



PORTLAND HARBOR RI/FS

**ROUND 3B SEDIMENT
DATA REPORT**

APPENDIX E

DATA VALIDATION REPORTS

DRAFT

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This document is currently under review by US EPA and its federal, state, and tribal partners, and is subject to change in whole or in part.

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Prepared for
The Lower Willamette Group

Prepared by
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EcoChem, INC.
Environmental Data Quality

**DATA QUALITY EVALUATION
PORTLAND HARBOR
ROUND 3B SEDIMENT SAMPLING**

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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 10: Round 3B Comprehensive Sediment and Bioassay Testing* (Integral 2007); and *National Functional Guidelines for Organic and/or Inorganic Data Review* (USEPA 1994, 1999 & 2002). Additional guidance for the dioxin/furan data was from *EPA Region 10 SOP for the Validation of Polychlorinated Dibenzodioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data* (EPA 1996).

The samples for this sampling event were analyzed for the following:

Analysis	Method
Semivolatile Compounds (SVOC)	SW8270C
Polycyclic Aromatic Hydrocarbons (PAH)	SW8270-SIM
Phenols	SW8151A
Chlorinated Pesticides	SW8081A
Polychlorinated Biphenyl (PCB) Aroclors	SW8082
Polychlorinated Biphenyl (PCB) Congeners	1668A
Dioxin/Furan Compounds	1613B
Diesel and Residual Range Organics (DRO and RRO)	NWTPH-Dx & NWTPH-Dx SG
Metals	SW6010B, 6020, & 7470A/7471A
Total Organic Carbon	PSEP
Sulfides	SW9030b
Ammonia	E350.1
Specific Gravity	ASTM D-854-83
Grain Size	PSEP
Percent Solids	E160.3

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

PROCESS FOR DATA VALIDATION

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

Ninety percent (90%) of the data received a Level III validation, which included evaluation (as appropriate for each method) of:

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

All other data packages received full (Level IV) data validation, which includes evaluation of compound identification and quantitation (transcription and calculation checks).

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

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

SUMMARY OF DATA VALIDATION: SEMIVOLATILE ORGANIC COMPOUNDS

A total of 431 sediment samples were analyzed for semivolatile organic compounds (SVOC) for the Portland Harbor Round 3B Sediment Sampling event. The sediment samples included 26 field replicates and 14 field split samples. Twenty (20) rinsate blanks were collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington performed the SVOC analyses.

The SVOC data for the sediment samples were generally acceptable. A total of 333 data points (1.61% of all sediment SVOC results) were of unacceptable quality and were rejected. A total of 333 data points (1.61% of all sediment SVOC results) were 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 the analyses are discussed below.

Completeness of Data Set

Completeness is defined as the total number of usable results (results that were not rejected during data validation) divided by the total results reported by the laboratory. The results reported by the laboratory were greater than 98% complete for the sediment SVOC analyses.

The table below summarizes the sediment results that were rejected during data validation and the associated QC item. Note that several data points were rejected for both reasons, so the numbers below do not add up to 333:

Number of Rejected Results	Reason for Rejection
272	LCS % Recovery
66	MS/MSD % Recovery

Holding Times and Sample Preservation

The initial sample preservation requirement (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) was not met for all samples. The majority of the sample coolers were received at the laboratory with temperatures outside the advisory control limits of 2° to 6°C , ranging from -0.8° to 7.9°C . These temperature outliers did not impact data quality and no action was taken.

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 all acceptance criteria.

The continuing calibration percent difference (%D) values were reviewed to evaluate instrument stability. When %D outliers were present, the potential bias was determined. If the %D outlier indicated a low bias, associated positive results and detection limits were estimated (J or UJ). If the %D outlier indicated a high bias, only associated positive results were estimated (J). A total of 53 detected results and detection limits were estimated (J/UJ). Overall, 0.26% of the sediment SVOC results were estimated based on calibration outliers.

Method Blank Analyses

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank for most compounds and ten times (10x) for phthalates. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Various target analytes were detected in the method blanks. A total of 142 results (0.69% of all sediment SVOC results) were qualified as not detected (U) based on method blank contamination. The qualifiers were issued to bis(2-ethylhexyl)phthalate (11 results), di-n-butyl phthalate (16 results), diethyl phthalate (28 results), and phenol (87 results).

Accuracy

Surrogate Compound Recovery

Surrogate compounds were added to all samples. The surrogate recovery values reported by the laboratory typically met the criteria for acceptable performance; however, surrogate recovery outliers were present in several sediment samples. However, in all cases either only one surrogate recovery value was outside the control limits, or the outliers were due to a required dilution. No data were qualified based on surrogate recovery outliers.

Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed at the proper frequency. Several of the recovery values reported by the laboratory for MS/MSD analyses did not meet the criteria for acceptable performance. If the outlier indicated a potential high bias, only the associated positive results in the parent samples were estimated (J). If the outlier indicated a potential low bias, positive results and detection limits were estimated (J/UJ). Results were rejected when the %R value was less than 10%, indicating an extremely low bias. A total of 72 results (0.35% of all SVOC sediment results) were estimated (J or UJ) based on MS/MSD accuracy outliers. Sixty-six (66) results (0.32% of all sediment SVOC results) were rejected based on very low MS/MSD recovery values.

Laboratory Control Sample Recovery

Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses were performed at the proper frequency. Several of the recovery values reported by the laboratory did not meet the criteria for acceptable performance. If the outlier indicated a potential high bias, only the associated positive results in the parent samples were estimated (J). If the outlier indicated a potential low bias, positive results and detection limits were estimated (J/UJ). Results were rejected when the %R value was less than 10%, indicating an extremely low bias. A total of 147 results

(0.71% of all SVOC sediment results) were estimated (J or UJ) based on LCS/LCSD accuracy outliers. A total of 272 results (1.3% of all sediment SVOC results) were rejected based on very low LCS/LCSD recovery values.

Precision

MS/MSD and LCS/LCSD analyses were evaluated for laboratory precision. Several of the relative percent difference (RPD) values for the MS/MSD and LCS/LCSD analyses did not meet the criteria for acceptable performance. For laboratory precision outliers, qualifiers were issued only if the affected compound was detected in the parent sample or was also qualified due to recovery outliers. Ninety-one (91) results (0.44% of all sediment SVOC results) were estimated (J) during the quality assurance review because control limits for RPD values were not met.

Method Detection Limits and Method Reporting Limits

To try to meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the method detection limits (MDLs), adjusted for sample size, percent moisture, and any dilution factor. These method reporting limits (MRLs) ranged from 1.0 µg/Kg to 82,000 µg/Kg for the non-detected results, with roughly 31% of the results greater than 10 µg/Kg. The ACG were not met for several of the SVOC compounds.

Field Quality Control Samples

Field QC samples collected for the Round 3B sediments included field blank and field replicate samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Twenty (20) field (rinsate) blanks were associated with the sediment samples. Various target analytes were detected in the field blanks. The contaminants present in each field blank are documented in the data validation worksheets.

In most cases, these analytes were either not detected or were present at concentrations greater than the action levels in the associated samples. Twenty-five (25) results (13 diethyl phthalate results, 3 bis(2-ethylhexyl)phthalate results, 5 1,4-dichlorobenzene results, 2 benzoic acid, and 2 butyl benzyl phthalate results) were qualified as not detected based on rinsate blank contamination.

Field Replicate Samples

The field replicate sample included field splits (aliquots from the same sampling location) and replicates (aliquots from a nearby location). Several of the RPD and/or absolute difference values for field replicate analyses did not meet the criteria for acceptable precision. The field replicate precision outliers are discussed in greater detail in the data validation reports. No data were qualified based on field precision; however, users of the data should consider the potential impact of precision outliers on the reported results.

SUMMARY OF DATA VALIDATION: POLYCYCLIC AROMATIC HYDROCARBONS

A total of 431 sediment samples were analyzed for polycyclic aromatic hydrocarbon compounds (PAH) for the Portland Harbor Round 3B Sediment Sampling event. The sediment samples included 26 field replicates and 14 field split samples. For the sediments, 335 samples were also analyzed for alkylated PAH compounds. Twenty (20) rinsate blanks were collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington performed the PAH analyses.

The PAH data for the sediment samples were generally acceptable. No data were rejected for any reason. A total of 164 data points (1.1% of all sediment PAH results) were estimated, and 162 data points (1.1% of all sediment PAH results) were qualified based on blank contamination. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

The laboratory data were evaluated in terms of completeness, holding times, instrument performance, bias, and precision. The results of the quality control (QC) procedures used during the 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 sediment PAH analyses.

Holding Times and Sample Preservation

The initial sample preservation requirement (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) was not met for all samples. The majority of the sample coolers were received at the laboratory with temperatures outside the advisory control limits of 2° to 6°C , ranging from -0.8° to 7.9°C . These temperature outliers did not impact data quality and no action was taken.

Instrument Performance

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

The continuing calibration percent difference (%D) values were reviewed to evaluate instrument stability. Several %D outliers were noted; however, the outliers indicated a potential high bias and the compounds were not detected in the associated samples. No data were affected.

Method Blank Analyses

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified as

not detected (U). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Various target analytes were detected in the method blanks. A total of 153 results (1.0% of all sediment PAH results) were qualified as not detected (U) based on method blank contamination.

Accuracy

Surrogate Compound Recovery

Surrogate compounds were added to all samples. The surrogate recovery values reported by the laboratory typically met the criteria for acceptable performance; however, surrogate recovery outliers were present in several sediment samples. However, in all cases the outliers were due to a required dilution. No data were qualified based on surrogate recovery outliers.

Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed at the proper frequency. Several of the recovery values reported by the laboratory for MS/MSD analyses did not meet the criteria for acceptable performance. Ten (10) data points (0.07% of all sediment PAH results) were estimated (J) based on MS/MSD accuracy outliers.

Laboratory Control Sample Recovery

Laboratory control sample (LCS) analyses met the criteria for frequency of analysis. The recovery values reported by the laboratory met the criteria for acceptable performance.

Precision

MS/MSD and LCS/LCSD analyses were evaluated for laboratory precision. Several of the relative percent difference (RPD) values reported by the laboratory for the MS/MSD and LCS/LCSD analyses did not meet the criteria for acceptable performance. Thirty-eight (38) data points (0.25% of all sediment PAH results) were estimated (J) based on laboratory precision outliers.

Method Detection Limits and Method Reporting Limits

The laboratory reported non-detects at the method detection limits (MDLs), adjusted for sample size and any dilution factor. These method reporting limits (MRLs) ranged from 0.14 to 0.75 µg/Kg for the non-detected PAH results, and from 0.21 to 270 µg/Kg for the alkylated PAH non-detects.

Compound Identification

The laboratory “X” flagged the C1-chrysenes and/or C1-fluoranthene/pyrene results to indicate a potential high bias from an unidentified peak within the elution range. The C1-chrysenes and/or C1-fluoranthene/pyrene results in 54 samples were estimated (J). This represents 0.35% of all sediment PAH results, and 0.80% of all alkylated PAH results.

Field Quality Control Samples

Field QC samples collected for the Round 3B sediments included field blank and field replicate samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Twenty (20) field (rinsate) blanks were associated with the sediment samples. These were analyzed using the SVOC (SW8270c) analytical method, and are discussed in the SVOC validation reports. Various target analytes were detected in the field blanks. The contaminants present in each field blank are documented in the data validation worksheets.

In most cases, these analytes were either not detected or were present at concentrations greater than the action levels in the associated samples. Nine (9) results (7 naphthalene results and 2 dibenzofuran results) were qualified as not detected based on rinsate blank contamination.

Field Replicate Samples

The field replicate sample included field splits (aliquots from the same sampling location) and replicates (aliquots from a nearby location). Several of the RPD and/or absolute difference values for field replicate analyses did not meet the criteria for acceptable precision. The field replicate precision outliers are discussed in greater detail in the data validation reports. No data were qualified based on field precision; however, users of the data should consider the potential impact of precision outliers on the reported results.

SUMMARY OF DATA VALIDATION: CHLORINATED PHENOLS

A total of 431 sediment samples were analyzed for chlorinated phenolic compounds for the Portland Harbor Round 3B Sediment Sampling event. The sediment samples included 26 field replicates and 14 field split samples. Twenty (20) rinsate blanks were collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington performed the analyses.

The phenols data for the sediment samples were generally acceptable. No data were rejected for any reason. A total of 173 data points (8.0% of all sediment phenols results) were estimated, and 17 data points (0.79% of all sediment phenols results) were qualified based on blank contamination. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

The laboratory data were evaluated in terms of completeness, holding times, instrument performance, bias, and precision. The results of the quality control (QC) procedures used during the 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 sediment phenols analyses.

Holding Times and Sample Preservation

The initial sample preservation requirement (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) was not met for all samples. The majority of the sample coolers were received at the laboratory with temperatures outside the advisory control limits of 2° to 6°C , ranging from -0.8° to 7.9°C . These temperature outliers did not impact data quality and no action was taken.

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 acceptance criteria.

Method Blank Analyses

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

Various target analytes were detected in the method blanks. A total of 17 results (0.79% of all sediment phenols results) were qualified as not detected (U) based on method blank contamination. The qualifiers were issued to 2,4,6-trichlorophenol (8 results) and pentachlorophenol (9 results).

Accuracy

Surrogate Compound Recovery

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

Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed at the appropriate frequency. Several of the MS/MSD recovery values were outside the accuracy control limits. In all cases, only one outlier (in the MS or MSD) existed, and the associated data were not significantly affected. No data were qualified based on MS/MSD accuracy outliers.

Laboratory Control Sample Recovery

Laboratory control sample (LCS) analyses met the criteria for frequency of analysis. Several of the recovery values were outside the accuracy control limits. Twenty (20) results (0.93% of all sediment phenols results) were estimated based on LCS/LCSD accuracy outliers.

Precision

MS/MSD and LCS/LCSD analyses were evaluated for laboratory precision. One of the MS/MSD relative percent difference (RPD) values was outside the acceptance limit; however, the compound was not detected in the associated parent sample and no action was taken. No data were qualified based on laboratory precision outliers.

Method Detection Limits and Method Reporting Limits

The laboratory reported non-detects at the method detection limits (MDL), adjusted for sample size and any dilution factor. These method reporting limits (MRL) ranged from 0.12 to 31 µg/Kg for the non-detected results, with the majority (99%) less than 10 µg/Kg. These generally met the MRL values from the QAPP.

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 was between 40% and 60% the results were estimated (J). Where the RPD value was greater than 60% the results were tentatively identified (NJ). Fifty-six (56) data points (2.6% of all sediment phenols results) were estimated (J) and 97 results (4.5%) were tentatively identified.

It was noted by the laboratory that 2,3,5,6-tetrachlorophenol could not be separated from 2,3,4,6-tetrachlorophenol. However, these compounds were reported separately for the equipment blanks. The QAPP target analyte list does not include 2,3,4,6-tetrachlorophenol.

Field Quality Control Samples

Field QC samples collected for the Round 3B sediments included field blank and field replicate samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Twenty (20) field (rinsate) blanks were associated with the sediment samples. These were analyzed using the SVOC (SW8270c) analytical method, and are discussed in the SVOC validation reports. No chlorinated phenols were detected in any field blank. No action was necessary.

Field Replicate Samples

The field replicate sample included field splits (aliquots from the same sampling location) and replicates (aliquots from a nearby location). One of the RPD and/or absolute difference values for field replicate analyses did not meet the criteria for acceptable precision. The field replicate precision outliers are discussed in greater detail in the data validation reports. No data were qualified based on field precision; however, users of the data should consider the potential impact of precision outliers on the reported results.

SUMMARY OF DATA VALIDATION: PESTICIDE COMPOUNDS

A total of 431 sediment samples were analyzed for pesticide compounds for the Portland Harbor Round 3B Sediment Sampling event. The sediment samples included 26 field replicates and 14 field split samples. Twenty (20) rinsate blanks were collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington performed the analyses.

The phenols data for the sediment samples were generally acceptable. One data point was rejected. A total of 1,699 data points (12.7% of all sediment phenols results) were estimated, and 38 data points (0.28% of all sediment phenols results) were qualified based on blank contamination. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

The laboratory data were evaluated in terms of completeness, holding times, instrument performance, bias, and precision. The results of the quality control (QC) procedures used during the 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. One pesticide reporting limit was rejected. The results reported by the laboratory were greater than 99% complete for the sediment pesticide analyses.

Holding Times and Sample Preservation

The initial sample preservation requirement (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) was not met for all samples. The majority of the sample coolers were received at the laboratory with temperatures outside the advisory control limits of 2° to 6°C , ranging from -0.8° to 7.9°C . These temperature outliers did not impact data quality and no action was taken.

