A
Continuing Cal Verification (CCV)	Every ten samples, immed. following ICV/ICB and end of run %R method specific	R(+/-) if %R sig < LCL J(+)/UJ(-) if %R < LCL J(+) if %R > UCL R(+) if %R sig > UCL	5B
Initial and Continuing Cal Blanks (ICB/CCB)	After each ICV and CCV every ten samples and end of run blank < +/- IDL	For positive blk results: UJ(+) < 5X blk contamination For negative blk results: J(+)/UJ(-) < abs. value of 5X blk contamination	7
Prep Blank	One per matrix per batch (not to exceed 20 samples)	For positive blk results: UJ(+) < 5X blk contamination For negative blk results: J(+)/UJ(-) < abs. value of 5X blk contamination	7
Matrix Spike	One per matrix per batch; 5% frequency 75-125% for samples less than 4 x spike level	J(+) if %R > 125% or < 75% UJ(-) if %R = 30-74% R(+/-) results < IDL 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(-) in assoc samples if RPD > 20% or diff > CRDL	9
Laboratory Control Sample	Waters: One per matrix per batch %R (80-120%)	R(+/-) if MS/MSD & LCS %R outside limits J(+)/UJ(-) if %R = 50-79% J(+) if %R > 120% R(+/-) if %R < 50%	10
	Soils: One per matrix per batch Result within manufacturer's certified acceptance range	J(+)/UJ(-) if < LCL, J(+) if > UCL	10

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EcoChem Validation Guidelines for Conventional Chemistry Analysis
(Based on EPA Standard Methods)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Field Blanks	taken on same day as samples	Action level is 5x blk conc. U(+) sample values < AL	6
Field Duplicates	Waters RPD < 35% Soils RPD < 50% for values > 5 x CRDL Diff < CRDL for samples >CRDL and <5 x CRDL (may use Diff < 2X CRDL for solids)	J(+)/UJ(-) in parent samples only	9

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Environmental Data Quality

APPENDIX B
DATA VALIDATION REPORTS

DATA VALIDATION REPORT
Portland Harbor RI/FS
Surface Water – R3 High Flow Sampling 2007
Semivolatile Organic Compounds by EPA Method 8270C
Columbia Analytical Services - Kelso

This report documents the review of analytical data from the analyses of surface water samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Columbia Analytical Services, Inc., Kelso, Washington.

SDG	No. Samples	Validation Level
K0700112	2 System Blank	Screening
K0700440	4 Surface Water	Full
K0701497	4 Surface Water	Summary
K0701518	8 Surface Water	Summary
K0701548	6 Surface Water	Summary
K0701588	4 Surface Water	Summary
K0701669	6 Surface Water 1 Field Blank	Summary
K0701738	5 Surface Water	Summary
K0701780	2 Surface Water	Summary
K0702017	3 Surface Water 1 System Blank	Full

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

A complete (100%) verification of the electronic data deliverable (EDD) results was performed by comparison to the hardcopy laboratory data package. Laboratory QC results were also verified (10%). No errors were found.

SDG K0701738: Sample LW3-W4023-M was originally collected on 1/15, and recollected on 3/02. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4023-M2 to differentiate from the original.

SDG K0702017: Sample LW3-W4025-M was originally collected on 1/15, and recollected on 3/10. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4025-M2 to differentiate from the original.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

1	Holding Times & Sample Receipt	2	Laboratory Control Samples (LCS)
	GC/MS Instrument Performance Check	1	Field Duplicates
	Initial Calibration (ICAL)		Internal Standards
1	Continuing Calibration (CCAL)	1	Target Analyte List
2	Laboratory Blanks	1	Reporting Limits (MDL and MRL)
1	Field Blanks		Compound Identification (Full validation only)
2	Surrogate Compounds	1	Calculation Verification (Full validation only)
2	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

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of 2° to 6°C. Several sample coolers were received outside the control limits of $4^{o}C \pm 2^{o}$. These temperature outliers did not impact data quality and no qualifiers were required.

Continuing Calibration

All relative response factor (RRF) values were greater than the 0.05 minimum control limit. All percent difference (%D) values were within the ±25% control limit for all continuing calibrations (CCAL), with the exceptions noted below. When the %D outlier indicates a potential high bias, only positive results in the associated samples were qualified as estimated (J-5B). When the %D outlier indicates a potential low bias, both positive results and reporting limits were estimated (J/UJ-5B).

SDG K0701497: CCAL 3/27/07 on instrument MS06: N-nitrosodimethylamine (high bias)

SDG K0701588: CCAL 3/27/07 on instrument MS06: N-nitrosodimethylamine (high bias)

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 reported in the blank. 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-7). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

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

SDG K0700440: Phenol (3 results)

SDG K0701497: Carbazole (1 result)

SDG K0701669: Phenol (3 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). The system blank results were not used to qualify field sample results.

SDG K0700112: Two system blanks (LW3-W4-Decon Blank and LW3-W4-Lab Blank) were reported. There were no positive results detected in either blank.

SDG K0701669: One field blank was reported. A positive result for 2-methylphenol remained in Sample LW3-W4902 after qualifiers based on the laboratory blanks were assigned. No positive results for this compound were detected in the associated samples.

SDG K0702017: One system blank was reported. A positive result for phenol was detected in Sample LW3-W4-FLAB-RB.

Surrogate Compounds

SDG K0701669: The percent recovery (%R) value for 2-fluorobiphenyl was less than the lower control limit in Sample LW3-W4028-NB. One surrogate outlier per fraction is allowed and no qualifiers were required.

SDG K0701738: The %R values for 2-fluorophenol, phenol-d6, nitrobenzene-d5, and 2-fluorobiphenyl were less than the lower control limits in Sample LW3-W4023-W. No positive results were detected in this sample and all reporting limits were estimated (UJ-13).

SDG K0702017: The %R value for terphenyl-d14 was greater than the upper control limit in Sample LW3-W4025-E and the laboratory control sample duplicate (LCSD). One surrogate outlier per fraction is allowed and no qualifiers were required.

Matrix Spike/Matrix Spike Duplicates

SDG K0700112, K0701588, K0701669, K0701738, K0701780, & K0702017: Matrix spike/matrix spike duplicate (MS/MSD) analyses were not performed. Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses were used to assess accuracy and precision.

SDG K0700440: The %R values were below the lower control limits for aniline and 3,3'-dichlorobenzidine in the MS/MSD set performed using Sample LW3-W4024-NS. Both reporting limits were qualified as estimated (UJ-8) in the parent sample. Also, the relative percent

difference (RPD) values exceeded control limits for hexachlorobutadiene and 3,3'-dichlorobenzidine. These analytes were not detected in the parent sample, no precision qualifiers were required.

The %R values were below the lower control limits for aniline and 3,3'-dichlorobenzidine in the MS/MSD set performed using Sample LW3-W4024-NB. These reporting limits were qualified as estimated (UJ-8) in the parent sample. Also, the RPD values exceeded control limits for benzoic acid and 3,3'-dichlorobenzidine. These analytes were not detected in the parent sample, no precision qualifiers were required.

SDG K0701497: Only a MS analysis was performed. The %R values for aniline and 3,3'-dichlorobenzidine were outside control limits in the MS performed using Sample LW3-4038-NS. The reporting limits for aniline and 3,3'-dichlorobenzidine were qualified as estimated (UJ-8) in the parent sample.

