



EcoChem, INC.
Environmental Data Quality

DATA QUALITY EVALUATION

PORTLAND HARBOR

Round 3A Surface Water – Storm Water Sampling Event, November 2006

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
Chlorinated Pesticides – Method SW8081A
Metals - Methods SW6020 & SW7470A
General Chemistry (Conventionals) - Methods 160.1, 160.2, 415.1 & 6010B
Hexavalent Chromium – Method SW7195

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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 analysis and 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 Integral Portland Harbor project database.

SUMMARY OF DATA VALIDATION: SEMIVOLATILE ORGANIC COMPOUNDS

A total of 38 peristaltic pump samples were analyzed for semi-volatile organic compounds (SVOC) for the Portland Harbor Surface Water Storm Water sampling event. Two field duplicate samples and two rinsate blanks were collected to monitor the field collection process and two system blanks (Lab Blank and Decon Blank) were also analyzed. Columbia Analytical Services, Kelso, Washington completed the SVOC analyses.

The SVOC data for the Storm Water samples were generally acceptable. Twenty-two (22) data points (1.1% of all SVOC results) were rejected. Rejected data must not be used for any purpose.

Thirty-five (35) data points (1.7% of all 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 99% complete for the Storm Water SVOC analyses.

Holding Times and Sample Preservation

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

The sample preservation requirements (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) were not met for most samples. The majority 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. A total of 28 results (1.4% of all SVOC results) were qualified as estimated (UJ) with potential low bias 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.

No target analytes were detected in the method blanks. No data were qualified based on laboratory 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 Recoveries

Surrogate compounds were added to all field and QC samples. The laboratory reported single surrogate recovery outliers in three field samples, which exceeded the criteria for acceptable performance due to high recoveries. The validation criteria permit single outliers provided that all remaining surrogate recoveries are within control limits, so no qualifiers were required.

Matrix Spike Recoveries

MS analyses were not performed with one data set. For this set, laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses were used to assess accuracy and precision. The recovery values of 3,3'-dichlorobenzidine from two MS/MSD sets were less than 10%. Two reporting limits for this compound (0.1% of all SVOC results) were rejected for potential very low bias in the parent sample. The recovery values of aniline from two MS/MSD sets and n-nitrosodimethylamine from one MS/MSD set were less than the control limits. Reporting limits for these compounds (0.1% of all SVOC results) were qualified as estimated (UJ) with potential low bias in the parent samples.

Laboratory Control Sample Recoveries

LCS/LCSD analyses met the criteria for frequency of analysis. The recovery values of hexachlorocyclopentadiene from two LCS sets were less than 10%. Reporting limits for this compound were rejected for potential very low bias in 20 associated samples. A total of 20 results (1.0% of all Storm Water SVOC results) were rejected for LCS accuracy. The recovery values of 3,3'-dichlorobenzidine and aniline from an LCS analysis were below the control limits. Reporting limits for these compounds (0.2% of all SVOC results) in the associated samples were qualified as estimated (UJ) with potential low bias. The recovery value of benzoic acid from an LCS analysis was greater than the control limits. A positive result for this compound (0.04% of all SVOC results) in the associated samples was qualified as estimated (J) with potential high 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. As the affected compounds were not detected in associated samples, no precision qualifiers were required.

Method Detection Limits and Method Reporting Limits

The laboratory reporting limits are based on the method detection limit (MDL), adjusted for sample size and dilution. The laboratory reporting detection limits ranged from 0.0074 to 2.1 µg/L for the non-detected results. These 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

Two field blanks (LW3-W3902 and LW3-W3903) were associated with all samples. Positive results for carbazole and several phthalates were reported in these field blanks, but were not detected in the associated field samples. No data were qualified based on field blank contamination.

System Blanks

Two system blanks (Decon Blank and Lab Blank) 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-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-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 38 peristaltic pump samples were analyzed for semi-volatile polyaromatic hydrocarbon compounds (PAH) for the Portland Harbor Surface Water Storm Water sampling event. Two field duplicate samples and two rinsate blanks were collected to monitor the field collection process and two system blanks (Lab Blank and Decon Blank) were also analyzed. Columbia Analytical Services, Kelso, Washington completed the PAH analyses.

The PAH data for the Storm Water samples were acceptable. No data points were rejected or estimated.

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 extraction and analytical holding time requirements were met for all samples and associated field QC samples.

The sample preservation requirements (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) were not met for most samples. The majority 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. Various target analytes were detected in the method blanks. A total of 28 results (3.5% of all PAH results) were qualified as not detected (U) based on method blank contamination.

Accuracy

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

Surrogate Compound Recoveries

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

Matrix Spike Recoveries

MS analyses were not performed with one data set. For this set, 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 Recoveries

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. All of the relative percent difference (RPD) values were acceptable.

Method Detection Limits and Method Reporting Limits

The laboratory reporting limits are based on the method detection limit (MDL), adjusted for sample size and dilution. The laboratory reporting 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

Two field blanks (LW3-W3902 and LW3-W3903) were associated with all samples. Positive results for 2-methylnaphthalene, phenanthrene, indeno(1,2,3-cd)pyrene and benzo(g,h,i)perylene were reported in these field blanks. A total of 38 results (4.8% of all PAH results) were qualified as not detected (U) based on field blank contamination.

System Blanks

Two system blanks (Decon Blank and Lab Blank) 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-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-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 38 peristaltic pump samples were analyzed for semi-volatile phthalate ester compounds (phthalates) for the Portland Harbor Surface Water Storm Water sampling event. Two field duplicate samples and two rinsate blanks were collected to monitor the field collection process and two system blanks (Lab Blank and Decon Blank) were also analyzed. Columbia Analytical Services, Kelso, Washington completed the analyses.

The phthalate data for the Storm Water samples were generally acceptable. No data points were rejected. Two hundred and thirty (230) data points (87% 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 extraction and analytical holding time requirements were exceeded for numerous samples and associated field QC samples. A total of 228 results (86% of all phthalate results) were qualified as estimated (J/UJ) based on holding time outliers.

The sample preservation requirements (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) were not met for most samples. The majority 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. One result (0.4% of all phthalate results) was qualified as estimated (J) with potential high bias based on a continuing calibration outlier.

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 11 results (4.2% of all phthalate results) were qualified as estimated (J) and 17 results (6.4% 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 Recoveries

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

Matrix Spike Recoveries

MS outliers were noted for a batch QC sample. All other reported recoveries met the criteria for acceptable performance. No data were qualified based on MS outliers.

Laboratory Control Sample Recoveries

LCS analyses met the criteria for frequency of analysis. The recovery value of di-n-octyl phthalate from an LCS analysis was above the control limits. This analyte was not detected in the associated samples despite the potential high bias. Therefore no data were qualified based on the LCS outlier.

Precision

Duplicate analyses were not performed with some data sets. When available, LCS/LCSD and MS/MSD analyses were evaluated for laboratory precision. All reported relative percent difference (RPD) values were acceptable.

Method Detection Limits and Method Reporting Limits

The laboratory reporting limits are based on the method detection limit (MDL), adjusted for sample size and dilution. The laboratory reporting limits ranged from 0.005 to 0.016 µg/L for the non-detected results. The laboratory reporting limits met the target MRL stated in *Round 2 QAPP, Supplement 1 to Addendum 1: Surface Water*, with one exception: the reported quantitation limit of 0.2 ug/L for di-n-octylphthalate exceeded the target MRL of 0.1 ug/L.