Instrument Performance

Calibrations

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

The continuing calibration (CCAL) percent difference (%D) values were used to evaluate instrument stability. The %D values for one or more compounds were outside the control limits for the secondary (confirmation) column in several CCAL. As the %D values were acceptable on the primary (quantitation) column, no action was necessary.

Endrin/DDT Breakdown

Performance evaluation mixtures (PEM) were analyzed at the proper frequency to measure percent breakdown of 4,4'-DDT and endrin. All breakdown values were acceptable.

Method Blank Analyses

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank for most compounds and ten times (10x) for phthalates. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Various target analytes were detected in the method blanks. A total of 38 results (0.28% of all sediment pesticides results) were qualified as not detected (U) based on method blank contamination. The qualifiers are documented in the validation reports and the worksheets.

Accuracy

Surrogate Compound Recovery

Surrogate compounds were added to all samples. The surrogate recovery values reported by the laboratory typically met the criteria for acceptable performance; however, surrogate recovery outliers were present in several sediment samples. However, in all cases either only one surrogate recovery value was outside the control limits, or the outliers were due to a required dilution. No data were qualified based on surrogate recovery outliers.

Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed at the proper frequency. Several of the recovery values reported by the laboratory for MS/MSD analyses did not meet the criteria for acceptable performance. Four (4) sediment pesticides results (0.03% overall) were estimated (J or UJ) because the control limits for MS/MSD recovery were not met.

Laboratory Control Sample Recovery

Laboratory control sample (LCS) analyses met the criteria for frequency of analysis. Several of the recovery values reported by the laboratory for LCS analyses did not meet the criteria for acceptable performance. Since the majority of the LCS outliers indicated a potential low bias, associated positive results and reporting limits were affected. A total of 69 results were estimated (J or UJ) based on LCS recovery outliers. This represents 0.52% of all sediment pesticide results.

Precision

MS/MSD and LCS/LCSD analyses were evaluated for laboratory precision. Several relative percent difference (RPD) values were outside the acceptance limits in the MS/MSD analyses. The affected compounds were estimated (J or UJ) in the parent samples. The 11 qualifiers represent 0.08% of the pesticide results.

Method Detection Limits and Method Reporting Limits

To try to meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the method detection limits (MDLs), adjusted for sample size and any dilution factor. The

reporting limits for non-detected results ranged from 0.027 µg/Kg to 700 µg/Kg (with toxaphene non-detects extending up to 6,500 µg/Kg) for the non-detected results. The ACG were not met for several of the pesticides. No action was taken.

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, the associated results were estimated (J). If the RPD value was greater than 60%, the result was qualified as a tentative identification (NJ). A total of 977 data points (7.3% of all sediment pesticide data points, and 34.1% of all positive results for pesticides) were estimated (J) and 644 data points (4.8% of all pesticide results, and 22.5% of all detected pesticide results) were qualified as tentative identifications (NJ). Overall, 56.5% of the detected pesticide results are affected by interference.

Field Quality Control Samples

Field QC samples collected for the Round 3B sediments included field blank and field replicate samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Twenty (20) field (rinsate) blanks were associated with the sediment samples. Various target analytes were detected in the field blanks. The contaminants present in each field blank are documented in the data validation worksheets. In all cases, the contaminants were either not present in the associated samples, or were present at concentrations greater than the action levels, No data were qualified based on rinsate blank contamination.

Field Replicate Samples

The field replicate sample included field splits (aliquots from the same sampling location) and replicates (aliquots from a nearby location). Several of the RPD and/or absolute difference values for field replicate analyses did not meet the criteria for acceptable precision. The field replicate precision outliers are discussed in greater detail in the data validation reports. No data were qualified based on field precision; however, users of the data should consider the potential impact of precision outliers on the reported results.

SUMMARY OF DATA VALIDATION: POLYCHLORINATED BIPHENYL (PCB) - AROCLOR COMPOUNDS

A total of 431 sediment samples were analyzed for PCB Aroclors for the Portland Harbor Round 3B Sediment Sampling event. The sediment samples included 26 field replicates and 14 field split samples. Twenty (20) rinsate blanks were collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington performed the analyses.

The Aroclor data for the sediment samples were generally acceptable. No data were rejected for any reason. A total of 400 data points (10.3% of all sediment Aroclor results) were estimated, and two (2) data points (0.05% of all sediment Aroclor results) were qualified as not detected. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

The laboratory data were evaluated in terms of completeness, holding times, instrument performance, bias, and precision. The results of the quality control (QC) procedures used during the 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 sediment PCB analyses.

Holding Times and Sample Preservation

The initial sample preservation requirement (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) was not met for all samples. The majority of the sample coolers were received at the laboratory with temperatures outside the advisory control limits of 2° to 6°C , ranging from -0.8° to 7.9°C . These temperature outliers did not impact data quality and no action was taken.

Thirteen samples were analyzed 41 days after extraction, one day outside the holding time for extracts. All positive results and reporting limits for these samples were estimated (J/UJ). A total of 117 data points (3.0% of all sediment Aroclor data points) were estimated.

Instrument Performance

Calibrations

Initial and continuing calibrations were completed for all reported analytes at the proper frequency. All initial calibrations met all acceptance criteria.

The continuing calibration (CCAL) percent difference (%D) values were used to evaluate instrument stability. The %D values for two compounds were outside the control limits for one column in two CCAL. As the %D values were acceptable on the other column, no action was necessary.

Method Blank Analyses

Method blanks were analyzed at the appropriate frequency. No target analytes were detected in any method blank.

Accuracy

Surrogate Compound Recovery

Surrogate compounds were added to all samples. Several of the recovery values were outside the accuracy control limits. Eleven (11) results (0.28% of all sediment Aroclor results) were estimated (J or UJ) based on surrogate accuracy outliers.

Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed at the proper frequency. Several of the recovery values were outside the accuracy control limits. One (1) result (0.03% of all sediment Aroclor results) was estimated (J) based on MS/MSD accuracy outliers.

Laboratory Control Sample Recovery

Laboratory control sample (LCS) analyses met the criteria for frequency of analysis. Several recovery values were outside the accuracy control limits. The associated data were not affected by the outliers, so no data were qualified based on LCS/LCSD accuracy outliers.

Precision

MS/MSD and LCS/LCSD analyses were evaluated for laboratory precision. The relative percent difference (RPD) values reported by the laboratory met the criteria for acceptable performance.

Method Detection Limits and Method Reporting Limits

To meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the method detection limits (MDLs), adjusted for sample size, percent moisture, and any dilution factor. These method reporting limits (MRLs) ranged from 0.73 $\mu\text{g}/\text{Kg}$ to 160 $\mu\text{g}/\text{Kg}$ for the non-detected results. The ACG of 0.004 $\mu\text{g}/\text{Kg}$ was not met, and the QAPP MRL of 4 $\mu\text{g}/\text{Kg}$ was not met for the reported PCB non-detects.

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, the associated results were estimated (J). If the RPD value was greater than 60%, the result was qualified as a tentative identification (NJ). A total of 241 data points (6.2% of all sediment Aroclor data points, and 34.5% of all positive results for Aroclors) were estimated (J) and 48 data points (1.2% of all Aroclor results, and 6.9% of all detected Aroclors results) were qualified as tentative identifications (NJ). Overall, 41.4% of the detected Aroclor results are affected by interference.

Field Quality Control Samples

Field QC samples collected for the Round 3B sediments included field blank and field replicate samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Twenty (20) field (rinsate) blanks were associated with the sediment samples. No target analytes were detected in any field blank.

Field Replicate Samples

The field replicate sample included field splits (aliquots from the same sampling location) and replicates (aliquots from a nearby location). Several of the RPD and/or absolute difference values for field replicate analyses did not meet the criteria for acceptable precision. The field replicate precision outliers are discussed in greater detail in the data validation reports. No data were qualified based on field precision; however, users of the data should consider the potential impact of precision outliers on the reported results.

SUMMARY OF DATA VALIDATION: PCB CONGENERS

A total of 170 sediment samples were analyzed for PCB congeners for the Portland Harbor Round 3B Sediment Sampling event. The sediment samples included eight field replicates and five field split samples. Thirteen (13) rinsate blanks were collected to monitor the field collection process. PCB congener analysis was performed by Vista Analytical Laboratories, El Dorado Hills, California.

The PCB congener data for these sediment samples were generally acceptable. No data were rejected for any reason. A total of 384 data points (1.2% of all PCB congener results) were qualified as estimated because control limits were exceeded in one or more laboratory quality control (QC) samples or procedures. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

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

Completeness of Data Set

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

Holding Times and Sample Preservation

All holding time and sample preservation criteria were met.

Instrument Performance

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

All other instrument performance criteria were met by the laboratory.

Laboratory Blank Analyses

Laboratory method blanks were analyzed at the required frequency. A total of 79 results (0.24% of all PCB congener results) were qualified for laboratory blank contamination.

Accuracy

Labeled Compound Recovery

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

Matrix Spike Recovery

Matrix and duplicate matrix spike analyses were not performed. Accuracy was assessed using the labeled compound and ongoing precision and recovery (OPR) analyses.

Ongoing Precision and Recovery Sample Recovery

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

Precision

Laboratory duplicate analyses were evaluated for precision. A total of 290 results and reporting limits (0.89% of all PCB congener results) were qualified as estimated for precision outliers in the parent sample and laboratory duplicate sample.

Method Detection Limits and Method Reporting Limits

No analytical concentration goals (ACG) or method reporting limits (MRL) were specified in the QAPP. For most samples, the laboratory reported results for specific toxic PCB congener (PCB77, PCB81, PCB105, PCB106/118, PCB114, PCB123, PCB126, PCB156, PCB157, PCB167, PCB169, and PCB189) using sample-specific reporting limits determined by the sample signal-to-noise ratios. All other PCB congener results were reported to the MRL. The MRL values ranged from 0.202 pg/g to 99.2 pg/g for non-detected results.

The laboratory noted chemical interferences for one or more PCB results in most samples. A total of 94 reporting limits (0.29% of all PCB congener results) were estimated due to interferences.

Field Quality Control Samples

Field QC samples collected for PCB congener analysis included field replicate and field blank samples. The results for the field QC samples are discussed in the following section.

Field Blanks

Twenty (20) field (rinsate) blanks were associated with the sediment samples. No results were qualified based on rinsate blank contamination.

Field Replicate Samples

The field replicate sample included field splits (aliquots from the same sampling location) and replicates (aliquots from a nearby location). Several of the RPD and/or absolute difference values for field replicate analyses did not meet the criteria for acceptable precision. The field replicate precision outliers are discussed in greater detail in the data validation reports. No data were qualified based on field precision; however, users of the data should consider the potential impact of precision outliers on the reported results.

SUMMARY OF DATA VALIDATION: FUELS

A total of 431 sediment samples were analyzed for diesel range organics (DRO) and residual range organics (RRO) for the Portland Harbor Round 3B Sediment Sampling event. The extracts for each of the sediment samples were also subjected to a silica gel cleanup, and then analyzed a second time. The sediment samples included 26 field replicates and 14 field split samples. Twenty (20) rinsate blanks were collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington performed the analyses.

The DRO/RRO data for the sediment samples were generally acceptable. No data were rejected for any reason. A total of 1,000 data points (58% of all sediment DRO/RRO results) were estimated, and 88 data points (5.1% of all sediment DRO/RRO results) were qualified as not detected. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

The laboratory data were evaluated in terms of completeness, holding times, instrument performance, bias, and precision. The results of the quality control (QC) procedures used during the 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 sediment DRO/RRO analyses.

Holding Times and Sample Preservation

The initial sample preservation requirement (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) was not met for all samples. The majority of the sample coolers were received at the laboratory with temperatures outside the advisory control limits of 2° to 6°C , ranging from -0.8° to 7.9°C . These temperature outliers did not impact data quality and no action was taken.

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 acceptance criteria.

Method Blank Analyses

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U). If the result is also less than the reporting limit, then the result is elevated to the

reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Method blanks are used to evaluate all associated samples, including field blanks. Any remaining positive results in the field blanks are used to evaluate all associated samples. Fuels were reported in several of the method blanks. Eighty-eight (88) data points (5.1% of all sediment DRO/RRO results) were qualified as not detected (U) based on method blank contamination. The qualifiers were issued to 33 DRO results and 55 RRO results.

Accuracy

Surrogate Compound Recovery

Surrogate compounds were added to all samples. Several of the recovery values were outside the accuracy control limits. Four (4) results (0.23% of all sediment DRO/RRO results) were estimated (J or UJ) based on surrogate accuracy outliers.

Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) analyses are not performed with fuels analyses.

Laboratory Control Sample Recovery

Laboratory control sample (LCS) analyses met the criteria for frequency of analysis. The recovery values reported by the laboratory met the criteria for acceptable performance.

Precision

Laboratory duplicate analyses were evaluated for laboratory precision. Three of the relative percent difference (RPD) values were greater than the acceptance limit. Three (3) data points (0.17% of all sediment DRO/RRO results) were estimated (J) based on laboratory precision outliers.

Method Detection Limits and Method Reporting Limits

To meet the project method reporting limit (MRL) goals of 25 mg/Kg for DRO and 100 mg/Kg for RRO, the laboratory reported non-detects at the method detection limits (MDLs), adjusted for sample size, percent moisture, and any dilution factor. The MRLs ranged from 1.3 mg/Kg to 2.1 mg/Kg for the DRO and 3.0 mg/Kg to 4.6 mg/Kg for the RRO non-detected results.

Compound Identification

Several different flags were used by the laboratory to provide information about the reported results. These flags indicated that the pattern in the sample did not match the calibration standard. During validation, the data were estimated (J) to indicate that the reported result may not accurately reflect the concentration of fuels present in the sample. A total of 998 data points (57.9% of all sediment DRO/RRO data points) were estimated.

Field Quality Control Samples

Field QC samples collected for the Round 3B sediments included field blank and field replicate samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Twenty (20) field (rinsate) blanks were associated with the sediment samples. Various target analytes were detected in the field blanks. The contaminants present in each field blank are documented in the data validation worksheets. In all cases, the contaminants were either not present in the associated samples, or were present at concentrations greater than the action levels. No data were qualified based on rinsate blank contamination.

Field Replicate Samples

The field replicate sample included field splits (aliquots from the same sampling location) and replicates (aliquots from a nearby location). Several of the RPD and/or absolute difference values for field replicate analyses did not meet the criteria for acceptable precision. The field replicate precision outliers are discussed in greater detail in the data validation reports. No data were qualified based on field precision; however, users of the data should consider the potential impact of precision outliers on the reported results.

SUMMARY OF DATA VALIDATION: DIOXIN/FURAN COMPOUNDS

A total of 83 sediment samples were analyzed for dioxin/furan compounds for the Portland Harbor Round 3B Sediment Sampling event. The sediment samples included six (6) field replicates and four (4) field split samples. Thirteen (13) rinsate blanks were collected to monitor the field collection process. Columbia Analytical Services, Houston, Texas performed the analyses.

The dioxin/furan data for the sediment samples were generally acceptable. No data were rejected for any reason. A total of nine data points (0.43% of all sediment dioxin/furan results) were estimated because control limits were exceeded in one or more laboratory quality control (QC) samples or procedures. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

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

Completeness of Data Set

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

Holding Times and Sample Preservation

The initial sample preservation requirement (cooler temperature of 4°C ±2°) was not met for all samples. The majority of the sample coolers were received at the laboratory with temperatures outside the advisory control limits of 2° to 6°C. These temperature outliers did not impact data quality and no action was taken.

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 all acceptance criteria.

Method Blank Analyses

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

Method blanks were analyzed at the appropriate frequency. Various target analytes were detected in the method blanks. A total of 18 data points (0.87% of all dioxin/furan sediment data) were qualified as not detected (U) based on method blank contamination.

Accuracy

Labeled Compound Recovery

Labeled compounds were added to all field and QC samples. The labeled compound recovery values reported by the laboratory typically met the criteria for acceptable performance; however, a recovery outlier was present in one sample. One data point was estimated (J) based on labeled compound recovery outliers.

Matrix Spike Recovery

One matrix and duplicate matrix spike (MS/MSD) analysis was performed. The recovery values reported by the laboratory typically met the criteria for acceptable performance.

Ongoing Precision and Recovery Sample Recovery

Ongoing precision recovery (OPR) analyses met the criteria for frequency of analysis. The recovery values reported by the laboratory met the criteria for acceptable performance.