SDG K0701518, K0701548: Only a MS analysis was performed. The %R values for aniline and 3,3'-dichlorobenzidine were below the lower control limits in the MS performed using Sample LW3-W4031-NB. The reporting limits for aniline and 3,3'-dichlorobenzidine were qualified as estimated (UJ-8) in the parent sample.

Laboratory Control Sample/Laboratory Control Sample Duplicate

SDG K0700112: The %R values for 2,4-dinitrophenol were below 10% in both LCS and LCSD. Reporting limits for 2,4-dinitrophenol were rejected (R-10) in the associated samples. The %R values for isophorone were below the lower control limits in this LCS/LCSD set. Reporting limits for isophorone were qualified as estimated (UJ-10) in the associated samples. The %R values for 2,3,4,6-tetrachlorophenol, 2-methyl-4,6-dinitrophenol, n-nitrosodiphenylamine, and azobenzene were below the lower control limits in either the LCS or the LCSD. The %R values from the corresponding LCS or LCSD were in control, so no qualifiers were applied. The RPD values for hexachlorocyclopentadiene, 2,4-dinitrophenol, and 2-methyl-4,6-dinitrophenol exceeded the control limit. No positive results for these analytes were detected, so no precision qualifiers were required.

SDG K0700440: The %R values for isophorone, 2,3,4,6-tetrachlorophenol, and n-nitrosodiphenylamine were below lower control limits in the LCS. Reporting limits for these analytes were qualified as estimated (UJ-10) in the associated samples.

SDG K0701497: One %R value each for bis(2-chloroethyl)ether, isophorone, and 2,3,4,6-tetrachlorobenzene were below lower control limits from the LCSD, but were within limits from the LCS. No qualifiers were applied. The %R value for benzoic acid was below 10% from the LCSD, but was within limits from the LCS. All reporting limits for benzoic acid were qualified as estimated (UJ-10) - but not rejected - in the associated samples.

The RPD values for 2,4-dinitrophenol, 4-nitrophenol, and 2-methyl-4,6-dinitrophenol from the LCS/LCSD exceeded control limits. No positive results for these analytes were reported and %R values from the LCS were in control, so no qualifiers were applied.

SDG K0701518, K0701548: The %R value for 3,3'-dichlorobenzidine was below the lower control limit from the LCSD, but were within limits from the LCS. The %R value for this compound from

the associated MS was also below the lower control limit. All reporting limits for 3,3'-dichlorobenzidine were qualified as estimated (UJ-10). The %R value for hexachlorocyclopentadiene was below 10% from the LCSD, but was in control from the LCS. All reporting limits for hexachlorocyclopentadiene were qualified as estimated (UJ-10) - but not rejected - in the associated samples.

RPD values for hexachlorobutadiene, hexachlorocyclopentadiene and 3,3'-dichlorobenzidiene from the LCS/LCSD exceeded the control limits. No positive results for these analytes were reported, so no qualifiers were applied.

SDG K0701588: The %R value for 4-nitrophenol was greater than the upper control limit from the LCSD. No positive results for the compound were reported and %R value from the associated LCS was in control; no qualifiers were applied.

The RPD value for benzoic acid from the LCS/LCSD exceeded the control limit. No positive results for the compound were reported; no qualifiers were applied.

SDG K0701669: The RPD values for 2,4-dimethylphenol, hexachlorocyclopentadiene, and benzoic acid were greater than the control limit in the LCS/LCSD . A positive result for benzoic acid was qualified as estimated (J-9) in Sample LW3-W4028-NB. No positive results were detected for the other analytes, so no precision qualifiers were required.

SDG K0701738 & K0701780: The %R values for 2,3,4,6-tetrachlorophenol were below the lower control limit in the LCS/LCSD. No positive results were detected for this compound and reporting limits were qualified as estimated (UJ-10) in all samples. The RPD value for 2-methyl-4,6-dinitrophenol exceeded the control limit. No positive result was detected for this analyte, so no precision qualifiers were required.

SDG K0702017: The %R value for 3,3'-dichlorobenzidine was below the lower control limit in the LCSD. Reporting limits for this compound were qualified as estimated (UJ-10) in all samples. The RPD values for benzoic acid, 2,4-dinitrophenol, and 3,3'-dichlorobenzidine exceeded the control limit. No positive results were detected for these analytes, so no precision qualifiers were required.

Field Duplicates

Note that the RPD value is used to assess precision **only** if both sample results are greater than five times the reporting limit for a given analyte, otherwise the absolute difference between the two results is used to evaluate precision. The RPD control limit for water samples is 50%. The absolute difference control limit is the reporting limit of the compound.

No qualifiers were applied based on field duplicate precision outliers. However, any outliers were noted below. Data users should take field precision into account when interpreting sample data.

SDG K0701548: Two sets of field duplicates, Samples LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB, were submitted. There were no positive results in these samples, except phenol - detected at a level below the method reporting limit (MRL) in Sample LW3-W4033-NS. Field precision was acceptable.

Target Analyte List

Dibenzofuran was reported from a separate analysis with the PAH compounds (EPA 8270C-SIM). It was noted by the laboratory that 3-methylphenol could not be separated from 4-methylphenol. Also, 1,2-diphenylhydrazine was reported as azobenzene.

Reporting Limits (Method Detection Limit and Method Reporting Limit)

The Quality Assurance Project Plan (QAPP) calls for a method reporting limit (MRL) of 0.002 µg/L for n-nitrosodimethylamine. The laboratory MRL for this analyte was 2.0 µg/L.

Calculation Verification

SDG K0700440 & K0702017: Calculation verifications were performed on these 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 surrogate, LCS/LCS, and MS/MSD %R values, with the exceptions noted above. Precision was also acceptable as demonstrated by the LCS/LCSD and field duplicate RPD values with the exceptions noted above.

Data were qualified as not detected based on laboratory blank contamination. Data were qualified as estimated because of surrogate, LCS/LCSD, and MS/MSD %R outliers and an LCS/LCSD RPD outlier.

Data were also rejected because of a very low LCS/LCSD %R outlier. Rejected data is not useable for any purpose.

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

DATA VALIDATION REPORT
Portland Harbor RI/FS
Surface Water – R3 High Flow Sampling 2007
Polyaromatic Hydrocarbons (PAH) by EPA Method 8270C-SIM
Columbia Analytical Services - Kelso

This report documents the review of analytical data from the analyses of surface water samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Columbia Analytical Services, Inc., Kelso, Washington.

SDG	No. Samples	Validation Level
K0700112	2 System Blanks	Screening
K0700440	4 Surface Water	Full
K0701497	4 Surface Water	Summary
K0701518	8 Surface Water	Summary
K0701548	6 Surface Water	Summary
K0701588	4 Surface Water	Summary
K0701669	6 Surface Water 1 Field Blank	Summary
K0701738	5 Surface Water	Summary
K0701780	2 Surface Water	Summary
K0702017	3 Surface Water 1 System Blank	Full Screening

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%). No errors were found.

SDG K0701738: Sample LW3-W4023-M was originally collected on 1/15, and recollected on 3/02. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4023-M2 to differentiate from the original.