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

Two field blanks (LW3-W3902 and LW3-W3903) were associated with all samples. Numerous target analytes were reported in these field blanks. A total of six results (2.3% of all phthalate results) were qualified as estimated (J) and 134 results (50.7% of all phthalate results) were qualified as not detected (U) based on field blank contamination.

System Blanks

Two system blanks (Decon Blank and Lab Blank) were generated and analyzed with the peristaltic pump samples. Several target analytes were reported in both 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-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-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: CHLORINATED HERBICIDES

A total of 38 peristaltic pump samples were analyzed for chlorinated herbicide compounds for the Portland Harbor Surface Water Storm Water sampling event. Two field duplicate samples and two rinsate blanks were collected to monitor the field collection process and two system blanks (Lab Blank and Decon Blank) were also analyzed. Columbia Analytical Services, Kelso, Washington performed the herbicide analyses.

The herbicide data for the Storm Water samples were generally acceptable. No data points were rejected. One data point (0.2% of all herbicide results) was 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 herbicide analyses.

Holding Times and Sample Preservation

The extraction and analytical holding time requirements were met for all samples and associated field QC samples.

The sample preservation requirements (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) were not met for most samples. The majority 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 Recoveries

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 Recoveries

MS analyses were not performed with one data set. For this set, 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 Recoveries

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. All of the relative percent difference (RPD) values were acceptable.

Method Detection Limits and Method Reporting Limits

The laboratory reporting limits are based on the method detection limit (MDL), adjusted for sample size and dilution. The laboratory reporting limits ranged from 0.034 to 9.4 µg/L for the non-detected results. The laboratory reporting limits met the target MRL stated in *Round 2 QAPP, Addendum 1: Surface Water*.

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). One result (0.2% of herbicide results) was qualified as a tentative identification (NJ) with potential high bias.

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

Two field blanks (LW3-W3902 and LW3-W3903) were associated with the samples. No target analytes were detected in any field blank.

System Blanks

Two system blanks (Decon Blank and Lab Blank) 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-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-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 38 peristaltic pump samples were analyzed for butyltin compounds for the Portland Harbor Surface Water Storm Water sampling event. Two field duplicate samples and two rinsate blanks were collected to monitor the field collection process and two system blanks (Lab Blank and Decon Blank) 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. Sixty-four (64) data points (36% 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 extraction and analytical holding time requirements were met for all samples and associated field QC samples.

The sample preservation requirements (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) were not met for most samples. The majority 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.

Tri-n-butyltin, di-n-butyl tin, and n-butyl tin were detected in the method blank. A total of 52 results (30% 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 Recoveries

A surrogate compound was added to all field and QC samples. The surrogate recovery from one sample was below the control limit. Reporting limits in the associated sample were qualified as estimated (UJ) with potential low bias. A total of four results (2.2% of butyltin results) were qualified for accuracy.

Matrix Spike Recoveries

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 recovery values met the criteria for acceptable performance.

Laboratory Control Sample Recoveries

LCS/LCSD analyses met the criteria for frequency of analysis. Several recovery values from two LCS analyses were below the control limits. Reporting limits for these compounds (34% of all butyltin 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. All of the relative percent difference (RPD) values were acceptable.

Method Detection Limits and Method Reporting Limits

The laboratory reporting limits are based on the method detection limit (MDL), adjusted for sample size and dilution. The laboratory reporting limits ranged from 0.0006 to 0.05 µg/L for the non-detected results. The laboratory reporting limits met the target MRL stated in *Round 2 QAPP, Addendum 1: Surface Water*.

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). One data point (0.6% of butyltin results) was qualified as estimated (J).

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

Two field blanks (LW3-W3902 and LW3-W3903) were associated with the samples. No target analytes were detected in any field blank.

System Blanks

Two system blanks (Decon Blank and Lab Blank) were generated and analyzed with the peristaltic pump samples. No target analytes were detected in these blanks.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-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: CHLORINATED PESTICIDES

A total of 16 peristaltic pump samples were analyzed for chlorinated pesticide compounds for the Portland Harbor Surface Water Storm Water sampling event. Two field duplicate samples were collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington performed the pesticide analyses.

The pesticide data for the Storm Water samples were generally acceptable. No data points were rejected. Fifty-one (51) data points (9.8% of all pesticide 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 dioxin analyses.

Holding Times and Sample Preservation

The extraction and analytical holding time requirements were met for all samples and associated field QC samples.

The sample preservation requirements (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) were not met for most samples. The majority 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. 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 MS analyses).

Surrogate Compound Recoveries

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

Matrix Spike Recoveries

MS/MSD analyses met the criteria for frequency of analysis. All reported recovery values met the criteria for acceptable performance.

Laboratory Control Sample Recoveries

LCS analyses met the criteria for frequency of analysis. The recovery values of hexachlorobutadiene from the LCS analyses were below the control limits. Reporting limits for this compound (3.4% of all pesticide results) in the associated samples were qualified as estimated (UJ) with potential low bias.

Precision

MS/MSD analyses were evaluated for laboratory precision. The relative percent difference (RPD) values of several analytes exceeded the control limits. One result for endosulfan sulfate (0.2% of all pesticide results) in the parent sample was qualified as estimated (J) for precision.

Method Detection Limits and Method Reporting Limits

The laboratory reporting limits are based on the method detection limit (MDL), adjusted for sample size and dilution. The laboratory reporting limits ranged from 0.057 to 25 µg/L for the non-detected results. The laboratory reporting limits met the target MRL stated in *Round 2 QAPP, Supplement 1 to Addendum 1: Surface Water*, with these exceptions: the reported quantitation limits for oxychlorane and 2,4'-DDD were exceeded the target MRL of 0.5 ng/L.

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 25% 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). Sixteen results (3.1% of pesticide results) were qualified as estimated and 17 results (3.3% of pesticide results) were qualified as tentative identifications (NJ) with potential high bias.

Field Quality Control Samples

Field QC samples collected for the pesticide analysis included field duplicate samples. The results for the field QC samples are discussed in the following sections.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-NB) were submitted. Results for one analyte exceeded absolute difference criteria from the latter pair, although no qualifiers are applied based on field duplicate precision outliers. The field duplicates are discussed in more detail in the data validation reports.

SUMMARY OF DATA VALIDATION: METALS

A total of 38 peristaltic pump samples were analyzed for total and dissolved metals for the Portland Harbor Surface Water Storm Water sampling event. Two field duplicate samples and two rinseate blanks were collected to monitor the field collection process and two system blanks (Lab Blank and Decon Blank) were also analyzed. Columbia Analytical Services, Kelso, Washington performed the metals analyses.

Parameter	Method
ICP-MS Metals	SW6020
Mercury	SW7470A

The metals data for the surface water samples were generally acceptable. No data were rejected. One hundred and twenty-seven (127) data points (10.9% 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 sample preservation requirements (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) were not met for most samples. The majority 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 summary of contaminant levels, associated samples, and action levels is provided in the data validation worksheets. A total of 275 metals results

(23.6% of all metals results) were qualified as not detected (U) based on blank contamination. The qualifiers were applied to antimony (90 results), arsenic (58 results), chromium (41 results), copper (one result), lead (28 results), selenium (25 results), and silver (32 results).

In addition, the results for some analytes were less than the negative method detection limit (MDL) in some instrument blanks. For negative blanks, action levels of five times the absolute value of the blank concentrations are established. Results less than the action levels in the associated samples are estimated (J/UJ) to indicate a potential low bias. A total of 21 results were estimated (1.8% of all metals results). The qualifiers were applied to lead (three results) and mercury (18 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 Recoveries

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

Laboratory Control Sample Recoveries

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. A total of eight results for nickel and one result for cadmium were qualified as estimated (J) for potential high bias based on CRDL standard recovery outliers (0.8% of all metals results).