Precision

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

Method Detection Limits and Method Reporting Limits

To try to meet the project analytical concentration goals (ACG), the laboratory reported non-detects at the method detection limits (MDLs), adjusted for sample size, percent moisture, and any dilution factor. These method reporting limits (MRLs) ranged from 0.002 pg/g to 0.405 pg/g for the non-detected results. However, the ACG were not met for many of the dioxin/furan congeners. No action was taken.

Compound Identification and Quantitation

Flags were used by the laboratory to provide information about the reported results. A “K” flag indicates that a peak was detected at the correct retention time for the target analyte; however, the ion abundance ratio criteria were not met. The reported result is an EMPC (estimated maximum possible concentration) value, which is essentially an elevated detection limit. Data flagged “K” by the laboratory were qualified as not detected (U) to make this relationship clear to the data user. A total of 96 data points (4.6% of all sediment dioxin/furan data points) were qualified as not detected because the ion abundance ratio criteria were not met.

A laboratory “E” flag indicates that the reported result is greater than the upper calibration range established by the initial calibration. If no dilution analysis was performed, the “E” flagged data were estimated (J). Eight (8) data points (0.39% of all sediment dioxin/furan data points) were estimated based on calibration range exceedance.

Field Quality Control Samples

Field QC samples collected for the Round 3B sediments included field blank and field replicate samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Various target analytes were detected in the field blanks. The contaminants present in each field blank are documented in the data validation worksheets. In all cases, the contaminants were either not present in the associated samples, or were present at concentrations greater than the action levels. No data were qualified based on rinsate blank contamination.

Field Replicate Samples

The field replicate sample included field splits (aliquots from the same sampling location) and replicates (aliquots from a nearby location). Several of the RPD and/or absolute difference values for field replicate analyses did not meet the criteria for acceptable precision. The field replicate precision outliers are discussed in greater detail in the data validation reports. No data were qualified based on field precision; however, users of the data should consider the potential impact of precision outliers on the reported results.

SUMMARY OF DATA VALIDATION: METALS

A total of 431 sediment samples were analyzed for total metals for the Portland Harbor Round 3B Sediment Sampling event. The sediment samples included 26 field replicates and 14 field split samples. Twenty (20) rinsate blanks were collected to monitor the field collection process. Columbia Analytical Services, Kelso, Washington performed the analyses.

The following analytical methods were used:

Parameter	Method
ICP Metals	SW6010B
ICP-MS Metals	SW6020
Mercury	SW7471A/SW7470A

The metals data for the sediment samples were generally acceptable. No data were rejected for any reason. A total of 756 data points (15.9% of all sediment metals results) were estimated because control limits were exceeded in one or more laboratory quality control (QC) samples or procedures. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

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

Holding Times and Sample Preservation

The initial sample preservation requirement (cooler temperature of 4°C ±2°) was not met for all samples. The majority of the sample coolers were received at the laboratory with temperatures outside the advisory control limits of 2° to 6°C, ranging from -0.8° to 7.9°C. These temperature outliers did not impact data quality and no action was taken.

Instrument Performance

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

Method Blank Analyses

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank. If a contaminant is detected in

an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U). No action is taken if the sample result is greater than the action level, or for non-detected results.

Laboratory method blanks and instrument blanks were analyzed at the appropriate frequency. A total of 14 cadmium results (0.3% of all metals results) were qualified as not detected based on laboratory blank contamination. A total of 16 antimony results (0.34% of all metals results) were qualified as estimated based on potential low bias exhibited in laboratory blanks.

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).

Matrix Spike Recovery

MS analyses met the criteria for frequency of analysis. All of the recovery values reported by the laboratory for the antimony analyses did not meet the criteria for acceptable performance, with all outliers indicating a potential low bias. A total of 449 metals results (9.5% overall) were estimated (J) during the quality assurance review because the control limits for MS recovery were not met.

Laboratory Control Sample Recovery

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

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 recoveries less than the 70% lower control limit, positive results less than twice the CRDL and non-detects are estimated (J/UJ) to indicate a potential low bias. Several CRDL outliers were present (high bias); however, all associated results were greater than two times the CRDL, so no data were qualified based on CRDL outliers.

Interference Check Samples

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

Serial Dilution Analyses

Serial dilution analyses were performed at the proper frequency. Serial dilution %D values greater than 10% for sample results greater than 50 times the MDL may indicate the presence of matrix interference, resulting in potential bias. For serial dilution outliers, all associated sample results were qualified. A total of 181 metals results (3.8% of all sediment metals results) were estimated (J/UJ) based on serial dilution outliers.

Precision

Laboratory duplicate analyses were evaluated for laboratory precision. Several relative percent difference (RPD) values were outside the acceptance limits. A total of 128 results (2.7% of all sediment metals results) were estimated (J) based on laboratory precision outliers.

Method Detection Limits and Method Reporting Limits

The laboratory reported non-detects at the MDLs, adjusted for sample size and any dilution factor. With the exception of three mercury results (MDL 0.005 mg/Kg), all metals were detected in all samples.

Field Quality Control Samples

Field QC samples collected for the Round 3B sediments included field blank and field replicate samples. The results for the field QC samples are discussed in the following sections.

Field Blanks

Twenty (20) field (rinsate) blanks were associated with the sediment samples. Various target analytes were detected in the field blanks. The contaminants present in each field blank are documented in the data validation worksheets.

In most cases, these analytes were either not detected or were present at concentrations greater than the action levels in the associated samples. Thirteen (13) results (all chromium) were qualified as not detected based on rinsate blank contamination.

Field Replicate Samples

The field replicate sample included field splits (aliquots from the same sampling location) and replicates (aliquots from a nearby location). Several of the RPD and/or absolute difference values for field replicate analyses did not meet the criteria for acceptable precision. The field replicate precision outliers are discussed in greater detail in the data validation reports. No data were qualified based on field precision; however, users of the data should consider the potential impact of precision outliers on the reported results.

SUMMARY OF DATA VALIDATION: CONVENTIONAL PARAMETERS

A total of 431 sediment samples were analyzed for some or all of the following parameters for the Portland Harbor Round 3B Sediment Sampling event. The sediment samples included 26 field replicates and 14 field split samples. Columbia Analytical Services, Kelso, Washington, completed all analyses. The following analytical methods were used:

Parameter	Method Number
Total Solids (TS)	EPA 160.3
Grain Size (GS)	PSEP 1986
Specific Gravity (SG)	ASTM D-854*83
Total Organic Carbon (TOC)	PSEP 1986
Sulfides	SW9030
Ammonia	EPA 350.1

Overall, the conventional parameters data for the sediment samples were acceptable. No data were rejected for any reason. Sixty-six (66) data points of 6,965 total data points (0.95% of all sediment conventional parameters data points) were estimated because control limits were exceeded in one or more laboratory quality control (QC) samples or procedures. These qualified data points may have a larger associated bias or may be less precise than unqualified data, but are usable for the intended purpose.

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

Completeness of Data Set

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

Holding Times and Sample Preservation

The initial sample preservation requirement (cooler temperature of $4^{\circ}\text{C} \pm 2^{\circ}$) was not met for all samples. The majority of the sample coolers were received at the laboratory with temperatures outside the advisory control limits of 2° to 6°C , ranging from -0.8° to 7.9°C . These temperature outliers did not impact data quality and no action was taken.

Thirty-three (33) of the sulfide and one of the TOC analyses were performed past the holding time limit specified in the QAPP. These results were estimated (J/UJ).

Instrument Performance

Initial and continuing calibrations were completed for the TOC and hexavalent chromium 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

Two types of laboratory blanks were evaluated for possible contamination effects. These blanks were: initial and continuing calibration blanks (ICB and CCB) and method blanks (MB). The required frequency of one at the beginning and one every ten samples for calibration blank analysis was met. The laboratory analyzed one MB for every 20 samples digested or one per batch, for each digestion procedure, as required. TOC was detected in one calibration blank. All associated results were greater than the action limit, no data were qualified. No other target analytes were detected in any 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 or matrix spike duplicate [MSD] analyses).

Matrix Spike Recovery

MS analyses were completed for the TOC analyses and met the criteria for frequency of analysis. All TOC MS recovery values were acceptable.

Laboratory Control Sample Recovery

An LCS was analyzed for the TOC analyses. All LCS recovery values were acceptable.

Precision

Laboratory duplicate and triplicate analyses (for grain size) were evaluated for laboratory precision. Several of the percent relative standard deviation (%RSD) values for grain size did not meet the criteria for acceptable performance. Thirty-three results (20 ammonia and 13 grain size results) were estimated (J) during the quality assurance review because control limits for relative percent difference (RPD) and %RSD were not met.

Method Reporting Limits

The project method reporting limit goals were met for all conventional parameters.

Field Quality Control Samples

Field QC samples collected for the Round 3B sediments included field replicate samples. The results for the field QC samples are discussed in the following sections.

Field Replicate Samples

The field replicate sample included field splits (aliquots from the same sampling location) and replicates (aliquots from a nearby location). Several of the RPD and/or absolute difference values for field replicate analyses did not meet the criteria for acceptable precision. The field replicate precision outliers are discussed in greater detail in the data validation reports. No data were qualified based on field precision; however, users of the data should consider the potential impact of precision outliers on the reported results.



EcoChem, INC.
Environmental Data Quality

APPENDIX A

**DATA QUALIFIER DEFINITIONS,
REASON CODES, AND CRITERIA TABLES**

DATA VALIDATION QUALIFIER CODES National Functional Guidelines

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

U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a “tentative identification”.
NJ	The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents the approximate concentration.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

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

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

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

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

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler/Storage Temperature	Aqueous/Soil/Sediment <4°C Tissues <-10°C	J(+)/UJ(-) if greater than 6 deg. C (EcoChem PJ)	1
Holding Time	Water: 7 days from collection Soil: 14 days from collection Analysis: 40 days from extraction	<u>Water:</u> J(+)/UJ(-) if ext. > 7 and < 21 days J(+)/R(-) if ext > 21 days (EcoChem PJ) <u>Solids/Wastes:</u> J(+)/UJ(-) if ext. > 14 and < 42 days J(+)/R(-) if ext. > 42 days (EcoChem PJ) J(+)/UJ(-) if analysis >40 days	1
Tuning	DFTPP Beginning of each 12 hour period Method acceptance criteria	R(+/-) all analytes in all samples associated with the tune	5A
Initial Calibration (Minimum 5 stds.)	RRF > 0.05	(EcoChem PJ, see TM-06) If MDL= reporting limit: J(+)/R(-) if RRF < 0.05 If reporting limit > MDL: note in worksheet if RRF <0.05	5A
	%RSD < 30%	(EcoChem PJ, see TM-06) J(+) if %RSD > 30%	5A
Continuing Calibration (Prior to each 12 hr. shift)	RRF > 0.05	(EcoChem PJ, see TM-06) If MDL= reporting limit: J(+)/R(-) if RRF < 0.05 If reporting limit > MDL: note in worksheet if RRF <0.05	5B
	%D <25%	(EcoChem PJ, see TM-06) If > +/-90%: J+/R- If -90% to -26%: J+ (high bias) If 26% to 90%: J+/UJ- (low bias)	5B
Method Blank	One per matrix per batch No results > QL	U(+) if sample (+) result is less than QL and less than appropriate 5X or 10X rule (raise sample value to QL)	7
		U(+) if sample (+) result is greater than or equal to QL and less than appropriate 5X and 10X rule (at reported sample value)	7
	No TICs present	R(+) TICs using 10X rule	7
Field Blanks	No results > QL	Apply 5X/10X rule; U(+) < action level	6

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

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
MS/MSD (recovery)	One per matrix per batch Use method acceptance criteria	Qualify parent only unless other QC indicates systematic problems: J(+) if both %R > UCL J(+)/UJ(-) if both %R < LCL J(+)/R(-) if both %R < 10% PJ if only one %R outlier	8
MS/MSD (RPD)	One per matrix per batch Use method acceptance criteria	J(+) if RPD > CL	9
Lab Duplicate	Results >5X reporting limit (RL): RPD criteria: use QAPP limits, all matrices Results <5X RL: Solid: Absolute difference < 2X RL Aqueous: Absolute difference < 1X RL	J(+)/UJ(-) if outside limits	9
LCS low conc. H2O SVOA	One per lab batch Within method control limits	J(+) assoc. cmpd if > UCL J(+)/R(-) assoc. cmpd if < LCL J(+)/R(-) all cmpds if half are < LCL	10
LCS regular SVOA (H2O & solid)	One per lab batch Lab or method control limits	J(+) if %R > UCL J(+)/UJ(-) if %R < LCL J(+)/R(-) if %R < 10% (EcoChem PJ)	10
LCS/LCSD (if required)	One set per matrix and batch of 20 samples RPD < 35%	J(+) assoc. cmpd. in all samples	9
Surrogates	Minimum of 3 acid and 3 base/neutral compounds Use method acceptance criteria	Do not qualify if only 1 acid and/or 1 B/N surrogate is out unless <10% J(+) if %R > UCL J(+)/UJ(-) if %R < LCL J(+)/R(-) if %R < 10%	13
Internal Standards	Added to all samples Acceptable Range: IS area 50% to 200% of CCAL area RT within 30 seconds of CC RT	J(+) if > 200% J(+)/UJ(-) if < 50% J(+)/R(-) if < 25% R T>30 seconds, narrate and Notify PM	19
Field Duplicates	Results >5X reporting limit (RL): RPD < 50% (all matrices, QAPP specified) Results <5X RL: Solid: Absolute difference < 2X RL Aqueous: Absolute difference < 1X RL	Narrate; do not qualify	na
TICs	Major ions (>10%) in reference must be present in sample; intensities agree within 20%; check identification	R(+) common laboratory contaminants R(+) target compounds from other fractions See Technical Director for ID issues	4
Quantitation/ Identification	RRT within 0.06 of standard RRT Ion relative intensity within 20% of standard All ions in std. at > 10% intensity must be present in sample	See Technical Director if outliers	14 21 (false +)

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

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

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

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

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

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
MS/MSD (RPD)	May not analyze MS/MSD RPD < 20%	J(+) if RPD > CL	9
Lab Duplicate	RPD < 25% if present.	J(+) if outside limits	9
Labeled Compounds Internal Standards	<i>Method 8290</i> : %R = 40% - 135% in all samples <i>Method 1668</i> : %R = 25% - 150% in all samples <i>Method 1613B</i> : %R must meet limits specified in Table 7	J(+)/UJ(-) if %R = 10% to LCL J(+) if %R > UCL J(+)/R(-) if %R < 10%	13
Quantitation/ Identification	Ions for analyte, IS, and rec. std. must max w/in 2 sec. S/N > 2.5 IA ratios meet limits in Table 9 of 1613B or Table 8 of 8290 RRTs w/in limits in table 2 of 1613B	If RT criteria not met, use PJ (see TM-05) If S/N criteria not met, J(+). if unlabelled ion abundance not met, change to EMPC If labelled ion abundance not met, J(+).	21
EMPC (est. max. possible concentration)	If quantitation identification criteria are not met, laboratory should report an EMPC value.	If laboratory correctly reported an EMPC value, qualify with U to indicate that the value is a detection limit.	14
Interferences	PCDF interferences from PCDPE	If both detected, change PCDF result to EMPC	14
Second Column Confirmation	All 2,3,7,8-TCDF hits must be confirmed on a DB-225 column (or equiv). All QC specs in this table must be met for the confirmation analysis.	Report lower of the two values. If not performed use PJ (see TM-05).	3
Field Duplicates	QAPP specified RPD < 50% (all matrices)	Narrate; do not qualify	na
Two analyses for one sample	Report only one result per analyte	"DNR" results that should not be used	11

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

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

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

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

DATA VALIDATION CRITERIA

EcoChem Validation Guidelines for Total Petroleum Hydrocarbons-Diesel & Residual Range (Based on EPA National Functional Guidelines as applied to criteria in NWTPH-Dx, June 1997, Wa DOE & Oregon DEQ)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler Temperature & Preservation	4°C±2°C Water: HCl to pH < 2	J(+)/UJ(-) if greater than 6 deg. C	1
Holding Time	Ext. Waters: 14 days preserved 7 days unpreserved Ext. Solids: 14 Days Analysis: 40 days from extraction	J(+)/UJ(-) if hold times exceeded J(+)/R(-) if exceeded > 3X (EcoChem PJ)	1
Initial Calibration	5 calibration points (All within 15% of true value) Linear Regression: R ² ≥ 0.990 If used, RSD of response factors ≤ 20%	Narrate if fewer than 5 calibration levels or if %R > 15% J(+)/UJ(-) if R ² < 0.990 J(+)/UJ(-) if %RSD > 20%	5A
Mid-range Calibration Check Std.	Analyzed before and after each analysis shift & every 20 samples. Recovery range 85% to 115%	Narrate if frequency not met. J(+)/UJ(-) if %R < 85% J(+) if %R > 115%	5B
Method Blank	At least one per batch (≤10 samples) No results >RL	U (at the RL) if sample result is < RL & < 5X blank result.	7
		U (at reported sample value) if sample result is ≥ RL and < 5X blank result	7
Field Blanks (if required by project)	No results > RL	Action is same as method blank for positive results remaining in the field blank after method blank qualifiers are assigned.	6
MS samples (accuracy) (if required by project)	%R within lab control limits	Qualify parent only, unless other QC indicates systematic problems. J(+) if both %R > upper control limit (UCL) J(+)/UJ(-) if both %R < lower control limit (LCL) No action if parent conc. > 5X the amount spiked. Use PJ if only one %R outlier	8
Precision: MS/MSD or LCS/LCSD or sample/dup	At least one set per batch (≤10 samples) RPD ≤ lab control limit	J(+) if RPD > lab control limits	9
LCS (not required by method)	%R within lab control limits	J(+)/UJ(-) if %R < LCL J(+) if %R > UCL J(+)/R(-) if any %R < 10% (EcoChem PJ)	10