SDG K0702017: Sample LW3-W4025-M was originally collected on 1/15, and recollected on 3/10. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4025-M2 to differentiate from the original.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

1	Holding Times & Sample Receipt	2	Laboratory Control Samples (LCS/LCSD)
	GC/MS Instrument Performance Check	1	Field Duplicates
	Initial Calibration (ICAL)		Internal Standards
	Continuing Calibration (CCAL)	1	Target Analyte List
	Laboratory Blanks	1	Reporting Limits (MDL and MRL)
1	Field Blanks		Compound Identification (Full validation only)
1	Surrogate 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

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of 2° to 6°C. Several sample coolers were received outside the control limits of $4^{o}C \pm 2^{o}$. These temperature outliers did not impact data quality and no qualifiers were required.

Field Blanks

Method blanks are used to evaluate all associated samples, including field and system blanks. Any remaining positive results in the field blanks are used to evaluate **all** field 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). The system blank results were not used to qualify field sample results.

SDG K0700112: Two system blanks (LW3-W4-Decon Blank and LW3-W4-Lab Blank) were reported. There were no target analytes detected in either blank.

SDG K0701669: One field blank was reported. There were no target analytes detected in Sample LW3-W4902.

SDG K0702017: One system blank was reported. There were no target analytes detected in Sample LW3-W4-FLAB-RB.

Surrogate Compounds

SDG K0702017: The percent recovery (%R) value for terphenyl-d14 was greater than the upper control limit in Sample LW3-W4025-E and the laboratory control sample duplicate (LCSD). The %R values for both other surrogate compounds were acceptable in both samples so no qualifiers were required.

Matrix Spike/Matrix Spike Duplicates

SDG K0700112, K0701588, K0701669, K0701738, K0701780, & K0702017: Matrix spike/matrix spike duplicate (MS/MSD) analyses were not performed. Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses were used to assess accuracy and precision.

Laboratory Control Sample/Laboratory Control Sample Duplicate

SDG K0701497: RPD values for naphthalene and 2-methylnaphthalene from the LCS/LCSD exceeded the control limit. Positive results for these analytes were qualified as estimated (J-9).

Field Duplicates

Note that the RPD value is used to assess precision **only** if both sample results are greater than five times the reporting limit for a given analyte, otherwise the absolute difference between the two results is used to evaluate precision. The RPD control limit for water samples is 50%. The absolute difference control limit is the reporting limit of the compound.

No qualifiers were applied based on field duplicate precision outliers. However, outliers were noted below. Data users should take field precision into account when interpreting sample data.

SDG K0701548: Two sets of field duplicates, Samples LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB, were submitted. There were no positive results in these samples, except that acenaphthylene was detected at a level between the method detection limit (MDL) and method reporting limit (MRL) in Sample LW3-W4033-2-NS. Field precision was acceptable.

Target Analyte List

Dibenzofuran, listed in the QAPP as a semi-volatile compound, was instead reported with the PAH target analyte list.

Reporting Limits (Method Detection Limit and Method Reporting Limit)

Many samples were analyzed at dilutions (factor 2x). Reporting limits met the QAPP target MRL values.

Calculation Verification

SDG K0700440 & K0702017: Calculation verifications were performed on these 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 surrogate, LCS/LCSD and MS %R values. Precision was also acceptable as demonstrated by the LCS/LCSD and field duplicate RPD values, with the exception noted above.

Data were qualified as estimated for laboratory precision outliers.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Surface Water – R3 High Flow Sampling 2007
Phthalate Compounds by EPA Method 525.2
Columbia Analytical Services - Kelso

This report documents the review of analytical data from the analyses of surface water samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Columbia Analytical Services, Inc., Kelso, Washington.

SDG	No. Samples	Validation Level
K0700112	2 System Blanks	Screening
K0700440	4 Surface Water	Full
K0701497	4 Surface Water	Summary
K0701518	8 Surface Water	Summary
K0701548	6 Surface Water	Summary
K0701588	4 Surface Water	Summary
K0701669	6 Surface Water 1 Field Blank	Summary
K0701738	5 Surface Water	Summary
K0701780	2 Surface Water	Summary
K0702017	3 Surface Water 1 System Blank	Full

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

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

SDG K0701738: Sample LW3-W4023-M was originally collected on 1/15, and recollected on 3/02. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4023-M2 to differentiate from the original.

SDG K0702017: Sample LW3-W4025-M was originally collected on 1/15, and recollected on 3/10. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4025-M2 to differentiate from the original.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

2	Holding Times & Sample Receipt		Laboratory Control Samples (LCS)
	GC/MS Instrument Performance Check	1	Field Duplicates
	Initial Calibration (ICAL)		Internal Standards
1	Continuing Calibration (CCAL)		Target Analyte List
2	Laboratory Blanks	1	Reporting Limits (MDL and MRL)
2	Field Blanks		Compound Identification (Full validation only)
1	Surrogate Compounds	1	Calculation Verification (Full validation only)
1	Matrix Spikes (MS)		

[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

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of 2° to 6°C. Several sample coolers were received outside the control limits of $4^{o}C \pm 2^{o}$. These temperature outliers did not impact data quality and no qualifiers were required.

SDG K0700440: All samples were extracted one day after expiration of the 7 day holding time. All results and reporting limits were qualified as estimated (J/UJ-1).

Continuing Calibration (CCAL)

All relative response factor (RRF) values were greater than the 0.05 minimum control limit. All percent difference (%D) values were within the ±25% control limit for all continuing calibrations, with the exceptions noted below. When the %D outlier indicates a potential high bias, only positive results in the associated samples were qualified as estimated (J-5B). When the %D outlier indicates a potential low bias, both positive results and reporting limits were estimated (J/UJ-5B).

SDG K0701497: CCAL 3/1/07: di-n-butyl phthalate (high bias)

SDG K0701518: CCAL 3/1/07: di-n-butyl phthalate (high bias)

SDG K0701548: CCAL 3/2/07: di-n-butyl phthalate (high bias)

SDG K0702017: CCAL 3/17/07: di-ethyl phthalate and di-n-octyl phthalate (low bias)

Laboratory Blanks

To assess the impact of each blank contaminant on the reported sample results, an action level is established at the phthalate concentration reported in the blank [2x the concentration for bis(2-ethylhexyl)phthalate]. 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-7). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. If the sample result is greater than the action level but less than five times the action level [10x the level for

bis(2-ethylhexyl)phthalate], the result is qualified as estimated (J-7). No action is taken for non-detected results.

Method blanks were analyzed at the appropriate frequency. For the analytical batches tabulated below, one or more target analytes were qualified due to contamination in the method blank. A summary of contaminant levels, associated samples, and action levels is provided in the data validation worksheets.

The following results from these SDG were qualified:

SDG	Compound	Number of Results Qualified
K0700112	Diethyl phthalate Di-n-butyl phthalate Butyl benzyl phthalate	1 estimated 1 not detected, 1 estimated 2 not detected
K0700440	Dibutyl phthalate Diethyl phthalate	4 estimated 2 not detected, 2 estimated
K0701497	Di-n-butyl phthalate Butyl benzyl phthalate	4 not detected 1 not detected, 2 estimated
K0701518	Di-n-butyl phthalate Butyl benzyl phthalate	8 not detected 3 not detected, 5 estimated
K0701548	Di-n-butyl phthalate bis(2-ethylhexyl) phthalate	1 not detected 1 not detected
K0701588	Dibutyl phthalate	3 not detected, 1 Estimated
K0701669	Dibutyl phthalate	6 not detected, 1 Estimated
K0701738	Butyl benzyl phthalate	5 estimated
K0701780	Di-n-butyl phthalate Butyl benzyl phthalate	2 estimated 2 estimated
K0702017	Diethyl phthalate Di-n-butyl phthalate bis(2-ethylhexyl) phthalate	3 estimated 2 not detected, 2 estimated 3 not detected, 1 estimated

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). If the sample result is greater than the action level but less than five times the action level [10x the level for bis(2-ethylhexyl)phthalate], the result is qualified as estimated (J-6). No action is taken for non-detected results. The system blank results were not used to qualify field sample results.