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. Ninety-five (95) results and reporting limits were qualified as estimated (J/UJ) based on serial dilution outliers (8.2% of all metals results). The qualifiers were applied to aluminum (34 results), and arsenic (61 results).

Precision

Laboratory duplicate analyses were evaluated for laboratory precision. Two results for zinc were qualified as estimated (J) for precision (0.2% of all metals results).

Method Detection Limits and Method Reporting Limits

The laboratory reporting limits are based on the method detection limit (MDL), adjusted for sample size and dilution. The laboratory reporting limits ranged from 0.02 µg/L to 2 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 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

Two field blanks (LW3-W3902 and LW3-W3903) were associated with the Storm Water samples. Numerous target elements were reported in these field blanks. A total of 146 dissolved results - seven for aluminum, 54 for chromium, and 85 for zinc - were qualified as not detected (U) based on field blank contamination (12.6% of all metals results).

System Blanks

Two system blanks (Decon Blank and Lab Blank) were generated and analyzed with the peristaltic pump samples. Positive results for aluminum 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-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-NB) were submitted. All relative percent difference values met 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 38 peristaltic pump samples were analyzed for general chemistry parameters for the Portland Harbor Surface Water Storm Water sampling event. Two field duplicate samples and two rinsate blanks were collected to monitor the field collection process and two system blanks (Lab Blank and Decon Blank) were also analyzed. Columbia Analytical Services, Kelso, Washington performed the metals analyses. The following analytical methods were used:

Parameter	Method
Total Dissolved Solids (TDS)	160.1
Total Suspended Solids (TSS) Using Glass Fiber Filter	160.2
Total Suspended Solids (TSS) Using 0.45 um Filter	160.2
Perchlorate	314.0
Total Organic Carbon (TOC)	415.1
Dissolved Organic Carbon (DOC)	415.1
Hardness as CaCO ₃	6010B/SM 2340B

Data for the general chemistry parameters analysis for the surface water samples were generally acceptable. No data were rejected. Twenty-two data points (8.7% 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

The sample preservation requirements (cooler temperature of 4°C ±2°) were not met for most samples. The majority 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.

The analysis holding time requirement of seven days was not met for the total suspended solids (TSS) analysis of one sample. One result for TSS was qualified as estimated (UJ) due to the holding time outlier.

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 Recoveries

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

Laboratory Control Sample Recoveries

LCS analyses met the frequency criteria for all analyses. The recovery values for TSS from the LCS analyses were below the control limits. Twenty-one TSS results and reporting limits in the associated samples (8.3% of all general chemistry results) were qualified as estimated (J/UJ) with potential bias.

Precision

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

Method Detection Limits and Method Reporting Limits

The laboratory reporting 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, field blank and system blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Two field blanks (LW3-W3902 and LW3-W3903) were associated with the Storm Water samples. Positive results for total dissolved solids (TDS) were reported in a field blank. A total of 43 results for TDS were qualified as not detected (U) based on field blank contamination (16.9% of all general chemistry results).

System Blanks

Two system blanks (Decon Blank and Lab Blank) were generated and analyzed for hardness and perchlorate. No target analytes were detected in any system blank.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-NB) were submitted. Results for TSS exceeded absolute difference criteria from the latter pair, although no qualifiers are applied based on field duplicate precision outliers. The field duplicates are discussed in more detail in the data validation reports.

SUMMARY OF DATA VALIDATION: HEXAVALENT CHROMIUM

A total of four peristaltic pump samples were analyzed for chlorinated pesticide compounds for the Portland Harbor Surface Water Storm Water sampling event. Two field duplicate samples and a rinsate blank were collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington performed the hexavalent chromium analysis.

The hexavalent chromium data for the surface water samples were generally acceptable. No data points were rejected. All eight data points (100% of all hexavalent chromium 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 hexavalent chromium analysis.

Holding Times and Sample Preservation

The sample preservation requirements (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) were not met for most samples. The majority 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.

All sample results were qualified as estimated (J/UJ) due to holding time outliers.

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. No target analytes were detected in the 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 Recoveries

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

Laboratory Control Sample Recoveries

LCS analyses met the frequency criteria. The recovery value from one LCS analysis was greater than the control limit. Five results for hexavalent chromium in the associated samples (62% of all results) were qualified as estimated (J) with potential high bias.

Precision

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

Method Detection Limits and Method Reporting Limits

The laboratory reporting 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 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 and field blank samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

One field blank (LW3-W3902) was associated with the samples. No target analyte was detected in the field blank.

Field Duplicate Samples

Two pairs of field duplicate samples (LW3-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-NB) were submitted. All relative percent difference values met the criteria for acceptable precision.



EcoChem, INC.
Environmental Data Quality

APPENDIX B

DATA VALIDATION REPORTS

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3A Surface Water – Storm Water Sampling Event
Semi-volatile Organic Compounds – 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
K0609247	2 System Blanks	Screening
K0609612	5 Surface Water & 1 Field Blank	Full
K0609672	11 Surface Water	Summary
K0609696	24 Surface Water & 1 Field Blank	Summary

I. DATA PACKAGE COMPLETENESS

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

SDG K0609696: The laboratory data package did not include a record of cooler temperatures at time of sample receipt.

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 that were reviewed are listed below.

- | | |
|---|--|
| <ul style="list-style-type: none"> 1 Holding Times & Sample Receipt GC/MS Instrument Performance Check Initial Calibration (ICAL) 2 Continuing Calibration (CCAL) Laboratory Blanks 1 Field Blanks 1 Surrogate Compounds 2 Matrix Spikes/Matrix Spike Duplicates (MS/MSD) | <ul style="list-style-type: none"> 2 Laboratory Control Samples (LCS) 1 Field Duplicates Internal Standards 1 Target Analyte List 1 Reporting Limits (MDL and MRL) Compound Identification (Full validation only) 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

Some coolers were received at temperatures less than the recommended range of $4^{\circ}\text{C} \pm 2^{\circ}$. It was determined that these temperature outliers did not impact data quality and no qualifiers were required.

SDG K0609696: The laboratory data package did not include a record of cooler temperatures at time of sample receipt.

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 $\pm 25\%$ 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 K0609612:

CCAL 11/17/06: benzoic acid (high bias)

CCAL 11/28/06: 2,4-dinitrophenol (low bias)

SDG K0609672:

CCAL 11/17/06 on Instrument MS06: benzoic acid (high bias)

CCAL 11/17/06 on Instrument MS10: benzoic acid, 2,4-dinitrophenol (low bias)

CCAL 11/28/06 on Instrument MS06: 2,4-dinitrophenol (low bias)

SDG K0609696:

CCAL 11/17/06 on Instrument MS06: benzoic acid (high bias)

CCAL 11/26/06 on Instrument MS06: benzoic acid (high bias)

CCAL 11/28/06 on Instrument MS06: 2,4-dinitrophenol (low bias)

CCAL 12/6/06 on Instrument MS06: hexachlorocyclopentadiene (high bias)

CCAL 11/26/06 on Instrument MS10: bis(2-chloroethyl) ether (low bias), 2,4,6-tribromophenol (high bias)

Field Blanks

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blanks are used to evaluate **all** samples. If a contaminant is reported in any field sample and the concentration is less than the action level, the result is qualified as not detected (U-6).

SDG K0609247: One decontamination blank (Decon Blank) and one laboratory blank (Lab Blank) were reported with this SDG. There were no positive results detected in the Decon Blank or the Lab Blank.