EcoChem Validation Guidelines for Total Petroleum Hydrocarbons-Diesel & Residual Range
 (Based on EPA National Functional Guidelines as applied to criteria in NWTPH-Dx,
 June 1997, Wa DOE & Oregon DEQ)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Surrogates	2-fluorobiphenyl, p-terphenyl, o-terphenyl, and/or pentacosane added to all samples (inc. QC samples). %R = 50-150%	J(+)/UJ(-) if %R < LCL J(+) if %R > UCL J(+)/R(-) if any %R <10% No action if 2 or more surrogates are used, and only one is outside control limits. (EcoChem PJ)	13
Pattern Identification	Compare sample chromatogram to standard chromatogram to ensure range and pattern are reasonable match. Laboratory may flag results which have poor match.	J(+)	2
Field Duplicates	Use project control limits, if stated in QAPP EcoChem default: water: RPD < 35% solids: RPD < 50%	Narrate (Use Professional Judgement to qualify)	9
Two analyses for one sample (dilution)	Report only one result per analyte	"DNR" (or client requested qualifier) all results that should not be reported. (See TM-04)	11

DATA VALIDATION CRITERIA

Table No.: Integral-ICP

Revision No.: 1

Last Rev. Date: 12/12/05

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

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler/Storage Temperature and Preservation	Aqueous/Soil/Sediment <4°C Tissues <-10°C Water Only: Nitric Acid to pH < 2 For Dissolved metals, 0.45 um filter preserve after filtration	EcoChem PJ J(+)/UJ(-) if preservation requirements are not met	1
Holding Time	180 days	EcoChem PJ J(+)/UJ(-)	1
Initial Calibration	Blank + minimum 1 standard once every 24 hours if more than 1 standard $r > 0.995$	EcoChem PJ J(+)/UJ(-) if $r < 0.995$ (multi point cal)	5A
Initial Calibration Verification (ICV)	Independent source analyzed immed. after cal. %R within +/- 10% of true value	EcoChem PJ J(+)/UJ(-) if %R 75%-89% J(+) if %R = 111-125% R(+) if %R > 125% R(+/-) if %R < 75%	5A
Continuing Cal Verification (CCV)	Every ten samples, immed. Before samples+ and end of run %R within +/- 10% of true value	EcoChem PJ J(+)/UJ(-) if %R = 75%-89% J(+) if %R 111-125% R(+) if %R > 125% R(+/-) if %R < 75%	5B
CRI Standard (to check RL)	2X RL (or 2X IDL if greater) analyzed beginning and end of run (at least 8 hrs) Not required for Al, Ba, Ca, Fe, Mg, Na, K %R = 70%-130% (50%-150% Sb, Pb, TI)	EcoChem PJ R(-),(+) < 2XRL if %R < 50% (< 30% Sb, Pb, TI) J(+)<2XRL, UJ(-) if %R 50-69% (30%-49% Sb, Pb, TI) J(+) < 2X RL if %R 130%-180% (150%-200% Sb, Pb, TI) R(+)<2X RL if %R > 180% (200% Sb, Pb, TI)	14
Initial and Continuing Cal Blanks (ICB/CCB)	after each ICV and CCV every ten samples and end of run blank < +/- IDL	Action level is 5x abs. value of blk conc. For (+) blk value, U(+) values < action level For (-) blk value, J(+)/UJ(-) values < action level	7
Prep Blank	One per matrix per batch (not to exceed 20 samples)	Action level is 5x abs. value of blk conc. For (+) blk value, U(+) values < action level For (-) blk value, J(+)/UJ(-) values < action level	7
Interference Check Samples ICSA/ICSAB	Beginning and end of each run or every eight hours ICSAB +/- 20% ICSA < +/- IDL	For samp with Al, Ca, Fe, Mg > ICS levels R(+/-) if %R < 50% J(+) if %R > 120% J(+)/UJ(-) if %R = 50% to 79% EcoChem PJ ICSA	17
Post Digestion Spike	If ICP Matrix Spike is outside 75-125%, spike at twice the sample conc.	No Quals assigned based on this element	

DATA VALIDATION CRITERIA

Table No.: Integral-ICP

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

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

DATA VALIDATION CRITERIA

Table No.: Integral-ICPMS

Revision No.: 1

Last Rev. Date: 12/12/05

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

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler/Storage Temperature and Preservation	Aqueous/Soil/Sediment <4°C Tissues <-10°C Water Only: Nitric Acid to pH < 2 For Dissolved metals, 0.45 um filter preserve after filtration	EcoChem PJ J(+)/UJ(-) if preservation requirements are not met	1
Holding Time	180 days	EcoChem PJ J(+)/UJ(-) if holding time exceeded J(+)/R(-) if HT exceeded by 3x	1
Tune	Prior to ICAL Analyzed 5 times with Std Dev. ≤ 5% mass calibration <0.1 amu from True Value Resolution < 0.9 AMU @ 10% peak height or <0.75 amu @ 5% peak height	EcoChem PJ No Tune - R all results criteria not met - J(+)/UJ(-)	5A
Initial Calibration	Minimum Blank+1 Standard every 24 hours	EcoChem PJ J(+)/UJ(-) >24 hours J(+)/UJ(-) if r<0.995 (for multi point cal)	5A
Initial Calibration Verification (ICV)	Independent source; analyzed post ICAL and prior to samples +/-10% of the True value	EcoChem PJ J(+)/UJ(-) if %R 75%-89% J(+) if %R = 111-125% R(+) if %R > 125% R(+/-) if %R < 75%	5A
Continuing Cal Verification (CCV)	Every 10 samples, post ICV/ICB and end of run +/- 10% of True value	EcoChem PJ J(+)/UJ(-) if %R 75%-89% J(+) if %R = 111-125% R(+) if %R > 125% R(+/-) if %R < 75%	5B
RL Standard (CRI)	2X RL (or 2X IDL if greater) analyzed beginning and end of run (at least 8 hrs) Not required for Al, Ba, Ca, Fe, Mg, Na, K %R = 70%-130% (50%-150% Co, Mn, Zn)	EcoChem PJ R(-),(+) < 2XRL if %R < 50% (< 30% Co, Mn, Zn) J(+)<2XRL, UJ(-) if %R 50-69% (30%-49% Co, Mn, Zn) J(+)<2X RL if %R 130%-180% (150%-200% Co, Mn, Zn) R(+)<2X RL if %R > 180% (200% Co, Mn, Zn)	14
Initial and Continuing Cal Blanks (ICB/CCB)	after each ICV and CCV every ten samples and end of run blank < +/- IDL	Action level is 5x abs. value of blk conc. For (+) blk value, U(+) values < AL For (-) blk value, J(+)/UJ(-) values < AL	7
Prep Blank	One per matrix per batch (not to exceed 20 samples)	Action level is 5x abs. value of blk conc. For (+) blk value, U(+) values < AL For (-) blk value, J(+)/UJ(-) values < AL	7
Field Blanks	No results > QL	Apply 5X rule; U(+) < action level	6

DATA VALIDATION CRITERIA

Table No.: Integral-ICPMS

Revision No.: 1

Last Rev. Date: 12/12/05

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

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

DATA VALIDATION CRITERIA

Table No.: Integral-HG
 Revision No.: 1
 Last Rev. Date: 12/12/05
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Integral - Portland Harbor Site Mercury by CVAA (Based on Inorganic NFG 1994 & 2002)

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler/Storage Temperature and Preservation	Aqueous/Soil/Sediment <4°C Tissues <-10°C Water Only: Nitric Acid to pH < 2 For Dissolved metals, 0.45 um filter preserve after filtration	EcoChem PJ J(+)/UJ(-) if preservation requirements are not met	1
Holding Time	28 days from date sampled	EcoChem PJ J(+)/UJ(-) if holding time exceeded	1
Initial Calibration	Blank + 4 standards $r > 0.995$ once every 24 hours	EcoChem PJ J(+)/UJ(-) if $r < 0.995$	5A
Initial Calibration Verification (ICV)	Independent source analyzed immediately after cal. %R within +/- 20% of true value	EcoChem PJ R(+/-) if %R < 65% R(+) if %R > 135% J(+)/UJ(-) if %R = 65%-79% J(+) if %R = 121-135%	5A
Continuing Cal Verification (CCV)	Every ten samples, immed. following ICV/ICB and end of run %R within +/- 20% of true value	R(+/-) if %R < 65% R(+) if %R > 135% J(+)/UJ(-) if %R = 65%-79% J(+) if %R = 121-135%	5B
RL Standard (CRA)	Beginning of run after ICV/ICB CCV/CCB Conc = RL 70% - 130%	EcoChem PJ R(-),(+) < 2XRL if %R < 50% J(+)<2XRL, UJ(-) if %R 50-69% J(+) < 2X RL if %R 130%-180% R(+)<2X RL if %R>180%	14
Initial and Continuing Cal Blanks (ICB/CCB)	After each ICV and CCV every ten samples and end of run blank < +/- IDL	Action level is 5x abs. value of blk conc. For (+) blk value, U(+) sample values < AL For (-) blk value, J(+)/UJ(-) sample values < AL	7
Prep Blank	One per matrix per batch (not to exceed 20 samples)	Action level is 5x abs. value of blk conc. For (+) blk value, U(+) sample values < AL For (-) blk value, J(+)/UJ(-) sample values < AL	7
Matrix Spike	One per matrix per batch 5% frequency 75-125% for samples less than 4x spike level	J(+) if %R > 125% J(+)/UJ(-) if %R < 75% J(+)/R(-) if %R < 30%	8
Laboratory Duplicate	One per matrix per batch RPD < 20% for samples > 5x RL (+/-)RL for samples > RL and < 5 x RL (may use RPD < 35%, Diff < 2X RL for solids)	J(+)/UJ(-) if RPD > 20% or diff > RL	9

DATA VALIDATION CRITERIA

Table No.: Integral-HG
 Revision No.: 1
 Last Rev. Date: 12/12/05
 Page: 2 of 2

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

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Laboratory Control Sample	Waters: One per matrix per batch %R (80-120%)	R(+/-) if %R < 50%; J(+) if %R > 120% J(+)/UJ(-) if %R = 50-79%	10
	Soils: One per matrix per batch Result within manufacturer's certified acceptance range	J(+)/UJ(-) if < LCL, J(+) if > UCL	10
Field Duplicates	QAPP specified RPD < 50% (all matrices)	Narrate; do not qualify	na

DATA VALIDATION CRITERIA

Table No.: Eco-Conv
 Revision No.: 0
 Last Rev. Date: FINAL DRAFT
 Page: 1 of 2

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

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Cooler Temperature and Preservation	Cooler Temperature 4°C ±2°C Preservation: Method Specific	Use Professional Judgment to qualify based to qualify for cooler temp outliers J(+)/UJ(-) if preservation requirements not met	1
Holding Time	Method Specific	Professional Judgment J(+)/UJ(-) if holding time exceeded J(+)/R(-) if HT exceeded by > 3X	1
Initial Calibration	Method specific r>0.995	Use professional judgment J(+)/UJ(-) for r < 0.995	5A
Initial Calibration Verification (ICV)	Where applicable to method Independent source analyzed immediately after calibration %R method specific, usually 90% - 110%	R(+/-) if %R significantly < LCL J(+)/UJ(-) if %R < LCL J(+) if %R > UCL R(+) if %R significantly > UCL	5A
Continuing Cal Verification (CCV)	Where applicable to method Every ten samples, immed. following ICV/ICB and end of run %R method specific, usually 90% - 110%	R(+/-) if %R significantly < LCL J(+)/UJ(-) if %R < LCL J(+) if %R > UCL R(+) if %R significantly > UCL	5B
Initial and Continuing Cal Blanks (ICB/CCB)	Where applicable to method After each ICV and CCV every ten samples and end of run blank < MDL	Action level is 5x absolute value of blank conc. For (+) blanks, U(+) results < action level For (-) blanks, J(+)/UJ(-) results < action level refer to TM-02 for additional details	7
Method Blank	One per matrix per batch (not to exceed 20 samples) blank < MDL	Action level is 5x absolute value of blank conc. For (+) blk value, U(+) results < action level For (-) blk value, J(+)/UJ(-) results < action level	7
Laboratory Control Sample	Waters: One per matrix per batch %R (80-120%)	R(+/-) if %R < 50% J(+)/UJ(-) if %R = 50-79% J(+) if %R >120%	10
	Soils: One per matrix per batch Result within manufacturer's certified acceptance range	J(+)/UJ(-) if < LCL, J(+) if > UCL	10
Matrix Spike	One per matrix per batch; 5% frequency 75-125% for samples less than 4 x spike level	J(+) if %R > 125% or < 75% UJ(-) if %R = 30-74% R(+/-) results < IDL if %R < 30%	8
Laboratory Duplicate	One per matrix per batch RPD <20% for samples > 5x RL Diff <RL for samples >RL and < 5 x RL (may use RPD < 35%, Diff < 2X RL for solids)	J(+)/UJ(-) if RPD > 20% or diff > RL all samples in batch	9

DATA VALIDATION CRITERIA

Table No.: Eco-Conv
 Revision No.: 0
 Last Rev. Date: FINAL DRAFT
 Page: 2 of 2

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

VALIDATION QC ELEMENT	ACCEPTANCE CRITERIA	ACTION	REASON CODE
Field Blank	blank < MDL	Action level is 5x blank conc. U(+) sample values < action level in associated field samples only	6
Field Duplicate	For results > 5X RL: Water: RPD < 35% Solid: RPD < 50% For results < 5 x RL: Water: Diff < RL Solid: Diff < 2X RL	J(+)/UJ(-) in parent samples only	9



EcoChem, INC.
Environmental Data Quality

APPENDIX B

DATA VALIDATION REPORTS

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3B Sediments
Semivolatile Organic Compounds by Method SW8270C

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

SDG	Number of Samples	Validation Level
K0710859	20 Sediment	Summary
K0710866	12 Sediment	Summary
K0710893	24 Sediment & 2 Equipment Blank	Summary
K0711067	19 Sediment	Summary
K0711172	20 Sediment	Summary
K0711174	21 Sediment & 1 Equipment Blank	Summary
K0711175	12 Sediment & 2 Equipment Blank	Full
K0711220	23 Sediment & 1 Equipment Blank	Summary
K0711252	19 Sediment & 2 Equipment Blank	Summary
K0711320	4 Sediment	Summary
K0711423	27 Sediment & 2 Equipment Blank	Summary
K0711577	4 Sediment	Summary
K0711706	19 Sediment & 1 Equipment Blank	Summary
K0711709	10 Sediment	Full
K0711828	13 Sediment & 2 Equipment Blank	Summary
K0711830	20 Sediment	Summary
K0712050	15 Sediment & 1 Equipment Blank	Summary
K0712101	9 Sediment	Full
K0712106	9 Sediment & 1 Equipment Blank	Summary
K0712149	6 Sediment	Summary
K0800158	28 Sediment & 2 Equipment Blank	Summary
K0800270	21 Sediment & 1 Equipment Blank	Summary
K0800325	13 Sediment & 1 Equipment Blank	Full
K0800450	27 Sediment	Summary
K0800487	22 Sediment	Summary
K0800516	14 Sediment & 1 Equipment Blank	Full

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

A complete (100%) verification of the 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 | Laboratory Control Samples (LCS/LCSD) |
| | GC/MS Instrument Performance Check | 1 | Field Replicates |
| | Initial Calibration (ICAL) | | Internal Standards |
| 2 | Continuing Calibration (CCAL) | 1 | Target Analyte List |
| 2 | Laboratory Blanks | 1 | Reporting Limits |
| 2 | Field Blanks | 1 | Compound Identification |
| 2 | Surrogate Compounds | 1 | Calculation Verification (Full validation only) |
| 2 | Matrix Spikes/Matrix Spike Duplicates (MS/MSD) | | |

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

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

Holding Times and Sample Receipt

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of 2° to 6°C. The laboratory received many of the sample coolers with temperatures outside the advisory control limits. The temperature outliers ranged from -0.8° to 7.9°C. These temperature outliers did not impact data quality and no qualifiers were required.