SDG K0700112: Two system blanks (LW3-W4-Decon Blank and LW3-W4-Lab Blank) were reported. After qualification for method blank contamination, results for diethyl phthalate, di-n-butyl phthalate and bis(2-ethylhexyl)phthalate remained in the Decon Blank. A result for diethyl phthalate remained in the Lab Blank.

SDG K0701669: One field blank (LW3-W4902) was reported. Positive results for diethyl phthalate, di-n-butyl phthalate, butylbenzyl phthalate, and bis(2-ethylhexyl) phthalate were detected in this blank. Qualifiers are tabulated below.

SDG K0702017: One system blank (LW3-W4-FLAB-RB) was reported. Positive results for diethyl phthalate, di-n-butyl phthalate, and butylbenzyl phthalate, were detected in this blank.

For the analytical batches listed in the table below, one or more target analytes were reported in the field blank and identified as contamination in the samples. A summary of contaminant levels, associated samples, and action levels is provided in the data validation worksheets. The following results were qualified:

SDG	Compound	Number of Results Qualified (U-6, J-6)
K0700440	Bis (2-ethylhexyl)phthalate	2 not detected, 2 estimated
	Butylbenzyl phthalate	1 not detected, 3 estimated
	Dibutyl phthalate	4 not detected
	Diethyl phthalate	2 estimated
K0701497	Bis (2-ethylhexyl)phthalate	2 not detected, 2 estimated
	Diethyl phthalate	1 not detected, 3 estimated
K0701518	Bis (2-ethylhexyl)phthalate	8 not detected
	Butylbenzyl phthalate	3 not detected, 5 estimated
	Dibutyl phthalate	8 not detected
	Diethyl phthalate	1 not detected, 6 estimated
K0701548	Bis (2-ethylhexyl)phthalate	5 not detected, 1 estimated
	Butylbenzyl phthalate	6 estimated
	Dibutyl phthalate	6 not detected
	Diethyl phthalate	1 not detected, 3 estimated
K0701588	Bis (2-ethylhexyl)phthalate	3 not detected, 1 estimated
	Butylbenzyl phthalate	3 estimated
	Dibutyl phthalate	3 not detected, 1 estimated
	Diethyl phthalate	3 not detected, 1 estimated
K0701669	Bis (2-ethylhexyl)phthalate	1 not detected, 4 estimated
	Butylbenzyl phthalate	3 estimated
	Diethyl phthalate	1 not detected, 1 estimated
K0701738	Bis (2-ethylhexyl)phthalate	3 not detected, 2 estimated
	Butylbenzyl phthalate	5 estimated
	Dibutyl phthalate	5 not detected
	Diethyl phthalate	2 not detected, 3 estimated
K0701780	Bis (2-ethylhexyl)phthalate	1 not detected, 1 estimated
	Butylbenzyl phthalate	2 estimated
	Dibutyl phthalate	1 not detected, 1 estimated
	Diethyl phthalate	1 not detected, 1 estimated
K0702017	Bis (2-ethylhexyl)phthalate	1 estimated
	Butylbenzyl phthalate	1 not detected
	Dibutyl phthalate	1 not detected
	Diethyl phthalate	2 not detected

Surrogate Compounds

The percent recovery (%R) values for the surrogates were within the specified control limits with the exceptions noted below. If the outlier indicated a potential high bias, only the associated positive results were estimated (J-13). If the outlier indicated a potential low bias, positive results and reporting limits were estimated (J/UJ-13).

SDG K0700112: The %R value for perylene-d12 (68%) was less than the lower control limit from the batch matrix spike (MS) sample. No qualifiers are applied to QC samples.

SDG K0700440: The %R values for perylene-d12 (61% and 65%) were less than the lower control limits from both MS samples. No qualifiers are applied to QC samples.

Matrix Spikes

SDG K0700112, K0701497, K0701669, & K0702017: Only a MS was analyzed. There was no measure of laboratory precision.

SDG K0700440: Only MS were analyzed. There was no measure of laboratory precision. The %R of bis (2-ethylhexyl) phthalate from the MS performed using Sample LW3-W4024-NB was less than the lower control limit. The result was qualified as estimated (J-8) in the parent sample.

SDG K0701738 & K0701780: A MS was analyzed using a batch QC sample. There was no measure of laboratory precision. The %R value for dimethyl phthalate was less than the lower control limit. The parent sample was not part of this SDG, so no qualifiers were assigned.

Field Duplicates

Note that the relative percent difference (RPD) value is used to assess precision **only** if both sample results are greater than five times the reporting limit for a given analyte, otherwise the absolute difference between the two results is used to evaluate precision. The RPD control limit for water samples is 50%. The absolute difference control limit is the reporting limit of the compound.

No qualifiers were applied based on field duplicate precision outliers. However, any outliers were noted below. Data users should take field precision into account when interpreting sample data.

SDG K0701548: Two sets of field duplicates, Samples LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB, were submitted. Benzyl butyl phthalate was detected at levels between the method detection limit (MDL) and method reporting limit (MRL) in all four samples. Bis(2-ethylhexyl) phthalate was detected in Sample LW3-W4033-2-NS, but not in the duplicate sample. The RPD for bis(2-ethylhexyl)phthalate exceeded the control limit. In all other cases, field precision was acceptable.

Reporting Limits (Method Detection Limit and Method Reporting Limit)

The reported quantitation limit for di-n-octylphthalate (0.2 ug/L) exceeded the target MRL of 0.1 ug/L listed in the QAPP supplement.

SDG K0701669: Samples LW3-W4011-NB, LW3-W4011-NS, LW3-W4028-NB, and LW3-W4028-NS were analyzed at dilution (2x). Reporting limits were elevated accordingly.

Calculation Verification

SDG K0700440 & K0702017: Calculation verifications were performed on these 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 surrogate, laboratory control sample and MS %R values, with the exceptions noted above. Precision, when assessed, was acceptable as demonstrated by the field duplicate RPD values, with the exception noted above.

Data were qualified as estimated because of holding time and MS %R outliers. Data were qualified as estimated or not detected based on contamination in the associated method and field blanks. A field duplicate outlier was also noted.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Surface Water – R3 High Flow Sampling 2007
Chlorinated Herbicides by EPA Method 8151A
Columbia Analytical Services - Kelso

This report documents the review of analytical data from the analyses of surface water samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Columbia Analytical Services, Inc., Kelso, Washington.

SDG	No. Samples	Validation Level
K0700112	2 System Blanks	Screening
K0700440	4 Surface Water	Full
K0701497	4 Surface Water	Summary
K0701518	8 Surface Water	Summary
K0701548	6 Surface Water	Summary
K0701588	4 Surface Waters	Summary
K0701669	6 Surface Water 1 Equipment Blank	Summary
K0701738	5 Surface Water	Summary
K0701780	2 Surface Water	Summary
K0702017	3 Surface Water 1 System Blank	Full

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

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

SDG K0701738: Sample LW3-W4023-M was originally collected on 1/15, and recollected on 3/02. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4023-M2 to differentiate from the original.