SDG K0609612: One field blank (LW3-W3902) was reported. A positive result for carbazole was reported. No qualifiers were applied since this analyte was not detected in any field samples.

SDG K0609696: One field blank (LW3-W3903) was reported. Positive results for diethyl phthalate, dibutyl phthalate, butylbenzyl phthalate, and bis(2-ethylhexyl) phthalate were reported. However, phthalate results were reported from a separate analysis (Method 525.2). No qualifiers were applied from this field blank.

Surrogates

The percent recovery (%R) values for the surrogates were within the specified control limits of with the exceptions noted below. Qualifiers were only assigned when more than one %R value per fraction (acid or base-neutral) is outside the control limits. If the outliers indicated a potential high bias, only the associated positive results were estimated (J-13). If the outliers indicated a potential low bias, positive results and reporting limits were estimated (J/UJ-13).

SDG K0609696: The %R values for 2,4,6-tribromophenol were greater than the upper control limits in Samples LW3-W3903, LW3-W3035-NS, LW3-W3005-NB, and the method blank from batch # KWG0619330. No qualifiers were applied as there was only one outlier per sample. Also, one or more of the %R values for 2-fluorophenol, phenol, and 2-fluorobiphenyl were greater than control limits in the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) samples from batch # KWG0619330. Qualifiers are not assigned to QC samples.

Matrix Spike/Matrix Spike Duplicates

SDG K0609247: Matrix spike/matrix spike duplicate (MS/MSD) analyses were not performed. LCS/LCSD analyses were used to assess accuracy and precision.

SDG K0609612, K0609672 & K0609696: The %R values for aniline were outside control limits in the MS/MSD set performed using Sample LW3-W3024-NS. Also, the %R values for 3,3'-dichlorobenzidine were less than 10%. The reporting limit for aniline was qualified as estimated (UJ) and the reporting limit for 3,3'-dichlorobenzidine was rejected (R) in the parent sample. The %R value for n-nitrosodimethylamine was outside control limits in the MSD only. The %R values from the associated MS and LCS were within limits. No qualifier was required.

The %R values for n-nitrosodimethylamine and aniline were outside control limits in the MS/MSD set performed using Sample LW3-W3024-NB. Also, the %R values for 3,3'-dichlorobenzidine were less than 10%. Reporting limits for n-nitrosodimethylamine and aniline were qualified as estimated (UJ) and reporting limits for 3,3'-dichlorobenzidine were rejected (R) in the parent sample. The relative percent difference (RPD) value for 4-nitroaniline exceeded the control limit in this set. There was no positive result for this compound in the parent sample. No qualifiers were required.

Laboratory Control Sample/Laboratory Control Sample Duplicate

SDG K0609247: One %R value each for 3,3'-dichlorobenzidine and aniline were less than 10% from the LCS, but were within limits from the LCSD. All reporting limits for these compounds were qualified as estimated (UJ-10), but not rejected, in the associated samples. The %R values for

benzoic acid and 4-chloroaniline from the LCS exceeded control limits and their RPD values were greater than control limits. No positive results for these analytes were reported and %R values from the LCSD were in control, so no qualifiers were necessary.

SDG K0609612 & K0609672: The %R value for benzoic acid exceeded the upper control limit. A positive result for this compound was qualified as estimated (J-10) in the associated samples. The %R value for hexachlorocyclopentadiene was less than 10%. All reporting limits for this compound were rejected (R-10) in the associated samples.

SDG K0609696: The %R value for benzoic acid exceeded the upper control limit in the LCS from batch # KWG0619112. Since there were no positive results in the associated samples, no action was taken. The %R value for hexachlorocyclopentadiene was less than 10% in this LCS. All reporting limits for this compound were rejected (R-10) in the associated samples.

The %R values for benzyl alcohol, 4-chlorophenyl phenyl ether, and azobenzene exceeded the upper control limit in one or both of the LCS/LCSD from batch # KWG0619330. There were no positive results for these analytes in the associated samples, so no qualifiers were applied.

Field Duplicates

Note that the RPD value is used to assess precision **only** if both sample results are greater than 5X 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 twice 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 K0609672: Two sets of field duplicates, Samples LW3-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-NB, were submitted with this SDG. There were no positive results in these samples. Field precision was judged to be acceptable.

Target Analyte List

Dibenzofuran was reported from a separate analysis (EPA 8270C-SIM) with the PAH compounds. 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 was 2.0 µg/L for this analyte in the water samples.

Calculation Verification

SDG K0609612: Calculation verifications were performed on this SDG. No calculation errors were found.

IV. OVERALL ASSESSMENT

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

Data were qualified as estimated because of CCAL %D, LCS and MS/MSD %R outliers.

Data were also rejected because of very low LCS and MS/MSD %R outliers. Rejected data may not be used for any purpose.

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

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3A Surface Water – Storm Water Sampling Event
Polyaromatic Hydrocarbons (PAHs) by EPA 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
K0609247	2 System Blanks	Screening
K0609612	5 Surface Water & 1 Field Blank	Full
K0609672	11 Surface Water	Summary
K0609696	24 Surface Water & 1 Field Blank	Summary

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

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

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

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

Some coolers were received at temperatures less than the recommended temperature range of 4°C ±2°. It was determined that these temperature outliers did not impact data quality and no qualifiers were required.

SDG K0609696: The laboratory data package did not include a record of cooler temperatures at time of sample receipt.

Laboratory Blanks

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5X) 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. Below is a summary of results that were qualified as not detected (U-7).

SDG K0609247: phenanthrene (1 result)

SDG K0609612: naphthalene (6 results)

SDG K0609672: naphthalene (11 results)

SDG K0609696: naphthalene (4 results) and phenanthrene (6 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 **field blank** action level, the result is qualified as not detected (U-6).

SDG K0609247: One decontamination blank (Decon Blank) and one laboratory blank (Lab Blank) were reported. There were no positive results detected in the Decon Blank or the Lab Blank.

All other SDGs: Two field blanks (LW3-W3902 and LW3-W3903) were reported. Positive results for 2-methylnaphthalene, phenanthrene, indeno(1,2,3-cd)pyrene and benzo(g,h,i)perylene were reported in these blanks, and qualified results are listed below. A summary of contaminant levels, associated samples, and action levels is provided in the data validation worksheets.

SDG K0609612: 2-methylnaphthalene (4 results) and phenanthrene (5 results)

SDG K0609672: 2-methylnaphthalene (10 results) and phenanthrene (11 results)

SDG K0609696: 2-methylnaphthalene (3 results), indeno(1,2,3-cd)pyrene (1 result), benzo(g,h,i)perylene (1 result), and phenanthrene (3 results)

Matrix Spike/Matrix Spike Duplicates

SDG K0609247: 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 K0609672: Samples LW3-W3024-NS and LW3-W3024-NB were used for the MS/MSD analyses. The relative percent difference (RPD) values for naphthalene and 2-methylnaphthalene were outside control limits in the MS/MSD performed using Sample LW3-W3024-NS. These analytes were not detected in the parent sample. No qualifiers were required.

SDG K0609696: A Batch QC sample was used (see **SDG K0609672**) for the MS/MSD analyses. There were many percent recovery (%R) and RPD values outside control limits. A complete listing of %R and RPD outlier values levels is provided in the data validation worksheets. No qualifiers were applied as the parent sample was not from this SDG.