Continuing Calibration

All relative response factor (RRF) values were greater than the 0.05 minimum control limit. All percent difference (%D) values were within the ±25% control limit for all continuing calibrations (CCAL), with the exceptions noted below. When the %D outlier indicates a potential high bias, and there were no positive results for these compounds, no qualifiers were required.

SDG K0710859: CCAL 12/21/07: benzoic acid (high bias)

SDG K0710893: CCAL 12/7/07: hexachlorocyclopentadiene (low bias)

SDGs K0711174, K0711175, K0711220, K0711252:

- CCAL 12/11/07: hexachlorocyclopentadiene (low bias)

SDG K0712101: CCAL 2/7/08: n-nitrosodimethylamine (high bias)

SDG K0712149: CCAL 1/29/08: bis (2-chloroisopropyl) ether (low bias)

SDG K0800158:

- CCAL 1/31/08: nitrobenzene (high bias), 2,4-dinitrotoluene (high bias), 2-methyl-4,6-dinitrophenol (high bias)
- CCAL 2/6/08: bis(2-chloroisopropyl)ether (high bias)

SDG K0800270: CCAL 2/7/08: n-nitrosodimethylamine (high bias)

SDG K0800325: CCAL 2/8/08: bis (2-chloroisopropyl) ether (low bias)

SDG K0800450:

- CCAL 2/8/08: bis (2-chloroisopropyl) ether (low bias)
- CCAL 2/10/08: 2,4-dinitrophenol (low bias)
- CCAL 2/15/08: 2,4-dinitrophenol (low bias)

SDG K0800487:

- CCAL 2/10/08: 2,4-dinitrophenol (low bias)
- CCAL 2/15/08: 2,4-dinitrophenol (low bias)

SDG K0800516: CCAL 2/10/08: 2,4-dinitrophenol (low bias)

Laboratory Blanks

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times the concentration reported in the blank (ten times for phthalates). If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified U-7 at the reported concentration to indicate an elevation of the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Laboratory (method) blanks were analyzed at the appropriate frequency. Various target analytes were detected in the method blanks. The detected analytes, concentrations, associated samples, and action levels are documented in the data validation worksheets. However, however only the following analytes required qualification in one or more samples in the associated laboratory data sets:

SDG K0710859: phenol (18 results)

SDG K0710866: phenol (12 results)

SDG K0710893: (aqueous): diethyl phthalate (2 results), di-n-butyl phthalate (2 results), and butyl benzyl phthalate (2 results)

SDG K0710893: (sediment): phenol (20 results) and diethyl phthalate (20 results)

SDG K0711174: (aqueous): phenol (1 result), diethyl phthalate (1 result), di-n-butyl phthalate (1 result), butyl benzyl phthalate (1 result), bis(2-ethylhexyl) phthalate (1 result)

SDG K0711175: (aqueous): phenol (2 results), diethyl phthalate (2 results), di-n-butyl phthalate (2 results), butyl benzyl phthalate (2 results), bis(2-ethylhexyl) phthalate (2 results)

SDG K0711220: (aqueous): phenol, diethyl phthalate, di-n-butyl phthalate, butyl benzyl phthalate, and bis(2-ethylhexyl) phthalate (1 result each)

SDG K0711252: (aqueous): phenol (1 result), diethyl phthalate (2 results), di-n-butyl phthalate (2 results), butyl benzyl phthalate (2 results), bis(2-ethylhexyl) phthalate (2 results)

SDG K0711423: (aqueous): diethyl phthalate (2 results), di-n-butyl phthalate (2 results), butyl benzyl phthalate (2 results), pyrene (2 results), fluoranthene (1 result)

SDG K0711423: (sediment): di-n-butyl phthalate (16 results), bis(2-ethylhexyl) phthalate (7 results)

SDG K0711577: (sediment): bis(2-ethylhexyl) phthalate (4 results)

SDG K0711706: (aqueous): di-n-butyl phthalate (1 result), diethyl phthalate (1 result), butyl benzyl phthalate (1 result), benzoic acid (1 result), benzyl alcohol (1 result)

SDG K0711828: (aqueous): diethyl phthalate (2 results), di-n-butyl phthalate (2 results), butyl benzyl phthalate (2 results), bis(2-ethylhexyl) phthalate (1 result), benzoic acid (2 results), benzyl alcohol (2 results)

SDG K0712050: (sediment): phenol (1 result)

SDG K0712050: (aqueous): dibutyl phthalate (1 result)

SDG K0712106 (sediment): phenol (4 results)

SDG K0712106 (aqueous): diethyl phthalate (1 result), di-n-butyl phthalate (1 result), butylbenzyl phthalate (1 result)

SDG K0712149: phenol (6 results)

SDG K0800158: (aqueous): diethyl phthalate (2 results), di-n-butyl phthalate (2 results), di-n-octyl phthalate (1 result), butylbenzyl phthalate (2 results), bis(2-ethylhexyl) phthalate (2 results)

SDG K0800158: (sediment): phenol (10 results)

SDG K0800270: (aqueous): di-n-butyl phthalate, butyl benzyl phthalate, bis(2-ethylhexyl) phthalate, benzoic acid (1 result each)

SDG K0800270: (sediment): diethyl phthalate (4 results), phenol (10 results)

SDG K0800325: (aqueous): benzoic acid, diethyl phthalate, di-n-butyl phthalate, butyl benzyl phthalate, bis(2-ethylhexyl) phthalate (1 result each)

SDG K0800325: (sediment): diethyl phthalate (4 results), phenol (4 results)

SDG K0800450: phenol (1 result)

SDG K0800516: (aqueous): benzyl alcohol, di-n-butyl phthalate (1 result each)

SDG K0800516: (sediment): phenol (1 result)

Field Blanks

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

The following analytes were detected in the field blanks. Except where noted below, the analytes were either not detected in the associated samples, or were present at concentrations greater than the action level.

SDG K0710893: Two equipment blanks were submitted, LW3-SAG901 & LW3-UG901. After qualifiers were issued based on method blank contamination, positive results for phenol and bis(2-ethylhexyl) phthalate remained in both of these blanks, and 1-methylnaphthalene was present in LW3-SAG901.

SDG K0711174: One equipment blank was submitted, LW3-SAG902. After qualifiers were issued based on method blank contamination, positive results for benzyl alcohol, naphthalene, and dibenzofuran were reported in this blank.

SDG K0711175: Two equipment blanks were submitted, LW3-SAG903 and LW3-SAG904. After qualifiers were issued based on method blank contamination, positive results for benzyl alcohol and naphthalene were reported in these blanks.

SDG K0711220: One equipment blank was submitted, LW3-MG901. After method blank contamination qualifiers were issued, positive results for benzyl alcohol and naphthalene remained.

SDG K0711252: Two equipment blanks were submitted, LW3-SAG905 and LW3-SAG906. After qualifiers were issued based on method blank contamination, positive results for naphthalene were reported in these field blanks.

SDG K0711423: Two equipment blanks were submitted, LW3-SAG907 and LW3-SAG908. After qualifiers were issued based on method blank contamination, a positive result for benzyl alcohol remained in blank LW3-SAG907 and a positive result for bis(2-ethylhexyl) phthalate in blank LW3-SAG908.

SDG K0711706: One equipment blank was submitted, LW3-MC901. After qualifiers were issued based on method blank contamination, a positive result for naphthalene remained. Five results were qualified as not detected (U-6). Two associated samples (in SDG K0712101) were also qualified as not detected (U-6) based on the naphthalene result in LW3-MC901.

SDG K0711828: Two equipment blanks were submitted, LW3-SAC901 and LW3-SAC902. After qualifiers were issued based on method blank contamination, 1,4-dichlorobenzene remained in both field blanks and azobenzene was detected in field blank LW3-SAC902.

SDG K0712050: One equipment blank was submitted, LW3-SAC903. Positive results for butylbenzyl phthalate, dibenzofuran, and diethyl phthalate were detected in this blank. Two butylbenzyl phthalate results and six diethyl phthalate results were qualified (U-6).

SDG K0712106: One equipment blank was submitted, LW3-SAC904. After qualifiers were issued based on method blank contamination, positive results for 1,4-dichlorobenzene and dibenzofuran remained. Two dibenzofuran results were less than the adjusted action level and were qualified as not detected (U-6) at the reported concentration.

SDG K0800158: Two equipment blanks were submitted, LW3-SAC905 and LW3-SAC906. After qualifiers were issued based on method blank contamination, 1,4-dichlorobenzene and naphthalene remained in both field blanks and a positive result for benzyl alcohol remained in field blank LW3-SAC906. Three 1,4-dichlorobenzene results were qualified as not detected (U-6) in the associated samples.

SDG K0800270: One equipment blank was submitted, LW3-SAC907. After qualifiers were issued based on method blank contamination, positive results remained for diethyl phthalate and naphthalene. The diethyl phthalate results in six associated samples were less than the action level and were qualified as not detected at the reported concentration.

SDG K0800325: One equipment blank was submitted, LW3-SAC908. After qualifiers were issued based on method blank contamination, positive results remained for 1,4-dichlorobenzene, benzyl alcohol, and naphthalene.

SDG K0800516: One equipment blank was submitted, LW3-SAC909. After qualifiers were issued based on method blank contamination, positive results remained for 1,4-dichlorobenzene, phenol, benzoic acid, diethyl phthalate, butyl benzyl phthalate, bis(2-ethylhexyl)phthalate, and naphthalene. Two 1,4-dichlorobenzene results, two benzoic acid results, one diethyl phthalate result, and three bis(2-ethylhexyl)phthalate results were qualified as not detected (U-6) at the reported concentrations.

Surrogate Compounds

The percent recovery (%R) values for the surrogates were within the specified control limits with the exceptions noted below. 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). No action was necessary if the %R value for only one surrogate per fraction (acid or base-neutral) was outside the control limits. All surrogate outliers are documented in the validation worksheets.

SDG K0711067: The 2,4,6-tribromophenol %R values were greater than the 110% upper control limit in Samples LW3-G747 and LW3-G772.

SDG K0711172: The 2,4,6-tribromophenol %R values were greater than the 110% upper control limit in eight samples.

SDG K0711174: The 2,4,6-tribromophenol %R values were greater than the 110% upper control limit in nine samples.

SDG K0711175: The 2,4,6-tribromophenol %R value was greater than the 110% upper control limit in Sample LW3-G642.

SDG K0711220: The 2,4,6-tribromophenol %R values were greater than the 110% upper control limit in Samples LW3-UG02F, LW3-G681, LW3-G682, and LW3-G691.

SDG K0711252: The %R value for 2,4,6-tribromophenol was greater than the upper control limit of 110% in Sample LW3-G651.

SDG K0711320: The %R value for 2,4,6-tribromophenol was greater than the upper control limit of 110% in Sample LW3-G654-2.

SDG K0711577: The %R value for 2,4,6-tribromophenol was greater than the upper control limit of 110% in Sample LW3-G783.

SDG K0711706: The %R values for 2,4,6-tribromophenol and terphenyl-d14 were greater than the upper control limits in Sample LW3-MC002-B.

SDG K0712106: The %R values for two acid-fraction surrogates (2,4,6-tribromophenol and 2-fluorophenol) were less than the lower control limits in Sample LW3-SAC904. All acid-fraction analytes were estimated (J/UJ-13) in this sample.

SDG K0800325: The %R values for 2,4,6-tribromophenol were below the lower control limit in Samples LW3-C783-B, LW3-C783-C, and LW3-C783-C due to dilution factors of 10x or more. No qualifiers were required for surrogate %R values due to dilutions (10x).

SDG K0800450: The %R values for several surrogates were outside control limits in several samples due to dilution factors of 10x or more. No qualifiers were required.

SDG K0800487: The %R values for several surrogates were outside control limits in Sample LW3-C708-C due to a dilution factor of 100x. The %R value for 2,4,6-tribromophenol was less than the lower control limit in Sample LW3-C703-C due to a dilution factor of 20x. No qualifiers were required for surrogate %R values due to dilutions.

SDG K0800516: The %R value for 2-fluorophenol was greater than the upper control limit in Sample LW3-C651-C. No qualifiers were required for this single acid outlier. The %R values for several surrogates were outside control limits in several samples due to dilution factors of 10x or more. No qualifiers were required for surrogate %R values due to dilutions (10x).

Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed at the required frequency. All MS/MSD recovery values were within the specified control limits, with the exceptions noted below. If the %R outlier was due to the presence of high levels of the target analyte present in the parent sample, no action was taken. If the concentration in the parent sample was less than four times the spike concentration, the results in the parent sample associated with the outlier were estimated (J-8) for a high bias, and results and reporting limits estimated (J/UJ-8) for a low bias. If the recovery value was less than 10%, detected results were estimated (J-8) and the reporting limits were rejected (R-8). A summary of outliers is provided in the data validation worksheets.

All MS/MSD relative percent difference (RPD) values were within the specified control limits, with the exceptions noted below. For RPD outliers, positive results for the affected compounds were estimated (J-9) in the parent sample.

SDG K0710859: MS/MSD analyses were performed using Sample LW3-UG03A-3.

- %R values < 10%: 2,4-dimethylphenol, 4-chloroaniline, 3,3'-dichlorobenzidine, aniline, and 3-nitroaniline
- Low bias - %R values < lower control limit (LCL) (but > 10%): N-nitrosodimethylamine and azobenzene
- RPD value outliers: 4-methylphenol and bis(2-ethylhexyl) phthalate

SDG K0710866: MS/MSD analyses were performed using Sample LW3-G785.

- %R values < 10%: 4-chloroaniline, 3,3'-dichlorobenzidine, aniline, and 3-nitroaniline
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine
- RPD value outliers: 4-methylphenol and bis(2-ethylhexyl) phthalate
- Single outlier (no action taken): 4-nitroaniline (low in MS only)

SDG K0710893: MS/MSD analyses were performed using Sample LW3-G674.

- %R values < 10%: N-nitrosodimethylamine, 3,3'-dichlorobenzidine, and aniline

SDG K0710893: MS/MSD analyses were performed using Sample LW3-UG02A.

- %R values < 10%: 4-chloroaniline, 3,3'-dichlorobenzidine, and aniline
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and 3-nitroaniline
- RPD value outliers: diethyl phthalate, carbazole, di-n-butyl phthalate, butyl benzyl phthalate, and bis(2-ethylhexyl) phthalate

SDG K0710893: MS/MSD analyses were performed using Sample LW3-SAG901

- No outliers

SDG K0711067: MS/MSD analyses were performed using Sample LW3-G670.

- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and aniline
- RPD value outliers: aniline, 2,4-dimethylphenol and 3,3'-dichlorobenzidine

- Single outlier (no action taken): bis(2-ethylhexyl) phthalate (high in MSD only)

SDG K0711172: MS/MSD analyses were performed using Sample LW3-G624.

- %R values < 10%: 3,3'-dichlorobenzidine and aniline
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and azobenzene

SDG K0711172: MS/MSD analyses were performed using Sample LW3-G614.

- %R values < 10%: 3,3'-dichlorobenzidine and aniline
- High bias - %R values > upper control limit (UCL): 2,4-dinitrophenol, 2-methyl-4,6-dinitrophenol, and di-n-octyl phthalate
- RPD value outliers: bis(2-ethylhexyl) phthalate and hexachlorocyclopentadiene
- Single outlier (no action taken): bis(2-ethylhexyl) phthalate (high in MS only) and N-nitrosodimethylamine (low in MSD only)

SDG K0711174: MS/MSD analyses were performed using Sample LW3-MG006.

- %R values < 10%: 3,3'-dichlorobenzidine and aniline
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine
- Single outlier (< 10%): 2,4-dimethylphenol %R < 10% in MSD, acceptable in MS. The 2,4-dimethylphenol reporting limit was estimated (UJ-8) rather than rejected.
- RPD value outliers: 2,4-dimethylphenol

Several %R and RPD values exceeded control limits in the batch QC MS/MSD. No qualifiers were applied as the parent sample was not from this SDG.

SDG K0711175: MS/MSD analyses were performed using Sample LW3-G650.