SDG K0702017: Sample LW3-W4025-M was originally collected on 1/15, and recollected on 3/10. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4025-M2 to differentiate from the original.

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
	Initial Calibration (ICAL)	1	Laboratory Control Samples
	Continuing Calibration (CCAL)	1	Field Duplicates
	Laboratory Blanks	1	Reporting Limits (MDL and MRL)
1	Field Blanks	2	Compound Identification
	Surrogate 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

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of 2° to 6°C. The laboratory received some sample coolers with temperatures below the advisory control limits. These temperature outliers did not impact data quality and no qualifiers were required.

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). The system blank results were not used to qualify field sample results.

SDG K0700112: Two system blanks (LW3-W4-Decon Blank and LW3-W4-Lab Blank) were reported. There were no positive results detected in these blanks.

SDG K0701669: One field blank (LW3-W4902) was reported. There were no positive results detected in this field blank.

SDG K0702017: One system blank (LW3-W4-FLAB-RB) was reported. There were no positive results detected in this blank.

Matrix Spike/Matrix Spike Duplicates

SDG K0701497, K0701518, K0701548, K0701588, K0701669, K0701738, K0701780, K0702017: No matrix spike/matrix spike duplicate (MS/MSD) analyses were performed. Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses were used to assess accuracy and precision.

Laboratory Control Sample/Laboratory Control Sample Duplicate

SDG K0701738: The relative percent difference (RPD) value for 2,4-DB exceeded the upper control limit in the LCS/LCSD. There were no positive results in the associated samples, no qualifiers were required.

SDG K0701780: The RPD value for 2,4-DB exceeded the upper control limit in the LCS/LCSD. There were no positive results in the associated samples, no qualifiers were required.

Field Duplicates

Note that the RPD value is used to assess precision **only** if both sample results are greater than five times the reporting limit for a given analyte, otherwise the absolute difference between the two results is used to evaluate precision. The RPD control limit for water samples is 50%. The absolute difference control limit is the reporting limit of the compound.

No qualifiers were applied based on field duplicate precision outliers. However, any outliers were noted below. Data users should take field precision into account when interpreting sample data.

SDG K0701548: Two sets of field duplicates, Samples LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB, were submitted. There were no positive results in these samples, except MCPP was detected at a level between the method detection MDL and MRL in Sample LW3-W4033-NS. Field precision was acceptable.

Reporting Limits (Method Detection Limit and Method Reporting Limit)

SDG K0701497: All samples were analyzed at 5x dilution. Method detection limits (MDLs) and method reporting limits (MRLs) were elevated accordingly and exceeded project MRL specified in the QAPP.

All other SDG: Method detection limits were elevated for dalapon, 2,4-DB, MCPA and/or MCPP due to the presence of non-target background components. Reporting limits met project target MRL.

Compound Identification

The laboratory applies a "P" qualifier to values when the RPD value between the two analytical columns is greater than 40%. When the RPD value was greater than 40% the reported value was qualified as estimated (J-3) for poor column confirmation agreement. When the RPD value was greater than 60% the reported value was qualified as tentatively identified and estimated (NJ-3). Refer to the data validation worksheets for a detailed list of these outliers.

SDG	No. of Results Estimated (J-3)	No. of Results Tentatively Identified (NJ-3)
K0701518	1 (MCPP)	1 (2,4-D)
K0701548	1 (2,4-D)	0
K0701588	1 (Dalapon)	0

Calculation Verification

SDG K0700440 & K0702017: Calculation verifications were performed on this SDG. No calculation errors were found.

IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory performed an appropriate analytical method. Accuracy was acceptable, as demonstrated by the surrogate and LCS/LCSD percent recovery values. Precision was acceptable as demonstrated by the RPD values for the LCS/LCSD analyses.

Data were qualified as tentatively identified and estimated because the confirmation criteria were not met.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Surface Water – R3 High Flow Sampling 2007
Butyltins by Krone Method
Columbia Analytical Services - Kelso

This report documents the review of analytical data from the analyses of surface water samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Columbia Analytical Services, Inc., Kelso, Washington.

SDG	No. Samples	Validation Level
K0700112	2 System Blanks	Screening
K0700440	4 Surface Water	Full
K0701497	4 Surface Water	Summary
K0701518	8 Surface Water	Summary
K0701548	6 Surface Water	Summary
K0701588	4 Surface Water	Summary
K0701669	6 Surface Water 1 Field Blank	Summary
K0701738	5 Surface Water	Summary
K0701780	2 Surface Water	Summary
K0702017	3 Surface Water 1 System Blank	Full

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables, with the exception noted below in **Holding Times and Sample Receipt**. 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%). No errors were found.

SDG K0701738: Sample LW3-W4023-M was originally collected on 1/15, and recollected on 3/02. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4023-M2 to differentiate from the original.

SDG K0702017: Sample LW3-W4025-M was originally collected on 1/15, and recollected on 3/10. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4025-M2 to differentiate from the original.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

1	Holding Times and Sample Receipt	1	Matrix Spike/Matrix Spike Duplicates (MS/MSD)
	Initial Calibration (ICAL)	2	Laboratory Control Samples (LCS/LCSD)
	Continuing Calibration (CCAL)	1	Field Duplicates
2	Laboratory Blanks		Reporting Limits (MDL and MRL)
1	Field Blanks	1	Compound Identification
1	Surrogate Compounds		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

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of 2° to 6°C. Several sample coolers were received with temperatures below the control limits of $4^{o}C \pm 2^{o}$. These temperature outliers did not impact data quality and no qualifiers were required.

SDG K0701669: Butyltin analyses were not selected on the chain-of-custody (COC) form, although sample volumes were received. All samples were analyzed for butyltins as normal.

Laboratory Method 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.

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

SDG K0700112: di-n-butyl tin (2 results)

SDG K0700440: n-butyl tin (4 results), di-n-butyl tin (4 results)

SDG K0701497: n-butyl tin (4 results), di-n-butyl tin (4 results)

SDG K0701518: n-butyl tin (8 results), di-n-butyl tin (8 results)

SDG K0701548: n-butyl tin (6 results), di-n-butyl tin (6 results)

SDG K0701588: n-butyl tin (4 results), di-n-butyl tin (4 results)

SDG K0701669: n-butyl tin (6 results), di-n-butyl tin (7 results)

SDG K0701738: n-butyl tin (4 results), di-n-butyl tin (5 results)

SDG K0701780: n-butyl tin (2 results), di-n-butyl tin (2 results)

SDG K0702017: n-butyl tin (4 results), di-n-butyl tin (4 results)

Field Blanks

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blank 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). The system blank results were not used to qualify field sample results.

SDG K0700112: Two system blanks (LW3-W4-Decon Blank and LW3-W4-Lab Blank) were reported. After qualification for method blank contamination, positive results for n-butyltin remained in both blanks.

SDG K0701669: One field blank (LW3-W4902) was reported. After qualification for method blank contamination, no positive results remained in the field blank.

SDG K0702017: One system blank (LW3-W4-FLAB-RB) was reported. After qualification for method blank contamination, no positive results remained in this blank.

Surrogate Compounds

SDG K0701738: The percent recovery (%R) value for tri-n-propyltin was greater than the upper control limit in a batch QC sample. Qualifiers are not assigned to QC samples.