Field Duplicates

Note that the RPD value is used to assess precision **only** if both sample results are greater than 5X 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 twice 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 K0609672: Two sets of field duplicates, Samples LW3-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-NB, were submitted with this SDG. The absolute values for all compounds were within the control limits. Field precision was judged to be 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 Limits)

SDG K0609612 & K0609672: All samples were analyzed at a 2X dilution. Reporting limits met the QAPP target MRL values.

SDG K0609696: All but three samples were analyzed at a 2X dilution. Reporting limits met the QAPP target MRL values.

Calculation Verification

SDG K0609612: Calculation verifications were performed on this SDG. No calculation errors were found.

IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD and MS/MSD %R values. Precision was also acceptable as demonstrated by the field duplicate, MS/MSD and LCS/LCSD RPD values.

Data were also qualified as not detected because of laboratory and field blank contamination.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3A Surface Water – Storm Water Sampling Event
Phthalate Compounds – 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
K0609247	2 System Blanks	Screening
K0609612	5 Surface Water & 1 Field Blank	Full
K0609672	11 Surface Water	Summary
K0609696	24 Surface Water & 1 Field Blank	Summary

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

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

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

- | | | | |
|---|------------------------------------|---|---|
| 2 | Holding Times & Sample Receipt | 1 | Laboratory Control Samples (LCS) |
| | GC/MS Instrument Performance Check | 1 | Field Duplicates |
| | Initial Calibration (ICAL) | | Internal Standards |
| 2 | 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

Some coolers were received at temperatures less than the recommended temperature range of 4°C ±2°. Results were judged to be unaffected by the temperature outliers and no action was taken.

SDG K0609672: All samples were extracted 4 days outside of the holding time of 7 days. All results and reporting limits were qualified as estimated (J/UJ).

SDG K0609696: All samples were extracted 3 to 5 days outside of the holding time of 7 days. All results and reporting limits were qualified as estimated (J/UJ). The laboratory data package did not include a record of cooler temperatures at time of sample receipt.

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 $\pm 25\%$ 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 positive results and reporting limits were estimated (J/UJ-5B).

SDG K0609612:

CCAL 11/6/06 (12:31) & 11/6/06 (23:23): di-n-octyl phthalate (high bias)

SDG K0609672 and K0609696:

CCAL 11/17/06: di-n-octyl phthalate (high 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 5x 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 noted below, one or more target analytes were reported in the method blank. A summary of contaminant levels, associated samples, and action levels is provided in the data validation worksheets.

SDG K0609247: di-n-butyl phthalate and butyl benzyl phthalate (2 results each estimated), and bis(2-ethylhexyl)phthalate (2 results not detected)

SDG K0609612: di-n-butyl phthalate (1 result not detected), butyl benzyl phthalate (3 results estimated, 1 not detected), bis(2-ethylhexyl)phthalate (1 result not detected)

SDG K0609672: diethyl phthalate (8 results estimated), di-n-butyl phthalate (7 results estimated, 4 not detected), butyl benzyl phthalate (11 results estimated), bis(2-ethylhexyl)phthalate (6 results estimated, 1 not detected)

SDG K0609696: diethyl phthalate (5 results estimated), di-n-butyl phthalate (1 result estimated, 5 not detected), and bis(2-ethylhexyl)phthalate (1 not detected)

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.

SDG K0609247: One decontamination blank (Decon Blank) and one laboratory blank (Lab Blank) were reported. After qualification for method blank contamination, estimated results for di-n-butyl phthalate and butyl benzyl phthalate remained in Lab Blank. After qualification for method blank contamination, a positive result for diethyl phthalate and estimated results for di-n-butyl phthalate and butyl benzyl phthalate remained in Decon Blank.

SDG K0609612: One field blank (LW3-W3902) was reported. Positive results for diethyl phthalate, di-n-butyl phthalate, butylbenzyl phthalate, and bis (2-ethylhexyl) phthalate were detected in this blank. This blank was also used to evaluate the presence of field contamination for **all** SDGs in this report.

SDG K0609696: One field blank (LW3-W3903) was reported. Positive results for diethyl phthalate, di-n-butyl phthalate, and butylbenzyl phthalate were detected in this blank. This blank was also used to evaluate the presence of field contamination for **all** SDGs in this report.

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 from the listed SDGs were qualified:

SDG	Compound	Number of Results Qualified
K0609612	Bis (2-ethylhexyl)phthalate	4 not detected
	Butylbenzyl phthalate	2 not detected
	Dibutyl phthalate	4 not detected
	Diethyl phthalate	1 not detected, 3 estimated
K0609672	Bis (2-ethylhexyl)phthalate	9 not detected
	Dibutyl phthalate	7 not detected
	Diethyl phthalate	4 not detected
K0609696	Bis (2-ethylhexyl)phthalate	23 not detected, 1 estimated
	Butylbenzyl phthalate	23 not detected, 1 estimated
	Dibutyl phthalate	19 not detected
	Diethyl phthalate	18 not detected, 1 estimated

Surrogates

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 K0609247: The %R value for triphenyl phosphate (135%) was greater than the upper control limit from the batch matrix spike sample. No qualifiers are applied to a QC sample.

Matrix Spike

SDGs K0609247, K0609612, and K0609672: Only a matrix spike (MS) was analyzed. There was no measure of laboratory precision for these SDGs.

SDG K0609247: Di-n-octyl phthalate, benzyl butyl phthalate, and bis(2-ethylhexyl)phthalate %R values were greater than the upper control limits. No qualifiers were applied as the parent sample was not from this SDG.

Laboratory Control Sample

SDG K0609247: The laboratory control sample (LCS) %R for di-n-octyl phthalate was greater than the upper control limit. No positive results were detected so no qualifiers were applied.

Field Duplicates

Note that the RPD value is used to assess precision **only** if both sample results are greater than 5x the reporting limit for a given analyte, otherwise the absolute difference between the two results is used to evaluate precision. The relative percent difference (RPD) control limit for water samples is 50%. The absolute difference control limit is twice 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 K0609672: Two sets of field duplicates, Samples LW3-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-NB, were submitted with this SDG. All absolute values were within the control limits. Precision was judged to be acceptable.

Reporting Limits (Method Detection Limit and Method Reporting Limit)

The reported quantitation limit of 0.2 ug/L for di-n-octylphthalate exceeded the target MRL of 0.1 ug/L.

Calculation Verification

SDG K0609612: Calculation verifications were performed on this SDG. No calculation errors were found.

IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the surrogate, LCS and MS %R values, with the exceptions noted above. Precision, when assessed, was also acceptable as demonstrated by the field duplicate RPD values.

Data were qualified as estimated because of holding time outliers. Data were also qualified as estimated or not detected based on contamination in the associated method and field blanks.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3A Surface Water – Storm Water Sampling Event
Chlorinated Herbicides – 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
K0609247	2 System Blanks	Screening
K0609612	5 Surface Water & 1 Field Blank	Full
K0609672	11 Surface Water	Summary
K0609696	24 Surface Water & 1 Field Blank	Summary

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables, with the exception noted below under **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%).

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

- | | |
|---|---|
| <ul style="list-style-type: none"> 1 Holding Times and Sample Receipt Initial Calibration (ICAL) Continuing Calibration (CCAL) Laboratory Blanks 1 Field Blanks Surrogate Compounds | <ul style="list-style-type: none"> 1 Matrix Spikes/Matrix Spike Duplicates Laboratory Control Samples 1 Field Duplicates 1 Reporting Limits (MDL and MRL) 2 Compound Identification 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

Some coolers were received at temperatures less than the recommended temperature range of $4^{\circ}\text{C} \pm 2^{\circ}$. These temperature outliers did not impact data quality and no qualifiers were required.

SDG K0609696: The laboratory data package did not include a record of cooler temperatures at time of sample receipt.