- %R values < 10%: 2,4-dimethylphenol and aniline
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine
- Single outlier (< 10%): 3,3'-dichlorobenzidine %R < 10% in MS, acceptable in MSD. The 3,3'-dichlorobenzidine reporting limit was estimated (UJ-8) rather than rejected.
- RPD value outliers: 3,3'-dichlorobenzidine
- Single outlier (no action taken): azobenzene (low in MS only)

SDG K0711220: MS/MSD analyses were performed using Sample LW3-G669.

- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine, aniline, and azobenzene
- Single outlier (< 10%): 3,3'-dichlorobenzidine %R < 10% in MS, acceptable in MSD. The 3,3'-dichlorobenzidine reporting limit was estimated (UJ-8) rather than rejected.

SDG K0711220: MS/MSD analyses were performed using Sample LW3-G681.

- %R values < 10%: 3,3'-dichlorobenzidine and aniline
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine
- High bias - %R values > UCL: 2,4-dinitrophenol and 2-methyl-4,6-dinitrophenol

- Single outlier (no action taken): azobenzene (low in MSD only)

SDG K0711252: MS/MSD analyses were performed using Sample LW3-G697.

- %R values < 10%: 3,3'-dichlorobenzidine, 4-chloroaniline, 3-nitroaniline, and aniline
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and azobenzene
- Single outlier (< 10%): 2,4-dimethylphenol %R < 10% in MSD, acceptable in MS. The 2,4-dimethylphenol reporting limit was estimated (UJ-8) rather than rejected.
- RPD value outliers: hexachlorocyclopentadiene, 2,4-dimethylphenol, 4-chloroaniline, and 3-nitroaniline

SDG K0711320 & K0711828: Several %R values exceeded control limits in the batch QC MS/MSD. No qualifiers were applied as the parent sample was not from this SDG. A complete list of these outliers is documented in the validation worksheets.

SDG K0711423: MS/MSD analyses were performed using Sample LW3-G717.

- %R values < 10%: 3,3'-dichlorobenzidine, 4-chloroaniline, 3-nitroaniline, and aniline
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and azobenzene
- Single outlier (< 10%): 4-nitroaniline %R < 10% in MSD, acceptable in MS. The 4-nitroaniline reporting limit was estimated (UJ-8) rather than rejected.
- RPD value outliers: benzoic acid, n-nitrosodimethylamine, 2,4-dinitrophenol, 4-nitrophenol, 4-chloroaniline, 2-methyl-4,6-dinitrophenol, 2,4-dinitrotoluene, 3-nitroaniline, 4-nitroaniline

SDG K0711577: MS/MSD analyses were performed using Sample LW3-G783.

- %R values < 10%: aniline, hexachlorocyclopentadiene, and 2,4-dinitrophenol
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and bis(2-ethyl hexyl) phthalate
- High bias - %R values > UCL: 1,4-dichlorobenzene
- Single outlier (< 10%): 3,3'-dichlorobenzidine %R < 10% in MSD, acceptable in MS. The 3,3'-dichlorobenzidine reporting limit was estimated (UJ-8) rather than rejected.

SDG K0711706: MS/MSD analyses were performed using Sample LW3-MC006-B.

- %R values < 10%: 3,3'-dichlorobenzidine and aniline
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and azobenzene
- Single outliers (< 10%): 4-chloroaniline, 2,4-dinitrophenol, 2-methyl-4,6-dinitrophenol, and benzoic acid %R < 10% in MS, acceptable in MSD. The reporting limits were estimated (UJ-8) rather than rejected.
- RPD value outliers: benzoic acid, n-nitrosodimethylamine, 2,4-dinitrophenol, 4-nitrophenol, 4-chloroaniline, 2-methyl-4,6-dinitrophenol, 2,4-dinitrotoluene, 3-nitroaniline, aniline, diethyl phthalate, 3,3'-dichlorobenzidine, 2,4-dimethylphenol, hexachlorocyclopentadiene
- Single outlier (no action taken): 2,4-dinitrotoluene (low in MS only)

SDG K0711706: MS/MSD analyses were performed using Sample LW3-C604-B.

- %R values < 10%: aniline, 4-chloroaniline, 3-nitroaniline, and 3,3'-dichlorobenzidine

- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and azobenzene
- RPD value outliers: 4-chloroaniline and 2,4-dimethylphenol

SDG K0711709: MS/MSD analyses were performed using Sample LW3-C600-D.

- %R values < 10%: 2,4-dimethylphenol
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine
- Single outliers (< 10%): aniline and 3,3'-dichlorobenzidine %R < 10% in MS, acceptable in MSD. The reporting limits were estimated (UJ-8) rather than rejected.
- RPD value outliers: benzoic acid, 2,4-dinitrophenol, and 2-methyl-4,6-dinitrophenol
- Single outlier (no action taken): azobenzene (low in MSD only)

SDG K0711830: MS/MSD analyses were performed using Sample LW3-C615-B.

- %R values < 10%: aniline, hexachlorocyclopentadiene, and 3,3'-dichlorobenzidine
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine
- Single outliers (< 10%): 4-chloroaniline and 2,4-dinitrophenol %R < 10% in MS, acceptable in MSD. The reporting limits were estimated (UJ-8) rather than rejected.
- RPD value outliers: hexachlorocyclopentadiene, 2,4-dimethylphenol, 4-nitrophenol, and 2,4-dinitrophenol
- Single outlier (no action taken): azobenzene (low in MSD only)

SDG K0711830: MS/MSD analyses were performed using Sample LW3-C634-B.

- %R values < 10%: aniline, hexachlorocyclopentadiene, and 3,3'-dichlorobenzidine
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine
- Single outliers (< 10%): 4-chloroaniline, 2-methyl-4,6-dinitrophenol, 2,4-dinitrophenol, and 2,4-dimethylphenol %R < 10% in either MS or MSD, acceptable in other analysis. The reporting limits were estimated (UJ-8) rather than rejected.
- RPD value outliers: 2-methylphenol, 4-methylphenol, 2,4-dimethylphenol, benzoic acid, 4-chloroaniline, 2,4-dinitrophenol, 4-nitrophenol, 2,4-dinitrotoluene, 2-methyl-4,6-dinitrophenol, and n-nitrosodimethylamine
- Single outlier (no action taken): azobenzene (low in MSD only)

SDG K0712050: MS/MSD analyses were performed using Sample LW3-C704-B.

- %R values < 10%: aniline and 3,3'-dichlorobenzidine
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and azobenzene
- Single outliers (< 10%): 4-chloroaniline %R < 10% in MSD, acceptable in MS. The reporting limits were estimated (UJ-8) rather than rejected.
- High bias - %R values > UCL: 4-nitrophenol
- RPD value outliers: 4-nitroaniline and 3-nitroaniline

SDG K0712101: MS/MSD analyses were not performed. Precision and accuracy were assessed using the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses.

SDG K0712106: MS/MSD analyses were performed using Sample LW3-C714-B.

- %R values < 10%: aniline, 4-chloroaniline, 3-nitroaniline, and 3,3'-dichlorobenzidine
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and azobenzene
- Single outliers (< 10%): 2,4-dimethylphenol %R < 10% in MSD, acceptable in MS. The reporting limit was estimated (UJ-8) rather than rejected.
- High bias - %R values > UCL: bis(2-ethylhexyl) phthalate
- RPD value outliers: n-nitrosodimethylamine, 4-chloroaniline, 4-nitroaniline, benzoic acid, and 2-methyl-4,6-dinitrophenol

SDG K0712149: MS/MSD analyses were performed using Sample LW3-C749-C.

- %R values < 10%: 2,4-dimethylphenol and 3,3'-dichlorobenzidine
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine and azobenzene
- Single outliers (< 10%): 4-chloroaniline %R < 10% in MS, acceptable in MSD. The reporting limit was estimated (UJ-8) rather than rejected.
- RPD value outliers: 2-methylphenol, 4-methylphenol, and 2-methyl-4,6-dinitrophenol

SDG K0800158: MS/MSD analyses were performed using Sample LW3-C760-C.

- %R values < 10%: 4-chloroaniline, 3-nitroaniline, 4-nitroaniline, 3,3'-dichlorobenzidine, benzoic acid, and bis (2-ethylhexyl) phthalate
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine, aniline, and azobenzene

SDG K0800158: MS/MSD analyses were performed using batch QC. The %R and RPD values for several compounds were outside of the control limits in the batch MS/MSD set. No qualifiers were required as the parent sample was from a different analytical group.

SDG K0800270: MS/MSD analyses were performed using Sample LW3-C757-C.

- %R values < 10%: 3,3'-dichlorobenzidine, hexachlorocyclopentadiene and 1,4-dichlorobenzene
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine, aniline, and azobenzene
- RPD value outliers: butyl benzyl phthalate and bis(2-ethylhexyl) phthalate

SDG K0800325: MS/MSD analyses were performed using Sample LW3-C748-D2.

- %R values < 10%: aniline and 2,4-dimethylphenol
- Low bias - %R values < LCL (but > 10%): azobenzene
- RPD value outliers: n-nitrosodimethylamine, n-nitrosodi-n-propylamine, and benzoic acid

SDG K0800325: MS/MSD analyses were performed using batch QC. The %R and RPD values for several compounds were outside of the control limits in the batch MS/MSD set. No qualifiers were required as the parent sample was from a different analytical group.

SDG K0800450: MS/MSD analyses were performed using Sample LW3-C645-C.

- %R values < 10%: benzoic acid
- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine, aniline, and azobenzene

SDG K0800450: MS/MSD analyses were performed using batch QC. The %R and RPD values for several compounds were outside of the control limits in the batch MS/MSD set. No qualifiers were required as the parent sample was from a different analytical group.

SDG K0800487: MS/MSD analyses were performed using Sample LW3-C706-C.

- Low bias - %R values < LCL (but > 10%): N-nitrosodimethylamine, aniline, and azobenzene

SDG K0800487: MS/MSD analyses were performed using batch QC. The %R values for several compounds were outside of the control limits in the batch MS/MSD set. No qualifiers were required as the parent sample was from a different analytical group.

SDG K0800516: MS/MSD analyses were performed using Sample LW3-C738-B.

- %R values < 10%: 3,3'-dichlorobenzidine
- Low bias - %R values < LCL (but > 10%): aniline and 3-nitroaniline
- RPD value outliers: aniline, 3-nitroaniline, and 4-nitroaniline

MS/MSD analyses were also performed using Sample LW3-C662-D. No spike recoveries were obtained as the sample required a dilution (factor 20x) which diluted out the spiked analytes. The RPD values for 2,4-dimethylphenol, carbazole, and di-n-octyl phthalate exceeded control limits; the result for carbazole was qualified as estimated (J-9) in the parent sample, the other analytes were not detected and required no qualifiers.

Laboratory Control Sample/Laboratory Control Sample Duplicates

SDG K0710859: The %R values for 2,4-dimethylphenol in the LCS and LCSD were less than 10%, at 2% and 2%. This compound was not detected in the associated samples, the reporting limits were rejected (R-10). The RPD for benzoic acid exceeded the control limit of 40%, at 75%; all positive results for benzoic acid were qualified as estimated (J-9).

SDG K0710866: The RPD value for 2,4-dimethylphenol was greater than the control limit. This analyte was not detected in the associated samples and no qualifiers were applied. The RPD value for aniline was greater than the control limit. All positive results for aniline were qualified as estimated (J-9) in the associated samples.

SDG K0710893 (aqueous): The %R values for 2,4-dimethylphenol were less than the lower control limit of 10%, at 2% and 5% in the aqueous LCS/LCSD set. This analyte was not detected in the associated field blanks and the reporting limits were rejected (R-10). The RPD value for 4-chloroaniline was greater than the 30% control limit, at 39%. This analyte was not detected in the associated field blanks and no action was taken. The LCSD %R value and the RPD value for 3,3'-dichlorobenzidine were outside of control limits. No qualifiers were applied as the LCS %R value was within control limits.

SDG K0710893 (sediment): Two sediment LCS/LCSD sets were prepared and analyzed. For LCS/LCSD (batch KWG0712899), the LCSD %R and RPD value for 2,4-dimethylphenol were outside of control limits. No action was required as the LCS %R value was within control limits. The RPD values for benzoic acid and 3,3'-dichlorobenzidine were greater than control limits. Positive results for benzoic acid were estimated (J-9); 3,3'-dichlorobenzidine was not detected. For LCS/LCSD (batch KWG0712900), the LCS %R value for benzoic acid was less than the lower control limit. As the LCSD %R value was in control; no qualifiers were applied.

SDG K0711220 (aqueous): The %R values for hexachlorocyclopentadiene were less than 10% in the aqueous LCS/LCSD set. The reporting limit for this analyte was rejected (R-10) in the associated field blank.

SDG K0711220 (sediment): Two sediment LCS/LCSD sets were prepared and analyzed. The benzoic acid RPD value was greater than the control limit in the first sediment LCS/LCSD. Positive results for this analyte were qualified as estimated (J-9) in the associated samples. The 2,4-dimethylphenol %R value in the LCSD was less than 10% in the first sediment LCS/LCSD set, the LCS %R value was acceptable. The reporting limits were estimated (UJ-10) in the associated samples. The 2,4-dimethylphenol %R values were less than 10% in the second sediment LCS/LCSD set. The reporting limits for this analyte were rejected (R-10) in the associated samples.

SDGs K0711174, K0711175, K0711252 (aqueous): The hexachlorocyclopentadiene %R values were less than 10%; the reporting limits for this analyte were rejected (R-10) in the associated samples. The %R values for di-n-butyl phthalate were greater than the upper control limit; this analyte was not detected in the associated samples, no qualifiers were applied. The RPD values for hexachlorocyclopentadiene were outside control limits; this analyte was not detected in associated samples, no qualifiers were applied.

SDGs K0711174, K0711175, K0711252 (sediment): The RPD value for 2,4-dimethylphenol in the sediment LCS/LCSD submitted with SDG K0711252 was outside control limits; this analyte was not detected in any associated samples, no qualifiers were applied.

SDG K0711320: The %R values for 2,4-dimethylphenol in the sediment LCS/LCSD were less than 10%. The reporting limits for this analyte were rejected (R-10) in the associated samples.

SDG K0711423 (aqueous): The RPD values for n-nitrosodimethylamine, 2,4-dimethylphenol, and hexachlorobutadiene were outside control limits. These compounds were not detected in the associated samples, no qualifiers were applied.

SDG K0711423 (sediment): In the first sediment LCS/LCSD set, the 2-methylphenol, 4-methylphenol, and 2,4-dimethylphenol RPD values were outside control limits; positive values for 4-methylphenol were estimated (J-9) in the associated samples, the other compounds were not detected in the associated samples. The LCSD %R value for the 2,4-dimethylphenol was less than 10%, the LCS %R value was within control limits; reporting limits for this compound were estimated (UJ-10) in the associated samples.

In the second sediment LCS/LCSD set, the bis(2-ethylhexyl) phthalate, 4-methylphenol, 3-nitroaniline, and 4-nitroaniline RPD values were outside control limits; positive values for

bis(2-ethylhexyl) phthalate and 4-methylphenol were estimated (J-9) in the associated samples, the other compounds were not detected in the associated samples. The 2,4-dimethylphenol %R value was less than 10% in the LCSD, the LCS %R value was within control limits; reporting limits were estimated (UJ-10) in the associated samples. The analyte 3,3'-dichlorobenzidine was not recovered in the LCSD, the LCS %R value was within control limits; reporting limits were estimated (UJ-10) in the associated samples. The LCS %R value for bis(2-ethylhexyl) phthalate was much greater than the upper control limit, the LCSD %R value was within control limits; positive results were estimated (J-10) in the associated samples. The LCS %R value for 4-nitroaniline was less than the lower control limit, the LCSD %R value was within control limits; no qualifiers were applied for a single outlier.

SDG K0711577: The RPD values for 2-methylphenol, 4-methylphenol, and 2,4-dimethylphenol in the LCS/LCSD were outside control limits. The positive results for 4-methylphenol were estimated (J-9) in the associated samples. The other analytes were not detected so no qualifiers were applied. The MSD %R value for 2,4-dimethylphenol was less than 10%, the MS %R value was within control limits, the reporting limit for this analyte was estimated (UJ-10).

SDG K0711706 (aqueous): The LCS or LCSD %R values and RPD values for 2-methylphenol and 4-methylphenol were outside control limits. These compounds were not detected in the associated samples, no qualifiers were applied. The 2,4-dimethylphenol %R values were less than 10%; reporting limits in the associated samples were rejected (R-10).

SDG K0711706 (sediment): The 2,4-dimethylphenol %R values were less than 10% and the RPD value was outside control limits, reporting limits in the associated samples were rejected (R-10).