SDG K0701669: The %R value for tri-n-propyltin was greater than the upper control limit in Sample LW3-W4028-NS. There were no positive results for target analytes in this sample, so no qualifiers were required.

SDG K0701780: The %R value for tri-n-propyltin was greater than the upper control limit in a batch QC sample. Qualifiers are not assigned to QC samples.

Matrix Spike/Matrix Spike Duplicates

SDG K0700112: Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed using a batch QC sample. The %R value for n-butyltin was greater than the control limits. The parent sample concentration exceeded four times the amount spiked into the sample, no qualifiers were required.

SDG K0700440: The %R value for tri-n-butyltin was greater than upper control limit in the MSD performed using Sample LW3-W4024-NB. No qualifiers were applied as this analyte was not detected in the parent sample.

SDG K0701548: MS/MSD analyses were performed using a batch QC sample. The %R values for n-butyltin and tri-n-butyltin were greater than the control limits in the MSD. The parent sample was not included in this SDG, so no qualifiers were required.

SDG K0701588: MS/MSD analyses were performed using a batch QC sample. The %R values for n-butyltin and tri-n-butyltin were greater than the control limits. The parent sample was not included in this SDG, so no qualifiers were required.

SDG K0701669: The %R value for n-butyltin was greater than upper control limit in the MSD performed using Sample LW3-W4028-NS. No qualifiers were applied, as this analyte was not detected in the parent sample.

SDG K0701738: MS/MSD analyses were performed using a batch QC sample. The %R value for n-butyltin was greater than the control limits. The parent sample was not included in this SDG, so no qualifiers were required.

SDG K0701780: The %R value for n-butyltin was greater than the control limits in the MS analysis performed using a batch QC sample. The parent sample was not included in this SDG, so no qualifiers were required.

SDG K0702017: The %R values for n-butyltin and tri-n-butyltin were greater than upper control limit in the MS analysis performed using Sample LW3-W4025-M. No qualifiers were applied, as these analytes were not detected in the parent sample.

Laboratory Control Sample/Laboratory Control Sample Duplicate

SDG K0700440: The %R values for tri-n-butyltin were greater than upper control limits in the laboratory control sample/laboratory control sample duplicate (LCS/LCSD). This analyte was not detected in associated samples, so no precision qualifiers were applied.

SDG K0701497: The %R values for tri-n-butyltin were greater than upper control limits in the LCS/LCSD. This analyte was not detected in associated samples, so no precision qualifiers were applied.

SDG K0701518: The %R values for tri-n-butyltin were greater than upper control limits in the LCS/LCSD. Positive results for tri-n-butyltin were qualified as estimated (J-10) in Samples LW3-W4035-NB and LW3-W4035-NS.

SDG K0701548: The %R values for n-butyltin and tri-n-butyltin were greater than upper control limits in the LCS. This analyte was not detected in associated samples, so no precision qualifiers were applied.

SDG K0701588: The %R values for n-butyltin and tri-n-butyltin were greater than upper control limits in the LCS. This analyte was not detected in associated samples, so no precision qualifiers were applied.

SDG K0701669: The relative percent difference (RPD) value for tri-n-butyltin exceeded the control limit in the LCS/LCSD. This analyte was not detected in associated samples, so no precision qualifiers were applied.

SDG K0701738: The %R values for tri-n-butyltin were greater than upper control limits in the LCS/LCSD. This analyte was not detected in associated samples, so no precision qualifiers were applied.

SDG K0701780: The RPD value for tetra-n-butyltin exceeded the control limit in the LCS/LCSD. This analyte was not detected in associated samples, so no precision qualifiers were applied.

SDG K0702017: The RPD value for tri-n-butyltin exceeded the control limit in the LCS/LCSD. This analyte was not detected in associated samples, so no precision qualifiers were applied.

Field Duplicates

Note that the RPD value is used to assess precision **only** if both sample results are greater than five times the reporting limit for a given analyte, otherwise the absolute difference between the two results is used to evaluate precision. The RPD control limit for water samples is 50%. The absolute difference control limit is the reporting limit of the compound.

No qualifiers were applied based on field duplicate precision outliers. However, any outliers were noted below. Data users should take field precision into account when interpreting sample data.

SDG K0701548: Two sets of field duplicates, Samples LW3-W4033-NS & LW3-W4033-2NS and LW3-W4033-NB & LW3-W4033-2NB, were submitted. No positive results were detected after blank qualifiers were applied. Field precision was acceptable.

IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, and MS %R values, with the exception noted above. Precision was acceptable, as demonstrated by the RPD values for the LCS/LCSD and field duplicate analyses.

Data were qualified as estimated because of LCS/LCSD %R outliers. Data were also qualified as not detected based on contamination in the associated laboratory blanks.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Surface Water – R3 High Flow Sampling 2007
Metals by EPA Methods 6020, 7470A
Columbia Analytical Services - Kelso

This report documents the review of analytical data from the analyses of surface water samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Columbia Analytical Services, Inc., Kelso, Washington.

SDG	No. Samples	Validation Level
K0700112	2 System Blanks	Screening
K0700440	4 Surface Water	Full
K0701497	4 Surface Water	Summary
K0701518	8 Surface Water	Summary
K0701548	6 Surface Water	Summary
K0701588	4 Surface Water	Summary
K0701669	6 Surface Water 1 Field Blank	Summary
K0701738	5 Surface Water	Summary
K0701780	2 Surface Water	Summary
K0702017	4 Surface Water 1 System Blank	Full

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables, with the 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 QC results were also verified (10%).

SDG K0701738: Sample LW3-W4023-M was originally collected on 1/15, and recollected on 3/02. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4023-M2 to differentiate from the original.

SDG K0702017: Sample LW3-W4025-M was originally collected on 1/15, and recollected on 3/10. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4025-M2 to differentiate from the original. The total zinc result for this sample was missed from the laboratory EDD file, and was added by the validator.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

1	Holding Times and Sample Preservation		Laboratory Duplicates
	Initial Calibration		Field Duplicates
	Continuing Calibration Verification		ICP Interference Check Samples
2	CRDL Standards	2	ICP Serial Dilution
2	Laboratory Blanks		ICP-MS Internal Standards
1	Field Blanks		Reporting Limits (MDL and MRL)
	Laboratory Control Samples	1	Calculation Verification (Full validation only)
2	Matrix Spikes/Matrix Spike Duplicates		

[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 Preservation

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of 2° to 6°C. Several sample coolers were received with temperatures outside the control limits of $4^{o}C \pm 2^{o}$. These temperature outliers did not impact data quality and no qualifiers were required.

Contract Required Detection Limit Standard

Contract required detection limit (CRDL) standards were analyzed at the beginning of each analytical sequence. For recovery values greater than upper control limit of 130%, positive results less than two times (<2x) the CRDL are estimated (J) to indicate a potential high bias. For recovery values less than the lower control limit of 70%, positive results less than twice (<2x) the CRDL and non-detects are estimated (J/UJ) to indicate a potential low bias. The following outliers resulted in qualification of data.

SDG K0700112: chromium, copper, and zinc (high bias)

SDG K0700440: zinc (high bias)

SDG K0701548: silver (low bias)

SDG K0701588: chromium (high bias)

SDG K0701738: chromium (high bias)

SDG K0702017: chromium (high bias)

Laboratory Blanks

Various analytes were detected in the method and instrument blanks at levels greater than the method detection limits (MDL). To evaluate the effect on the sample data, action levels of five times (5x) the blank concentrations were established. Positive results less than the action levels in

the associated samples were qualified as not detected (U) at the reported concentration. No action was taken for non-detects.