Field Blanks

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blanks are used to evaluate all samples. If a contaminant is reported in any field sample and the concentration is less than the action level, the result is qualified as not detected (U-6).

SDG K0609247: One decontamination blank (Decon Blank) and one laboratory blank (Lab Blank) were reported. There were no positive results in the Decon Blank or Lab Blank.

SDG K0609612: One field blank was reported. No positive results were reported in field blank LW3-W3902.

SDG K0609696: One field blank was reported. No positive results were reported in field blank LW3-W3903.

Matrix Spike/Matrix Spike Duplicates

SDG K0609696: 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.

Field Duplicates

Note that the relative percent difference (RPD) value is used to assess precision **only** if both sample results are greater than 5x 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 twice 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 K0609672: Two sets of field duplicates were submitted with this SDG, Sample pairs LW3-W033-NB & LW3-W033-2-NB and LW3-W033-NS & LW3-W033-2-NS. No positive results were reported in any sample. Field precision was acceptable.

Reporting Limits (Method Detection Limit and Method Reporting Limit)

SDG K0609247: Method detection limits (MDL) were elevated for dalapon and 2,4-DB due to the presence of non-target background components. Reporting limits met project method reporting limits (MRL) targets.

SDG K0609612, K0609672, K0609696: MDLs were elevated for dalapon, MCPP and 2,4-DB due to the presence of non-target background components.

The reporting limits for several samples slightly exceeded project MRL targets after adjustment for sample volumes of less than one liter.

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).

SDG K0609612: The RPD between the two columns was greater than 90% for 2,4-DB in Sample LW3-W3023-M and the result was qualified as tentatively identified and estimated (NJ-3).

Calculation Verification

SDG K0609612: 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, LCS/LCSD, and MS/MSD %R values. Precision was acceptable as demonstrated by the RPD values for the LCS/LCSD, MS/MSD, and field duplicate 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
Round 3A Surface Water – Storm Water Sampling Event
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
K0609247	2 System Blanks	Screening
K0609612	5 Surface Water & 1 Field Blank	Full
K0609672	11 Surface Water	Summary
K0609696	24 Surface Water & 1 Field Blank	Summary

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables, with the exception noted below under **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%). The following errors were found:

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 | 1 Reporting Limits (MDL and MRL) |
| 1 Field Blanks | 2 Compound Identification |
| 2 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

Some coolers were received at temperatures below the recommended range of $4^{\circ}\text{C} \pm 2^{\circ}$. These temperature outliers did not impact data quality and no qualifiers were required.

SDG K0609696: The laboratory data package did not include a record of cooler temperatures at time of sample receipt.

Laboratory 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.

SDG K0609247: Di-n-butyl tin (2 results qualified), n-butyl tin (1 result qualified)

SDG K0609612: Di-n-butyl tin (6 results qualified), n-butyl tin (1 result qualified)

SDG K0609672: Di-n-butyl tin (9 results qualified), n-butyl tin (6 results qualified)

SDG K0609696: Tri-n-butyltin (1 result qualified), Di-n-butyl tin (18 results qualified), n-butyl tin (8 results qualified)

Field Blanks

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blanks are used to evaluate all samples. If a contaminant is reported in any field sample and the concentration is less than the action level, the result is qualified as not detected (U-6).

SDG K0609247: One decontamination blank (Decon Blank) and one laboratory blank (Lab Blank) were included with this SDG. After qualification for method blank contamination, no positive results remained.

SDG K0609612: One field blank, LW3-W3902, was included with this SDG. After qualification for method blank contamination, no positive results remained.

SDG K0609696: One field blank, LW3-W3903, was included with this SDG. After qualification for method blank contamination, no positive results remained.

Surrogate Compounds

SDG K0609612 & K0609672: The percent recovery (%R) value for tri-n-propyltin was less than the lower control limit in the laboratory control sample (LCS). Qualifiers are not assigned to QC samples.

SDG K0609696: The %R value for tri-n-propyltin was less than the lower control limit in Sample LW3-W3036-NS. All reporting limits for this sample were qualified as estimated (UJ-13) for potential low bias.

Matrix Spike/Matrix Spike Duplicates

SDGs K0609247, K0609696: Matrix spike/matrix spike duplicate (MS/MSD) analyses were not performed with these SDG. Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses were used to assess accuracy and precision.

Laboratory Control Samples

SDG K0609612: The %R values for all compounds were less than the lower control limits in the LCS. Reporting limits were estimated (UJ-10) in all samples.

SDG K0609672: The %R values for all compounds were less than the lower control limits in the LCS. Results and reporting limits were estimated (J/UJ-10) in all samples except Samples LW3-W3024-NS and LW3-W3024-NB. The %R values for all compounds were acceptable in the MS/MSD performed on these samples and reporting limits were determined to be unaffected.

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 twice 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 K0609672: Two sets of field duplicates were submitted with this SDG, Sample pairs LW3-W033-NB & LW3-W033-2-NB and LW3-W033-NS & LW3-W033-2-NS. No positive results were detected after blank qualifiers were applied. Field precision was acceptable.

Reporting Limits (Method Detection Limit and Method Reporting Limit)

SDG K0609612: Method detection limits (MDLs) for n-butyltin were elevated in five samples due to the presence of non-target background compounds. Reporting limits met project method reporting limits (MRL) targets.

SDG K0609672: MDLs for n-butyltin and di-n-butyltin were elevated in Samples LW3-W3011-NB and LW3-W3033-NB due to the presence of non-target background compounds. Reporting limits met project MRL targets.

SDG K0609696: MDLs for di-n-butyltin were elevated in 13 samples due to the presence of non-target background compounds. Reporting limits met project MRL targets.

Compound Identification

The results from the two analytical columns were compared for agreement. In cases where the RPD value between the two columns was greater than 40% the reported result was “P” flagged by the laboratory. As the elevated RPD value may indicate the presence of an interferent that may result in a high bias, when the RPD value was greater than 40% but less than 60% the reported value was estimated (J-3). If the RPD value was greater than 60%, the result was qualified as a tentative identification (NJ-3).

SDG K0609672: The RPD between columns was greater than 40% for tri-n-butyltin in Sample LW3-W3011-NB and the result was qualified as estimated (J-3).

Calculation Verification

SDG K0609612: Calculation verifications were performed on this SDG. No calculation errors were found.

IV. OVERALL ASSESSMENT

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

Data were qualified as estimated because of a surrogate recovery outlier, LCS %R outliers, and because the confirmation criteria were not met. Data were 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
Round 3A Surface Water – Storm Water Sampling Event
Pesticides - EPA Method 8081A
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
K0609672	2 Surface Water	Full
K0609696	16 Surface Water	Summary

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables, with the exception noted below under **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%).

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

- | | | | |
|---|----------------------------------|---|---|
| 1 | Holding Times and Sample Receipt | 2 | Matrix Spikes/Matrix Spike Duplicates |
| | Instrument Breakdown Check | 2 | Laboratory Control Samples (LCS) |
| | Initial Calibration (ICAL) | 1 | Field Duplicates |
| | Continuing Calibration (CCAL) | 1 | Reporting Limits (MDL and MRL) |
| | Laboratory Blanks | 1 | Compound Identification |
| | Field Blanks | | Calculation Verification (full validation only) |
| | Surrogate Compounds | | |

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

Some coolers were received at temperatures below the recommended range of $4^{\circ}\text{C} \pm 2^{\circ}$. These temperature outliers did not impact data quality and no qualifiers were required.