SDG K0711709: The LCS %R value for 2,4-dimethylphenol was less than 10%, the LCSD %R value was within control limits, the reporting limit for this analyte was estimated (UJ-10).

SDG K0711828 (aqueous): The LCSD %R values and RPD values for 2-methylphenol and 4-methylphenol were greater than control limits. These compounds were not detected in the associated samples, no qualifiers were applied. The %R values for 2,4-dimethylphenol were less than 10%, reporting limits in the associated samples were rejected (R-10).

SDG K0711828 (sediment): The RPD values for 4-methylphenol, carbazole, di-n-butyl phthalate, butyl benzyl phthalate, and bis(2-ethylhexyl) phthalate were outside control limits; positive values for these analytes were estimated (J-9) in the associated samples. The RPD values for 2-methylphenol, 3,3'-dichlorobenzidine, di-n-octyl phthalate, hexachlorobenzene, and 4-bromophenyl phenyl ether were outside control limits; these other compounds were not detected in the associated samples, no qualifiers were applied. The LCS %R values for the hexachlorobenzene and 4-bromophenyl phenyl ether were less than the lower control limit, the LCSD %R value was within control limits, no qualifiers were applied. The %R values for 2,4-dimethylphenol were less than 10%, reporting limits in the associated samples were rejected (R-10).

SDG K0711830: The %R values for 2,4-dimethylphenol in the LCS/LCSD set were less than 10%; reporting limits in the associated samples were rejected (R-10). The LCS %R value for hexachlorocyclopentadiene and the LCSD %R value for benzoic acid were less than 10%, their

LCS or LCSD %R values were within control limits, reporting limits in the associated samples were estimated (UJ-10). The 2,4-dimethylphenol and hexachlorocyclopentadiene RPD values exceeded control limits; there were no positive results detected for these analytes; no further qualifiers were required.

SDG K0712050 (aqueous): The LCSD %R value and the RPD value for 2-methylphenol were outside control limits; this compound was not detected in the associated sample, no qualifiers were applied. The %R values and RPD value for 4-methylphenol were outside control limits; this compound was not detected in the associated sample, the reporting limit was estimated (UJ-10). The %R values for 2,4-dimethylphenol were less than 10%, the reporting limit in the associated sample was rejected (R-10).

SDG K0712050 (sediment): The %R values for 2,4-dimethylphenol were less than 10%, reporting limits in the associated samples were rejected (R-10).

SDG K0712101: The %R values for benzoic acid in the LCS/LCSD set were less than 10%; reporting limits in the associated samples were rejected (R-10).

SDG K0712106 (aqueous): The LCSD %R value and the RPD value for 2-methylphenol were outside control limits; this compound was not detected in the associated sample, no qualifiers were applied. The %R values and RPD value for 4-methylphenol were outside control limits; this compound was not detected in the associated sample, the reporting limit was estimated (UJ-10). The %R values for 2,4-dimethylphenol were less than 10%, the reporting limit in the associated sample was rejected (R-10).

SDG K0712106 (sediment): The %R values for aniline and 2,4-dimethylphenol were less than 10%, reporting limits in the associated samples were rejected (R-10). The LCSD %R value for benzoic acid were less than 10%, the LCS %R value was within control limits, reporting limits in the associated samples were estimated (UJ-10). The RPD values for 2,4-dimethylphenol and hexachlorocyclopentadiene exceeded control limits; there were no positive results detected for these analytes; no further qualifiers were required.

SDG K0712149: The %R value for benzoic acid was less than 10%, positive results were estimated (J-10) and reporting limits were rejected (R-10) in the associated samples.

SDG K0800158 (aqueous): The %R values for di-n-butyl phthalate exceeded control limits; this compound was not detected in the associated sample, no qualifiers were applied.

SDG K0800158 (sediment): In both sediment LCS/LCSD sets, the %R values for aniline and 2,4-dimethylphenol were less than 10%. The reporting limits for these analytes were rejected (R-10) in the associated samples.

SDG K0800270 (aqueous): The %R values for hexachlorocyclopentadiene were less than 10% in the aqueous LCS/LCSD set. The reporting limit for this analyte was rejected (R-10) in the associated field blank. The RPD value for 3,3'-dichlorobenzidine exceeded the upper control limit. This analyte was not detected in the associated field blank and no qualifier was required.

SDG K0800270 (sediment): Two sediment LCS/LCSD sets were prepared and analyzed. The 2,4-dimethylphenol %R values were less than 10% in the both sediment LCS/LCSD sets. The reporting limits for this analyte were rejected (R-10) in the associated samples. The aniline %R values were less than 10% in the sediment LCS/LCSD set for batch KWG0800462. The reporting limits for this analyte were rejected (R-10) in the associated samples.

SDG K0800325 (aqueous): The %R values for hexachlorobutadiene were less than 10%, the reporting limit in the associated rinsate blank was rejected (R-10). The 3,3'-dichlorobenzidine RPD value exceeded control limits; there were no positive results detected for this analyte; no further qualifiers were required.

SDG K0800325 (sediment): The %R values for benzoic acid in one sediment LCS/LCSD set and the 2,4-dimethylphenol %R values in another LCS/LCSD set were less than 10%, reporting limits in the associated samples were rejected (R-10). The RPD value for 2,4-dimethylphenol in the second LCS/LCSD set also exceeded control limits; there were no positive results detected for this analyte; no further qualifiers were required.

SDG K0800450: The %R values for benzoic acid were less than 10% in the LCS/LCSD set. The benzoic acid reporting limit was rejected (R-10) in the associated samples.

SDG K0800487: The %R values for benzoic acid were less than 10% in the LCS/LCSD set. The reporting limit for this analyte was rejected (R-10) in the associated samples.

SDG K0800516 (aqueous): The 1,3-dichlorobenzene, 1,2-dichlorobenzene, 2-methylphenol, hexachloroethane, hexachlorobutadiene, 4-chloro-3-methylphenol, 4-methylphenol, 2,4-dimethylphenol, 1,2,4-trichlorobenzene, and 3,3'-dichlorobenzidine RPD values exceeded control limits; there were no positive results detected for these analytes; no qualifiers were required.

SDG K0800516 (sediment): In the sediment LCS/LCSD associated with extraction batch KWG0800689 the %R values for benzoic acid were less than 10%, results and reporting limits in the associated samples were rejected (J/R-10). In the sediment LCS/LCSD associated with extraction batch KWG0800691 the %R values for aniline were less than 10%, reporting limits in the associated samples were rejected (R-10).

Field Replicates

Two types of field replicates were submitted, field replicates (co-located samples) and field splits. Replicate sample sets are listed below. Field splits were compared to the parent sample results. Field replicates were compared to the average of the field split and parent sample result.

The following acceptance criteria were used to evaluate precision: the RPD control limit is 50% for results greater than five times the reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and replicate must be less than two times the RL. No data were qualified based on field replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

SDG K0710859: Samples LW3-UG03A-2 (field replicate) and LW3-UG03A-3 (field split) were submitted with parent Sample LW3-UG03A-1. Samples LW3-G637-2 (field replicate) and LW3-G637-3 (field split) were submitted with parent Sample LW3-G637-1. All RPD values and/or absolute differences met the acceptance criteria.

SDG K0710866: The data for one set of field replicates was submitted: Samples LW3-G612-1 and LW3-G612-2. All absolute difference values were within control limits.

SDG K0711067: Samples LW3-G684-2 (field replicate) and LW3-G684-3 (field split) were submitted with parent Sample LW3-G684-1. For the comparison of the field split to the parent sample, the bis(2-ethylhexyl)phthalate RPD value and the benzyl alcohol absolute difference value were greater than control limits. For the field replicate, all RPD and/or absolute difference values were within control limits.

SDG K0711172: One pair of samples, LW3-G627-1 & LW3-627-2 were submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711174: Samples LW3-M002-2 (field replicate) and LW3-M002-3 (field split) were submitted with parent Sample LW3-M002-1. The RPD and/or absolute difference values were within control limits.

SDG K0711174 & K0711320: One pair of samples, LW3-G641-1 (submitted in SDG K0711174) & LW3-G641-2 (in K0711320) was submitted as field replicates. The carbazole RPD value was greater than the control limit.

SDG K0711175: One pair of samples, LW3-G659-1 & LW3-G659-2, was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711220: One pair of samples, LW3-UG02E-1 & LW3-UG02E-2, was submitted as field replicates. The absolute difference values were within control limits.

SDG K0711252: One pair of samples, LW3-G732-1 & LW3-G732-2 was submitted as field replicates. The bis(2-ethylhexyl) phthalate RPD value was greater than the control limit.

SDG K0711423: Samples LW3-G692-2 (field replicate) and LW3-G692-3 (field split) were submitted with parent Sample LW3-G692-1. For the comparison of the field split to the parent sample, the di-n-butyl phthalate RPD value was greater than the control limit. For the field replicate, all RPD and/or absolute difference values were within control limits.

A second field replicate set (LW3-G759-1 & LW3-G759-2) was also submitted. The RPD value for di-n-butyl phthalate was greater than the control limit.

SDG K0711706: The data for two field replicate sets (LW3-MC001-B1 & LW3-MC001-B2 and LW3-MC001-C1 & LW3-MC001-C2) were submitted. All RPD and/or absolute difference values were within control limits.

SDG K0711709: Samples LW3-C601-B2 (field replicate) and LW3-C601-B3 (field split) were submitted with parent Sample LW3-C601-B1. The RPD and/or absolute difference values were within control limits.

SDG K0711828 & K0711830: The data for two sets of field precision samples were submitted. Samples LW3-C644-B2 (field replicate) and LW3-C644-B3 (field split) were submitted with parent Sample LW3-C644-B1, and Samples LW3-C644-C2 (field replicate) and LW3-C644-C3 (field split) were submitted with parent Sample LW3-C644-C1. All RPD and/or absolute difference values were within control limits.

SDG K0800158: The data for three sets of field precision samples were submitted. Samples LW3-C773-B2 (field replicate) and LW3-C773-B3 (field split) were submitted with parent Sample LW3-C773-B1, Samples LW3-C773-C2 (field replicate) and LW3-C773-C3 (field split) were submitted with parent Sample LW3-C773-C1, and LW3-C773-D2 (field replicate) and LW3-C773-D3 (field split) were submitted with parent Sample LW3-C773-D1. The RPD value for 4-methylphenol was greater than the control limit for the comparison of LW3-C773-D1 to LW3-C773-D3. All other RPD and/or absolute difference values were within control limits.

SDG K0800270 & K0800325: The following field replicates were submitted (all parent samples were reported in SDG K0800270, all field replicates (sample ID ends in 2) and splits (ends in 3) were reported in SDG K0800325):

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C748-B1	LW3-C748-B3	LW3-C748-B2
LW3-C748-C1	LW3-C748-C3	LW3-C748-C2
LW3-C748-D1	LW3-C748-D3	LW3-C748-D2
LW3-C764-B1		LW3-C764-B2
LW3-C764-C1		LW3-C764-C2
LW3-C764-D1		LW3-C764-D2

The following outliers were noted:

Replicates	RPD > 50%	Diff > 2x RL
LW3-C748-B1 and LW3-C748-B3	Bis(2-ethylhexyl)phthalate	
LW3-C748-C1/C3 and LW3-C748-C2	Bis(2-ethylhexyl)phthalate	Carbazole
LW3-C748-C1 and LW3-C748-C3		Benzyl alcohol
LW3-C764-C1 and LW3-C764-C2	Bis(2-ethylhexyl)phthalate	
LW3-C764-D1 and LW3-C764-D2	Benzoic acid	Carbazole, diethyl phthalate, n-nitrosodiphenylamine

Target Analyte List

Dibenzofuran was reported from a separate analysis (EPA 8270C-SIM) with the PAH compounds.

Compound Identification

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

The method detection limits (MDL) for several compounds were greater than the QAPP MDL. No action was taken since the QAPP method reporting limits (MRL) were met, except when several samples were diluted due to matrix interference.

SDGs K0711174, K0711423: Several samples were diluted, or a smaller sample aliquot was extracted, due to matrix interference. Reporting limits were elevated accordingly.

SDG K0711320: Sample LW3-G641-2 was diluted (10x) due to matrix interference. Reporting limits were elevated accordingly.

SDG K0712101: Sample LW3-G613-E was diluted (10x) due to matrix interference. Reporting limits were elevated accordingly.

Calculation Verification

SDGs K0711175, K0711709, K0712101, K0800325 & K0800516: Calculation verifications were performed on these SDG. No calculation errors were found.

IV. OVERALL ASSESSMENT

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

Data were qualified as estimated because of surrogate, MS/MSD and LCS/LCSD recovery outliers, precision outliers, and calibration %D outliers. Data were also qualified as not detected based on contamination in the associated laboratory and field blanks.

Data were rejected because of very low LCS/LCSD and MS/MSD recovery outliers. Data that has been rejected should not be used for any purpose.

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

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3B Sediments
Polycyclic Aromatic Hydrocarbons (PAH) and Alkylated PAH by
Method SW8270-SIM

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

SDG	Number of Samples	Validation Level
K0710859	20 Sediment	Summary
K0710866	12 Sediment	Summary
K0710893	24 Sediment	Summary
K0711067	19 Sediment	Summary
K0711172	20 Sediment	Summary
K0711174	21 Sediment	Summary
K0711175	12 Sediment	Full
K0711220	23 Sediment	Summary
K0711252	19 Sediment	Summary
K0711320	4 Sediment	Summary
K0711423	27 Sediment	Summary
K0711577	4 Sediment	Summary
K0711706	19 Sediment	Summary
K0711709	10 Sediment	Full
K0711828	13 Sediment	Summary
K0711830	20 Sediment	Summary
K0712050	15 Sediment	Summary
K0712101	9 Sediment	Full
K0712106	9 Sediment	Summary
K0712149	6 Sediment	Summary
K0800158	28 Sediment	Summary
K0800270	21 Sediment	Summary
K0800325	13 Sediment	Full
K0800450	27 Sediment	Summary
K0800487	22 Sediment	Summary
K0800516	14 Sediment	Full

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and 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 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	1	Laboratory Control Samples (LCS/LCSD)
	GC/MS Instrument Performance Check	1	Field Replicates
	Initial Calibration (ICAL)	1	Internal Standards
1	Continuing Calibration (CCAL)		Target Analyte List
2	Laboratory Blanks	1	Reporting Limits
1	Field Blanks	2	Compound Identification
1	Surrogate Compounds	2	Reported Results
2	Matrix Spikes/Matrix Spike Duplicates (MS/MSD)	1	Calculation Verification (Full validation only)

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

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

Holding Times and Sample Receipt

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of 2° to 6°C. The laboratory received many of the sample coolers with temperatures outside the advisory control limits. The temperature outliers ranged from -0.8° to 7.9°C. These temperature outliers did not impact data quality and no qualifiers were required.

Continuing Calibration

All relative response factor (RRF) values were greater than the 0.05 minimum control limit. All percent difference (%D) values were within the ±25% control limit for all continuing calibrations (CCAL), with the exceptions noted below. When the %D outlier indicates a potential high bias, and there were no positive results for these compounds, no qualifiers were required.

SDG K0711175: CCAL 1/11/08: dibenz(a,h)anthracene, benzo(g,h,i)perylene (high bias)

Laboratory Blanks

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

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

SDG K0710859: Naphthalene (7 results)

SDG K0710893: Naphthalene (1 result) and 2-methylnaphthalene (1 result)

SDG K0711220: Naphthalene (6 results), acenaphthene (12 results), dibenzothiophene (10 results), phenanthrene (13 results), anthracene (11 results), pyrene (1 result)

SDG K0711423: Benzo(a)anthracene (1 result), benzo(b)fluoranthene (1 result), chrysene (1 result), benzo(k)fluoranthene (1 result), benzo(a)pyrene (1 result), dibenzo(a,h)anthracene (3 results)

SDG K0711577: Dibenzo(a,h)anthracene (2 results)

SDG K0711828: Naphthalene (5 results), chrysene (1 result), indeno(1,2,3-cd)pyrene (4 results), fluoranthene (1 result), pyrene (2 results), C2-fluorenes (1 result), C1-phenanthrenes/anthracenes (2 results)

SDG K0711830: Naphthalene (6 results), benzo(a)pyrene (1 result), benzo(b)fluoranthene (2 results), benzo(k)fluoranthene (1 result), chrysene (2 result), indeno(1,2,3-cd)pyrene (2 results), pyrene (2 results)

SDG K0712050: Naphthalene (1 result)

SDG K0712101: Benzo(b)fluoranthene (2 results), chrysene (1 result), fluoranthene (1 result), indeno(1,2,3-cd)pyrene (1 result), pyrene (1 result)

SDG K0712149: Naphthalene (3 results), indeno(1,2,3-cd)pyrene (3 results)

SDG K0800158: Naphthalene (5 results)

SDG K0800270: Naphthalene (12 results) and indeno(1,2,3-cd)pyrene (3 results)

SDG K0800450: Naphthalene (7 results), indeno(1,2,3-cd)pyrene (2 results), fluoranthene (1 result), pyrene (1 result), chrysene (1 result), benzo(b)fluoranthene (1 result), benzo(a)pyrene (1 result), indeno(1,2,3-cd)pyrene (1 result)

SDG K0800516: Dibenzo(a,h)anthracene (3 results)

Field Blanks

With the exception of equipment rinsate blank LW3-SAG901, the PAH fraction of the rinsate blanks were analyzed using the standard SVOC methodology, SW8270C. All rinsate blanks for both SVOC and PAH analytes are discussed in the SVOC report.