In addition, some analytes were found at levels less than the negative MDL in some instrument blanks. For negative blanks, action levels of 5x the absolute value of the blank concentrations were established. Results below the action levels in the associated samples were qualified as estimated (J/UJ) to indicate a potential low bias.

SDG K0700112: Nickel and silver were detected in some laboratory blanks at levels greater than the MDL. These analytes were not detected in the associated samples and no action was taken.

The chromium, mercury, nickel, and selenium values for some continuing calibration blanks (CCB) were less than the negative MDL. Chromium results were positive and below the action levels; these results were estimated (J-7). Mercury, nickel, and selenium were not detected in the associated samples; reporting limits were estimated (UJ-7).

SDG K0700440: Arsenic, lead, nickel and silver were detected in some laboratory blanks at levels greater than the MDL. Some results for nickel and silver were below the action levels and were qualified as not detected (U-7).

The chromium, nickel and selenium values for some CCB were less than the negative MDL. Some chromium and nickel results were positive and below the action levels; these results were estimated (J-7). Selenium was not detected in the associated samples; reporting limits were estimated (UJ-7).

SDG K0701497: Aluminum, antimony, arsenic, copper, lead, nickel, silver and zinc were detected in various laboratory blanks at levels greater than the MDL. Some results for antimony, lead, silver and zinc were less than the action levels and were qualified as not detected (U-7).

SDG K0701518: Arsenic, chromium, silver and zinc were detected in some laboratory blanks at levels greater than the MDL. Several results for chromium, silver, and zinc were below the action levels and were qualified as not detected (U-7).

SDG K0701548: Antimony, arsenic, copper, lead, nickel, silver and zinc were detected in some laboratory blanks at levels greater than the MDL. Several results for antimony and silver were below the action levels and were qualified as not detected (U-7).

SDG K0701588: Arsenic and silver were detected in some laboratory blanks at levels greater than the MDL. Some results for these analytes were below the action levels and were qualified as not detected (U-7).

SDG K0701669: Aluminum and arsenic were detected in some laboratory blanks at levels greater than the MDL. Some results for aluminum were below the action level and were qualified as not detected (U-7).

The chromium values for some CCB were less than the negative MDL. Several chromium results were qualified as estimated (J/UJ-7).

SDG K0701738: Aluminum, arsenic, chromium and silver were detected in various laboratory blanks at levels greater than the MDL. All positive results for silver were below the action levels and were qualified as not detected (U-7).

SDG K0701780: Aluminum and arsenic were detected in some laboratory blanks at levels greater than the MDL. Results for these analytes were greater than the action levels; no qualifiers were required.

The chromium values for some continuing calibration blanks were less than the negative MDL. Both results for dissolved chromium were estimated (J-7).

SDG K0702017: Aluminum, arsenic, chromium and silver were detected in some laboratory blanks at levels greater than the MDL. Some results for aluminum and silver were below the action levels and were qualified as not detected (U-7).

The chromium values for some continuing calibration blanks were less than the negative MDL. The associated results were positive and greater than the action levels; no qualifiers were required.

Field Blanks

Method blanks are used to evaluate all associated samples, including field blanks. The remaining positive results in the field blank are used to evaluate **all** samples. Positive results in the field samples that are less than five times the field blank concentration are qualified as not detected (U-6). Field blanks and outliers are noted below. The system blank results were not used to qualify field sample results.

SDG K0700112: Two system blanks (Lab Blank and Decon Blank), were reported. After qualification for laboratory blank contamination, the following positive results remained:

Lab Blank: chromium and zinc
Decon Blank: aluminum, chromium, copper, and zinc

SDG K0701669: One field blank, LW3-W4902, was reported. After qualification for laboratory blank contamination, positive results remained for the following analytes: dissolved chromium, dissolved copper, and dissolved zinc.

SDG K0702017: One system blank, LW3-W4-FLAB-RB, was reported. After qualification for laboratory blank contamination, positive results remained for copper, lead and zinc.

Matrix Spike Samples

A matrix spike sample (MS) was analyzed at the proper frequency. The percent recovery (%R) values were within the control limits of 70%-130%, with the exception noted below. For %R values greater than 130%, the associated positive results were estimated (J-8) to indicate a possible high bias. No action was taken for non-detects. For %R values below 70%, the associated positive results and reporting limits were qualified as estimated (J/UJ-8) to indicate a possible low bias.

SDG K070154: Sample LW3-W4033-NS - arsenic (low bias)

Field Duplicates

Note that the RPD value is used to assess precision **only** if both sample results are greater than five times the reporting limit for a given analyte, otherwise the absolute difference between the two results is used to evaluate precision. The RPD control limit for water samples is 50%. The absolute difference control limit is the reporting limit of the compound.

No qualifiers were applied based on field duplicate precision outliers. However, any outliers were noted below. Data users should take field precision into account when interpreting sample data.

SDG K0701548: Two sets of field duplicates, LW3-W4033-NS & LW3-W4033-2NS and LW3-W4033-NB & LW3-W4033-2NB, were submitted. For LW3-W4033-NB & LW3-W4033-2NB, the RPD value for dissolved aluminum (173.6%) exceeded the control limit. Also, the concentration of dissolved lead was less than five times the reporting limit and the absolute difference exceeded the reporting limit.

ICP Serial Dilution

Serial dilutions were analyzed at the proper frequency. The percent difference (%D) values were less than the control limit of 10% for results greater than 50 times the MDL, with the following exceptions. For %D outliers, all associated results were qualified estimated (J/UJ-16) in the following SDG.

SDG K0701518: LW3-W4034-NB - arsenic (30%)

SDG K0701588: LW3-W4036-NS - arsenic (46%)

SDG K0701669: LW3-W4011-NB - arsenic (12%), LW3-W4028-NB – dissolved aluminum (24%)

SDG K0701780: Batch QC1 - arsenic (12%), LW3-W4028-NB – dissolved aluminum (24%)

Calculation Verification

SDG K0700440 & K0702017: Several results were verified by recalculation from the raw data. No calculation or transcription errors were noted.

IV. OVERALL ASSESSMENT

As determined by this evaluation, the laboratory followed the specified analytical methods. The laboratory and field duplicate relative percent difference values indicated acceptable precision, except as previously noted. Accuracy was also acceptable as demonstrated by MS and laboratory control sample recovery, except as noted above.

Data were qualified as not detected or estimated based on laboratory blank results. Data were also qualified as estimated based on CRDL standard and matrix spike/matrix spike duplicate %R outliers, and serial dilution %D outliers.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Surface Water – R3 High Flow Sampling 2007
General Chemistry Parameters
Columbia Analytical Services - Kelso

This report documents the review of analytical data from the analyses of surface water samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Columbia Analytical Services, Inc., Kelso, Washington.