SDG K0609696: The laboratory data package did not include a record of cooler temperatures at time of sample receipt.

Matrix Spike/Matrix Spike Duplicates

SDG K0609672 & K0609696: The matrix spike/matrix spike duplicate (MS/MSD) analyses were performed on Sample LW3-W3037-NS. The relative percent difference (RPD) values for endrin aldehyde, endosulfan sulfate, 4,4-DDT, and methoxychlor were greater than the control limit of 30%. A positive result for endosulfan sulfate was reported in the parent sample and qualified as estimated (J-9) for precision. The other compounds were not detected in the parent sample and reporting limits were judged to be unaffected. No further qualifiers were required.

Laboratory Control Samples

SDG K0609672 & K0609696: The percent recovery (%R) values for hexachlorobutadiene were less than the lower control limit in the laboratory control sample (LCS) and laboratory control sample duplicate (LCSD). No positive values were reported in the associated samples. Reporting limits for hexachlorobutadiene were qualified as estimated (UJ-10). The %R values for oxychlorane, cis-nonachlor, and trans-nonachlor exceeded the control limits in the LCS only. As the %R values for these compounds were acceptable in the LCSD, no further qualifiers were required.

Field Duplicates

Note that the RPD value is used to assess precision **only** if both sample results are greater than 5X 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 twice 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 K0609696: Two pairs of field duplicate samples were submitted. In Samples LW3-W3036-NS & LW3-3036-2-NS, the absolute difference between gamma-chlordane results exceeded the control limit.

In Samples LW3-W3036-NB & LW3-3036-2-NB, the absolute difference values between all positive results were within the control limit. Precision was judged to be acceptable.

Reporting Limits (Method Detection Limit and Method Reporting Limit)

SDGs K0609672 & K0609696: The laboratory reporting limits for oxychlordane and 2,4'-DDD were greater than the QAPP specified reporting limit of 0.5 ng/L. In addition, the reporting limits were elevated for several compounds in all samples due to the presence of non-target background components.

Compound Identification

The results from the two analytical columns were compared for agreement. In cases where the RPD value between the two columns was greater than 40% the reported result was "P" flagged by the laboratory. As the elevated RPD value may indicate the presence of an interferent that may result in a high bias, when the RPD value was greater than 25% but less than 60% the reported value was estimated (J-3). If the RPD value was greater than 60%, the result was qualified as a tentative identification (NJ). Refer to the data validation worksheets for a detailed list of these outliers.

SDG K0609672: Values for one or more compounds were qualified as estimated (J-3) or tentatively identified and estimated (NJ-3) in both samples.

SDG K0609696: Values for one or more compounds were qualified as estimated (J-3) or tentatively identified and estimated (NJ-3) in 15 of the 16 samples.

Calculation Verification

SDG K0609672: 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, LCS/LCSD, and MS/MSD %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the RPD values for the LCS/LCSD, MS/MSD, and field duplicate analyses, with the exceptions noted above.

Data were qualified as estimated based on LCS/LCSD %R and MS/MSD RPD outliers. Data were qualified as estimated or 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
Round 3A Surface Water – Storm Water Sampling Event
Metals – 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
K0609247	2 System Blanks	Screening
K0609612	5 Surface Water & 1 Field Blank	Full
K0609672	11 Surface Water	Summary
K0609696	24 Surface Water & 1 Field Blank	Summary

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.

SDG K0609247: The laboratory could not determine which samples had been filtered. The dissolved metals analyses were not performed.

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 that were reviewed are listed below.

- | | |
|---|--|
| <ul style="list-style-type: none"> 1 Holding Times and Sample Preservation Initial Calibration Continuing Calibration Verification 2 CRDL Standards 2 Laboratory Blanks 2 Field Blanks Laboratory Control Samples Matrix Spikes & Matrix Spike Duplicates | <ul style="list-style-type: none"> 2 Laboratory Duplicates 1 Field Duplicates ICP Interference Check Samples ICP Serial Dilution ICP-MS Internal Standards 1 Reporting Limits (MDL and MRL) Calculation Verification (Full validation only) |
|---|--|

¹ *Quality control results are discussed below, but no data were qualified*

² *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

Holding Times and Sample Receipt

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 -0.1° to 1.9°C. These temperature outliers did not impact data quality and no qualifiers were required.

SDG K0609696: The laboratory data package did not include a record of cooler temperatures at time of sample receipt.

CRDL 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 were noted:

SDG K0609696: Total cadmium and silver – high bias; dissolved cadmium, nickel, and silver – high bias. Qualifiers were applied to positive results for total cadmium and dissolved nickel.

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 less than the action levels in the associated samples were qualified as estimated (J/UJ) to indicate a potential low bias.

SDG K0609247: Arsenic, chromium and silver were detected in some laboratory blanks at levels greater than the MDL. All associated chromium results were qualified as not detected (U-7).

The lead values for the continuing calibration blanks were less than the negative MDL. Lead was not detected in the associated samples; the reporting limits were estimated (UJ-7).

SDG K0609612: Chromium and silver were detected in some method blanks at levels greater than the MDL. Antimony, arsenic, selenium, and silver were detected in some instrument blanks at levels greater than the MDL. Some results for antimony, arsenic, chromium, selenium, and silver were less than the action levels and were qualified as not detected (U-7).

SDG K0609672: Total chromium and silver and dissolved chromium were detected in the method blanks at levels greater than the MDL. Antimony, arsenic, selenium, and silver were detected in

some instrument blanks at levels greater than the MDL. All positive results for antimony, arsenic, chromium, and silver and one result for dissolved selenium were qualified as not detected (U-7).

SDG K0609696: Total copper and silver and dissolved arsenic, lead and silver were detected in the method blanks at levels greater than the MDL. Antimony, arsenic, selenium, and silver were detected in some instrument blanks at levels greater than the MDL. Positive results for antimony, arsenic, copper, lead, selenium, and silver were qualified as not detected (U-7).

The mercury values for the continuing calibration blanks were less than the negative MDL. Mercury results and reporting limits in the associated samples were qualified as estimated (J/UJ-7).

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 K0609247: Two system blanks, Lab Blank and Decon Water, were submitted with this SDG. After qualification for laboratory blank contamination, the following positive results remained:

Lab Blank –zinc

Decon Blank –aluminum and zinc

SDG K0609612: One field blank, LW3-W3902, was submitted. After qualification for laboratory blank contamination, results remained for dissolved zinc and total copper.

SDG K0609696: One field blank, LW3-W3903, was submitted. After qualification for laboratory blank contamination, results remained for total aluminum, chromium, lead, and zinc and dissolved aluminum, chromium, and zinc.

Laboratory Duplicates

Laboratory duplicate percent difference (RPD) values were used to evaluate precision. The RPD values were within the control limit of 20% for sample results greater than 5X the reporting limit (for results less than 5X the reporting limit, the absolute difference was less than twice the reporting limit) with the exceptions noted below. For RPD values exceeding the control limits, associated positive results and non-detects were qualified as estimated (J/UJ-9).

SDG K0609612: Four batch QC samples were analyzed in duplicate. The RPD for zinc was 44% for one of the Batch QC samples. No qualifiers were applied since there was only one outlier and the parent sample was from another SDG.

SDG K0609672: Duplicate analyses were performed using Samples LW3-W3024-NB and LW3-W3024-NS. Using LW3-W3024-NB the RPD values for silver (41%) and chromium (21%) were greater than control limits but sample and duplicate results were less than five times reporting

limits, no qualifiers were applied. The RPD for zinc (44%) was greater than control limits, the zinc result in Sample LW3-W3024-NB was estimated (J-9).