SDG	No. Samples	Validation Level
K0700440	4 Surface Water	Full
K0701497	4 Surface Water	Summary
K0701518	8 Surface Water	Summary
K0701548	6 Surface Water	Summary
K0701588	4 Surface Water	Summary
K0701669	6 Surface Water 1 Field Blank	Full
K0701738	5 Surface Water	Summary
K0702017	3 Surface Water	Summary

The analytical tests that were performed are summarized below:

Parameter	EPA Method
Hardness as $CaCO_3$	6010B/SM 2340B
Total Dissolved Solids (TDS)	SW160.1
Total Suspended Solids (TSS & TSS 0.45um)	SW160.2
Total Organic Carbon (TOC)	SW415.1
Dissolved Organic Carbon (DOC)	SW415.1
Perchlorate	SW314.1

I. DATA PACKAGE COMPLETENESS

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

SDG K0701497: The laboratory did not submit a result for total suspended solids using a 0.45 micron filter (TSS 0.45) from Sample LW3-4038-NS. This result was taken from the raw analytical data and added to the EDD by the validator.

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%). The analytical method name for TSS analyses using a 0.45 micron filter was changed in the EDD files to "TSS 0.45" to differentiate from the default TSS analyses.

SDG K0701738: Sample LW3-W4023-M was originally collected on 1/15, and recollected on 3/02. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4023-M2 to differentiate from the original.

SDG K0701588: The total and dissolved organic carbon results for Sample LW3-W4036-NS were missing from the laboratory EDD file, and were added by the validator.

SDG K0702017: Sample LW3-W4025-M was originally collected on 1/15, and recollected on 3/10. The original sample data was validated in SDG K0700440. The recollected sample was renamed in the EDD (sys_sample_code field) as LW3-W4025-M2 to differentiate from the original.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

2	Holding Times and Sample Preservation		Matrix Spike (MS)
	Initial Calibration		Laboratory Duplicates
	Calibration Verification	1	Field Duplicates
	Laboratory Blanks		Reporting Limits (MDL and MRL)
1	Field Blanks	1	Calculation Verification (Full validation only)
1	Laboratory Control Samples (LCS)		

[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 Preservation

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of 2° to 6°C. Several sample coolers were received outside the control limits of $4^{o}C \pm 2^{o}$. These temperature outliers did not impact data quality and no qualifiers were required.

SDG K0701497: The samples collected on 2/21/07 were analyzed for total suspended solids (TSS) using a 0.45 micron filter one day beyond the QAPP specified 7 day holding time. These results were qualified as estimated (J-1).

SDG K0701669: The samples were analyzed for total dissolved solids (TDS) 6 days beyond the QAPP specified 7 day holding time. The samples were analyzed for TSS 5 days beyond the 7 day holding time. The results for these analytes were qualified as estimated (J-1).

Field Blanks

Laboratory blanks are used to evaluate all associated samples, including field blanks. The remaining positive results in the field blank are used to evaluate all samples. Positive results in the field samples that are less than five times the field blank concentration are qualified as not detected (U-6).

All samples from the sampling event are associated with the field blanks. The system blank results were not used to qualify field sample results. Field blanks and outliers are noted below:

SDG K0701669: One field blank, LW3-W4902, was reported for hardness, perchlorate, total organic carbon (TOC) and dissolved organic carbon (DOC). No target analytes were detected in this blank.

Laboratory Control Samples

Generally, duplicate laboratory control samples (LCS) were analyzed for the total suspended solids analyses. The comparison of the two results was used to determine precision.

SDG K0701548: The two LCS for TSS determined using the glass fiber filter used two different sources. Precision could not be evaluated.

SDG K0701588: For the TSS determined using the glass fiber filter, only one LCS was analyzed. Laboratory precision could not be evaluated.

Field Duplicates

Note that the relative percent difference (RPD) value is used to assess precision **only** if both sample results are greater than five times the reporting limit for a given analyte, otherwise the absolute difference between the two results is used to evaluate precision. The RPD control limit for water samples is 50%. The absolute difference control limit is the reporting limit of the compound.

No qualifiers were applied based on field duplicate precision outliers. However, any outliers were noted below. Data users should take field precision into account when interpreting sample data.

SDG K0701548: Two sets of field duplicates, Samples LW3-W4033-NS & LW3-W4033-2-NS and LW3-W4033-NB & LW3-W4033-2-NB, were submitted. Field precision was acceptable.

Calculation Verification

SDG K0700440 & K0701669: Several results were verified by recalculation from the raw data. No calculation or transcription errors were noted.

IV. OVERALL ASSESSMENT

As determined by this evaluation, the laboratory followed the specified analytical methods. The laboratory duplicate and field duplicate RPD values indicated acceptable precision. Accuracy was also acceptable, as demonstrated by the matrix spike (MS) and LCS percent recovery values.

Data were qualified based on holding time outliers.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Surface Water – R3 High Flow Sampling 2007
Hexavalent Chromium by EPA SW7196A
Columbia Analytical Services - Kelso

This report documents the review of analytical data from the analyses of sediment samples and the associated laboratory and field quality control (QC) samples. Columbia Analytical Services, Inc., Kelso, Washington, analyzed the samples.

SDG	No. Samples	Validation Level
K0701548	4 Surface Water	Summary
K0701669	2 Surface Water, 1 Field Blank	Full

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

A complete (100%) verification of the electronic data deliverable (EDD) results was performed by comparison to the hardcopy laboratory data package. Laboratory QC results were also verified (10%). No errors were found.

III. TECHNICAL DATA VALIDATION

The QC requirements for review are listed below.

2	Holding Times and Sample Preservation		Laboratory Control Samples
	Initial Calibration		Matrix Spike/Matrix Spike Duplicates (MS/MSD)
	Calibration Verification		Laboratory Duplicates
2	Laboratory Blanks	1	Reporting Limits (MDL and MRL)
1	Field Blanks	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

The laboratory received many of the sample coolers with temperatures outside the advisory control limits of 2° to 6°C. The temperature outliers ranged from 0.8º to 0.6°C. These outliers did not impact data quality and no qualifiers were required.

SDG K0701548: The hexavalent chromium analyses were performed 6 days beyond the QAPP specified 24 hour holding time. All results were qualified as estimated (UJ-1).

SDG K0701669: The hexavalent chromium analyses were performed 3 days beyond the 24 hour holding time. All results were qualified as estimated (UJ-1).

Laboratory Blanks

SDG K0701548: Hexavalent chromium was detected in the method blank at a level greater than the method detection limits (MDL). To evaluate the effect on the sample data, an action level of five times (5x) the blank concentration was established. Positive results less than the action level in the associated samples were qualified as not detected (U-7) at the reported concentration. No qualifiers were required for non-detects.

Field Blanks

Method blanks are used to evaluate all associated samples, including field blanks. The highest remaining positive results in the field blanks are used to evaluate all samples. Positive results in the field samples that are less than five times the field blank concentration are qualified as not detected (U-6). All samples from the sampling event are associated with the field blanks. The Decon Blank and Lab Blank samples were not used to qualify field sample results. Field blanks and outliers are noted below:

SDG K0701669: One field blank, LW3-W4902, was submitted. Hexavalent chromium was not detected in this blank.

Calculation Verification

SDG K0701669: Several results were verified by recalculation from the raw data. No calculation or transcription errors were noted.

IV. OVERALL ASSESSMENT

As determined by this evaluation, the laboratory followed the specified analytical methods. The laboratory duplicate relative percent difference values indicated acceptable precision. Accuracy was also acceptable, as demonstrated by the matrix spike and/or laboratory control sample percent recovery values.

Data were qualified as not detected or estimated based on laboratory blank results. Data were also qualified as estimated based on exceeded holding times.

All data, as qualified, are acceptable for use.