Field Duplicates

Note that the RPD value is used to assess precision **only** if both sample results are greater than 5X the reporting limit for a given analyte, otherwise the difference between the two results is used to evaluate precision. The RPD control limit for water samples is 50%. The 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 K0609672: Two sets of field duplicates, Samples LW3-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-NB, were submitted with this SDG. All field precision criteria were met.

ICP Serial Dilution

Serial dilutions were analyzed at the proper frequency of one per 20 samples or one per batch; whichever was more frequent. 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 estimated (J/UJ-16). The sample used for the serial dilution analysis and the outliers were as follows:

SDGs K0609612 and K0609672: LW3-W3024-NS - total aluminum (21%) and total arsenic (12%)

SDG K0609696: LW3-W3035-NS - total aluminum (20%)

LW3-W3038-NB – total arsenic (13%)

LW3-W3037-NS - total arsenic (11%)

LW3-W3037-NS-DISS – dissolved arsenic (11%)

LW3-W3032-NB-DISS – dissolved arsenic (13%)

Calculation Verification

SDG K0609612: 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 RPD values indicated acceptable precision, except as previously noted. Accuracy was also acceptable, as demonstrated by matrix spike and laboratory control sample recovery values.

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

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3A Surface Water – Storm Water Sampling Event
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
K0609247	2 System Blanks	Screening
K0609612	5 Surface Water & 1 Field Blank	Full
K0609672	11 Surface Water	Summary
K0609696	24 Surface Water & 1 Field Blank	Summary

The analytical tests that were performed are summarized below:

Parameter	Method
Total Dissolved Solids (TDS)	160.1
Total Suspended Solids (TSS) Using Glass Fiber Filter	160.2
Total Suspended Solids (TSS) Using 0.45 um Filter	160.2
Perchlorate	314.0
Total Organic Carbon (TOC)	415.1
Dissolved Organic Carbon (DOC)	415.1
Hardness as CaCO ₃	6010B/SM 2340B

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 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)
2	Field Blanks	1 Calculation Verification (Full validation only)
2	Laboratory Control Samples	

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 laboratory received many of the sample coolers with temperatures outside the advisory control limits of 2° to 6°C. The temperature outliers ranged from -0.1° to 1.9°C. These temperature outliers did not impact data quality and no qualifiers were required.

SDG K0609612: Sample LW3-W3903 was analyzed for TSS using 0.45 um filters after the 7 day holding time. The reporting limit was estimated (UJ-1).

SDG K0609696: The laboratory data package did not include a record of cooler temperatures at time of sample receipt.

Field Blanks

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blanks are used to evaluate all samples. If a contaminant is reported in any field sample and the concentration is less than the action level, the result is qualified as not detected (U-6).

SDG K0606983: Two system blanks, Decon Blank and Lab Blank, were submitted with this SDG. No positive results for perchlorate or hardness were reported in these samples.

SDG K0609612: One field blank (LW3-W3902) was submitted. There were no positive results for the analytes of interest.

SDG K0609696: One field blank (LW3-W3903) was submitted. A positive result for TDS was reported in this blank. In order to evaluate the effect on the field samples, an action level of 5x the blank concentration was established. Positive results less than the action level were qualified as not-detected (U-6). The field blank is associated with all samples from this sampling event.

Laboratory Control Sample

Laboratory control samples (LCS) were analyzed at the proper frequency of one per 20 samples or one per batch; whichever was more frequent. The percent recovery (%R) values were within the control limits of 85%-115%, with the exceptions noted below.

SDGs K0609612 and K0609672: For the TSS analysis using glass fiber filters, one LCS recovery was greater than the upper control limit and one LCS recovery was less than the lower control limit. Associated positive results and reporting limits were qualified as estimated (J/UJ-10) with no bias indicated.

For batch KWG0601724, one LCS %R value for the TSS analysis using 0.45 µm filters was less than the lower control limit. No action was taken as the recovery for the second LCS was acceptable.

Field Duplicates

Note that the relative percent difference (RPD) value is used to assess precision **only** if both sample results are greater than 5x the reporting limit for a given analyte; otherwise the difference between the two results is used. The RPD control limit for water samples is 50%. The 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 K0609672: Two sets of field duplicates were submitted with this SDG: LW3-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-NB. For samples LW3-W3033-NB & LW3-3033-2-NB, the difference between the two results for TSS using glass fiber filters was greater than the reporting limit.

Calculation Verification

SDG K0609612: 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 LCS %R values.

Data were qualified as not detected due to field blank contamination. Data were qualified as estimated due to exceeded holding times and LCS %R outliers.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3A Surface Water – Storm Water Sampling Event
Hexavalent Chromium - EPA Method 7195
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
K0609612	1 Field Blank	Full
K0609672	6 Surface Water	Summary

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%). No errors were found.

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

- | | |
|--|---|
| <ul style="list-style-type: none"> 2 Holding Times and Sample Preservation Initial Calibration Continuing Calibration Verification CRDL Standards Laboratory Blanks 1 Field Blanks | <ul style="list-style-type: none"> 2 Laboratory Control Samples Matrix Spikes & Matrix Spike Duplicates Laboratory Duplicates 1 Field Duplicates 1 Reporting Limits (MDL and MRL) 1 Calculation Verification (Full validation only) |
|--|---|

¹ *Quality control results are discussed below, but no data were qualified*

² *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

Holding Times and Sample Receipt

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 -0.1° to 1.9°C. These temperature outliers did not impact data quality and no qualifiers were required.

SDG K0609612: Sample LW3-W3903 was analyzed for hexavalent chromium after the 24-hour holding time. The reporting limit was qualified as estimated (UJ-1).

SDG K0609672: All samples were analyzed for hexavalent chromium after the holding time had expired. Results and reporting limits were qualified as estimated (J/UJ-1).

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. Positive results in the field samples that are less than 5x the field blank concentration are qualified as not detected (U-6).

SDG K0609612: One field blank, LW3-W3902, was submitted. Hexavalent chromium was not detected in this sample.

Laboratory Control Sample

Laboratory control samples (LCS) were analyzed at the proper frequency of one per 20 samples or one per batch; whichever was more frequent. The percent recovery (%R) values were within the control limits of 85%-115%, with the exception noted below.

SDG K0609672: The LCS %R value for hexavalent chromium was greater than the upper control limit. Associated results were qualified as estimated (J-10) to indicate a potential high bias.

Field Duplicates

Note that the relative percent difference (RPD) value is used to assess precision only if both sample results are greater than 5x the reporting limit for a given analyte, otherwise the difference between the two results is used to evaluate precision. The RPD control limit for water samples is 50%. The 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 K0609672: Two sets of field duplicates, Samples LW3-W3033-NS & LW3-3033-2-NS and LW3-W3033-NB & LW3-3033-2-NB, were submitted with this SDG. All field precision criteria were met.

Reporting Limits (Method Detection Limit and Method Reporting Limit)

SDG K0609612: Sample LW3-W3902 was analyzed by two methods - SW7196A and SW7195. The detection limit for SW7196A is greater than the target method detection limit (MDL) specified in the QAPP, therefore the result for method SW7195 was reported. The result for method SW7196A was qualified as do-not-report (DNR-11).

Calculation Verification

SDG K0609612: 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 RPD values indicated acceptable precision. Accuracy was also acceptable, as demonstrated by matrix spike and LCS recoveries, except as noted above.

One data point was qualified as do-not-report (DNR) because a more appropriate result was reported. Data were also qualified as estimated based on exceeded holding times and an LCS %R outlier.

All data, as qualified, are acceptable for use.