Surrogate Compounds

SDG K0712101: The spike concentrations for the surrogates were listed in the raw data as 200 ng/mL; however the percent recovery (%R) values in the summary sheets are calculated using a true value of 500 ng/mL. The EDD also confirms that 500 ng/mL was the correct true value, and this is also the mid-point of the curve. No qualifiers were required.

SDG K0800516: The %R values for several surrogates were outside control limits in several samples due to dilution factors of 10x or more. No qualifiers were required for surrogate %R values due to dilutions (10x).

The spike concentrations for the surrogates were listed in the raw data as 200 ng/mL; however the %R values in the summary sheets are calculated using a true value of 500 ng/mL. The EDD also confirms that 500 ng/mL was the correct true value, and this is also the mid-point of the curve. No qualifiers were required.

Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed at the required frequency. All MS/MSD %R values were within the specified control limits, with the exceptions noted below. If the %R outlier was due to the presence of high levels of the target analyte present in the parent sample, no action was taken. If the concentration in the parent sample was less than four times the spike concentration, the results associated with the outlier were estimated (J-8) in the parent sample. If the recovery value was less than 10%, the reporting limits were rejected (R-8). A summary of outliers is provided in the data validation worksheets.

All MS/MSD relative percent difference (RPD) values were within the specified control limits, with the exceptions noted below. For RPD outliers, positive results for the affected compounds were estimated (J-9) in the parent sample.

SDG K0710893: MS/MSD analyses were performed using Sample LW3-G674. The MS %R values for fluoranthene and pyrene were greater than the upper control limit, but the MSD %R values for these analytes were within control limits. No action was required. The RPD values for fluoranthene, pyrene, and benzo(a)anthracene were greater than the control limits. The results for these analytes were qualified as estimated (J-9) in the parent sample.

SDG K0711175: MS/MSD analyses were performed using Sample LW3-G650. The %R values for fluoranthene and pyrene were greater than upper control limits; results for these analytes were estimated (J-8) in the parent sample. The RPD value for chrysene was outside control limits; the result for this analyte was estimated in the parent sample. The %R value for either the MS or MSD was greater than upper control limits for benzo(a)anthracene, chrysene, benzo(b)fluoranthene, and benzo(a)pyrene; no qualifiers were applied for these single outliers.

SDG K0711423: Several %R and RPD values exceeded control limits in the batch QC MS/MSD. No qualifiers were applied as the parent sample was not from this SDG.

SDG K0711577: MS/MSD analyses were performed using Sample LW3-G783. The %R values for acenaphthylene, fluorene, dibenzothiophene, dibenz(a,h)anthracene, benzo(k)fluoranthene, and anthracene were less than lower control limits; results for these analytes were estimated (J-8) in the parent sample. The RPD values for fluoranthene and pyrene were outside control limits; the results for these analytes were estimated (J-9) in the parent sample.

The %R values for phenanthrene, benzo(a)pyrene, benzo(a)anthracene, benzo(g,h,i)perylene, chrysene, indeno(1,2,3-cd)pyrene, and benzo(b)fluoranthene were also less than lower control limits; however, the parent sample concentration was greater than four times the spike concentration and no qualifiers were applied.

SDG K0711709: MS/MSD analyses were performed using Sample LW3-C600-D. The RPD values for 17 of 20 analytes were outside control limits; the positive results for these analytes were estimated in the parent sample. A complete list of the outliers is documented in the validation worksheets.

SDG K0711830: MS/MSD analyses were performed using Sample LW3-C615-B. The %R values for phenanthrene were less than 10%, the positive result was estimated (J-8) in the parent sample. The %R values were less than 10% for indeno(1,2,3-cd)pyrene and fluoranthene in either the MS or MSD analysis, while the other %R value was within control limits; the positive results for these analytes were estimated (J-8) in the parent sample.

MS/MSD analyses were also performed using Sample LW3-C634-B. The MSD %R value for naphthalene was greater than the upper control limit and the RPD value was outside control limits. The positive result for naphthalene was estimated (J-9) in the parent sample. The %R value for phenanthrene were outside control limits but the spike concentration was less than four times the concentration of the parent sample; no qualifiers were applied.

SDG K0712101: Due to limited sample volume an laboratory control sample/laboratory control sample duplicate (LCS/LCSD) was analyzed instead.

SDG K0712106: MS/MSD analyses were performed using Sample LW3-C714-B. The RPD values for nine analytes were outside control limits. These analytes were estimated (J-9) in the parent sample. A complete list of outliers is documented in the validation worksheets.

SDG K0800325: MS/MSD analyses were performed using Sample LW3-C748-D2. The RPD values for phenanthrene, fluoranthene, and pyrene were outside control limits. These analytes were estimated (J-9) in the parent sample.

SDG K0800487: Several %R and RPD values exceeded control limits in the batch QC MS/MSD. No qualifiers were applied as the parent sample was not from this SDG.

SDG K0800516: MS/MSD analyses were performed using Sample LW3-C738-B. The %R values for phenanthrene, fluoranthene, and pyrene were outside control limits but the spike concentrations were less than four times the concentrations of the parent sample; no qualifiers were applied.

MS/MSD analyses were also performed using Sample LW3-C662-D. The %R values for all analytes were outside control limits but the spike concentrations were less than four times the concentrations of the parent sample; no qualifiers were applied. The RPD values for acenaphthylene and dibenzothiophene exceeded control limits; results for the affected compounds were estimated (J-9) in the parent sample.

Laboratory Control Sample/Laboratory Control Sample Duplicate

SDG K0711220: The LCS %R value and RPD value for dibenzothiophene were outside of control limits. No action was required as the LCSD %R value was within control limits.

SDG K0711828: The LCSD %R values for 15 of 20 were outside of control limits. No action was required as the LCS %R values were within control limits. A complete list of the outliers is documented in the validation worksheets.

Field Replicates

Two types of field replicates were submitted, field replicates (co-located samples) and field splits. Replicate sample sets are listed below. Field splits were compared to the parent sample results. Field replicates were compared to the average of the field split and parent sample result.

The following acceptance criteria were used to evaluate precision: the RPD control limit is 50% for results greater than five times the reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and replicate must be less than two times the RL. No data were qualified based on field replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

SDG K0710859: Samples LW3-UG03A-2 (field replicate) and LW3-UG03A-3 (field split) were submitted with parent Sample LW3-UG03A-1. All RPD values and/or absolute differences met the acceptance criteria.

SDG K0710859: Samples LW3-G637-2 (field replicate) and LW3-G637-3 (field split) were submitted with parent Sample LW3-G637-1. For the comparison of the field split to the parent sample, four RPD values and one absolute difference were greater than control limits. For the field replicate, the RPD and/or absolute difference values were outside of the control limits for 17 of 40 analytes. The outliers are documented in the validation worksheets.

SDG K0710866: The data for one set of field replicates was submitted: Samples LW3-G612-1 and LW3-G612-2. The C3-phenanthrenes/anthracenes and C1-fluoranthenes/pyrenes RPD values were greater than the control limit. The C4-chrysenes absolute difference was greater than control limits. All other RPD and absolute difference values were within control limits.

SDG K0711067: Samples LW3-G684-2 (field replicate) and LW3-G684-3 (field split) were submitted with parent Sample LW3-G684-1. For the comparison of the field split to the parent sample, the C3-fluorenes, benzo(a)anthracene, chrysene, and C1-chrysenes RPD values were greater than the control limit. The C2-fluorenes absolute difference was greater than control

limits. For the field replicate, the indeno(1,2,3-cd)pyrene RPD value was greater than the control limit.

SDG K0711172: One pair of samples, LW3-G627-1 & LW3-627-2 were submitted as field replicates. The RPD and/or absolute difference values were outside of the control limits for 6 of 40 analytes. A complete list of these outliers is documented in the validation worksheets.

SDG K0711174: Samples LW3-M002-2 (field replicate) and LW3-M002-3 (field split) were submitted with parent Sample LW3-M002-1. For the comparison of the field split to the parent sample, the RPD and/or absolute difference values were outside of the control limits for 16 of 40 analytes. For the field replicate, eight RPD values were outside of control limits. A complete list of these outliers is documented in the validation worksheets.

SDG K0711174 & K0711320: One pair of samples, LW3-G641-1 (submitted in SDG K0711174) & LW3-G641-2 (in K0711320) was submitted as field replicates. The RPD values were outside of control limits for all 40 analytes. A complete list of these outliers is documented in the validation worksheets.

SDG K0711175: One pair of samples, LW3-G659-1 & LW3-G659-2, was submitted as field replicates. The RPD values for 13 of the 40 analytes were greater than the control limit. A complete list of these outliers is documented in the validation worksheets.

SDG K0711220: One pair of samples, LW3-UG02E-1 & LW3-UG02E-2, was submitted as field replicates. The absolute difference values were within control limits.

SDG K0711252: One pair of samples, LW3-G732-1 & LW3-G732-2 was submitted as field replicates. The C2-chrysenes and C3-Fluorenes RPD values were greater than the control limit.

SDG K0711423: Samples LW3-G692-2 (field replicate) and LW3-G692-3 (field split) were submitted with parent Sample LW3-G692-1. For the comparison of the field split to the parent sample, the benzo(b)fluoranthene RPD value was greater than the control limit. For the field replicate, the RPD and/or absolute difference values were outside of the control limits for 20 of 40 analytes. A complete list of these outliers is documented in the validation worksheets.

A second field replicate set (LW3-G759-1 & LW3-G759-2) was also submitted. The RPD and/or absolute difference values were outside of the control limits for 19 of 40 analytes. A complete list of these outliers is documented in the validation worksheets.

SDG K0711706: The data for two field replicate sets (LW3-MC001-B1 & LW3-MC001-B2 and LW3-MC001-C1 & LW3-MC001-C2) were submitted. For the comparison of LW3-MC001-B1 & LW3-MC001-B2, the RPD or absolute difference value for 18 of 20 analytes exceeded control limits. A complete list of these outliers is documented in the validation worksheets. For Samples LW3-MC001-C1 & LW3-MC001-C2 the RPD and/or absolute difference values were within control limits.

SDG K0711709: Samples LW3-C601-B2 (field replicate) and LW3-C601-B3 (field split) were submitted with parent Sample LW3-C601-B1. For the comparison of the field split to the parent

sample, all RPD and/or absolute difference values were within control limits. For the field replicate, the fluorene RPD value was greater than the control limit.

SDG K0711828 & K0711830: The data for two sets of field precision samples were submitted. Samples LW3-C644-B2 (field replicate) and LW3-C644-B3 (field split) were submitted with parent Sample LW3-C644-B1, and Samples LW3-C644-C2 (field replicate) and LW3-C644-C3 (field split) were submitted with parent Sample LW3-C644-C1. All RPD and/or absolute difference values were within control limits.

SDG K0800158: The data for three sets of field precision samples were submitted. Samples LW3-C773-B2 (field replicate) and LW3-C773-B3 (field split) were submitted with parent Sample LW3-C773-B1, Samples LW3-C773-C2 (field replicate) and LW3-C773-C3 (field split) were submitted with parent Sample LW3-C773-C1, and LW3-C773-D2 (field replicate) and LW3-C773-D3 (field split) were submitted with parent Sample LW3-C773-D1. The RPD and/or absolute difference values exceeded the control limits for several analytes. A complete list of these outliers is documented in the validation worksheets.

SDG K0800270 & K0800325: The following field replicates were submitted (all parent samples were reported in SDG K0800270, all field replicates (sample ID ends in 2) and splits (ends in 3) were reported in SDG K0800325):

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C748-B1	LW3-C748-B3	LW3-C748-B2
LW3-C748-C1	LW3-C748-C3	LW3-C748-C2
LW3-C748-D1	LW3-C748-D3	LW3-C748-D2
LW3-C764-B1		LW3-C764-B2
LW3-C764-C1		LW3-C764-C2
LW3-C764-D1		LW3-C764-D2

For both the field split and field replicate precision assessments, the RPD and/or absolute difference values exceeded the control limits for several analytes. A complete list of these outliers is documented in the validation worksheets.

Internal Standards

SDG K0711175: The %R value for the internal standard naphthalene-d8 was less than the lower control limit in Sample LW3-G652. This analysis was run at dilution; the analytes associated with this internal standard were not reported from this analysis. No qualifiers were applied.

SDG K0711423: The %R values for the internal standards chrysene-d12 and perylene-d12 were less than the lower control limits in Sample LW3-G738. This analysis was run at dilution; the analytes associated with these internal standards were not reported from this analysis. No qualifiers were applied.

Reporting Limits

Several samples were diluted to bring target concentrations within range. Reporting limits were elevated accordingly.

SDGs K0712101, K0800487 & K0800516: The method detection limits (MDL) for several compounds were greater than the QAPP MDL. No action was taken since the QAPP method reporting limits (MRL) were met.

Compound Identification

The laboratory “X” flagged the C1-chrysenes results to indicate a potential high bias from an unidentified peak within the C1-chrysenes elution range. The C1-chrysenes results in the following samples were qualified as estimated (J-14):

SDG K0710893: Samples LW3-G741, LW3-G744, LW3-G745, LW3-UG02B, and LW3-UG02C

SDG K0711067: Samples LW3-G747, LW3-G752, LW3-G775, LW3-G769, and LW3-G767

SDG K0711172: Samples LW3-G605, LW3-G607, and LW3-G631

SDG K0711174: Samples LW3-G602, LW3-G603, and LW3-G604

SDG K0711220: 7 of 23 samples

SDG K0711252: 15 of 19 samples

SDG K0711423: 5 of 27 samples

SDG K0711577: Sample LW3-G768

SDG K0711706: Samples LW3-C602-B, LW3-C604-B, and LW3-C605-B

SDG K0712106: Sample LW3-C714-B

SDG K0800158: Samples LW3-G760-B and LW3-G760-D.

SDG K0800487: Sample LW3-C721-B

SDG K0800516: Samples LW3-C739-C, LW3-C739-D, and LW3-C739-E

Reported Results

For several of the alkylated PAH compounds, the reported result was at a concentration greater than the upper end of the initial calibration linear range. These results were “E” flagged by the laboratory. The laboratory does not perform dilutions of these alkylated homologues which are reported as a range of analytes, and not as single components. For the following, the results were estimated (J-20) due to the calibration range exceedance:

SDG K0711174: Samples LW3-G609 and LW3-G639 - C1-phenanthrenes/anthracenes and C1-fluoranthenes/pyrenes

Sample LW3-G609 - C2-naphthalene, C3-naphthalene, C2-phenanthrenes/anthracenes, and C3-phenanthrenes/anthracenes

SDG K0711175: Sample LW3-G655 - C1-phenanthrenes/anthracenes and C1-fluoranthenes/pyrenes

SDG K0711709: Sample LW3-C609-B - C1-fluoranthenes/pyrenes, C1-phenanthrenes/anthracenes, C2-phenanthrenes/anthracenes, C3-phenanthrenes/anthracenes, and C1 and C2-naphthalenes

SDG K0711828: Sample LW3-C652-D - C1-phenanthrene/anthracene

Sample LW3-C648-B - C1-phenanthrene/anthracene and C1-fluoranthene/pyrene

SDG K0711830: Sample LW3-C613-C - C1- and C2-phenanthrene/anthracene

SDG K0712050: Sample LW3-C678-B - C2-naphthalene, C3-naphthalene, and C4-naphthalene

Sample LW3-C678-C - C2-fluorene, C3-fluorene, C2-naphthalene, C3-naphthalene, C4-naphthalene, C1-phenanthrene/anthracene, C2-phenanthrene/anthracene, C3-phenanthrene/anthracene, and C1-fluoranthene/pyrene

SDG K0712106: Samples LW3-C695-D and LW3-C695-E – multiple results. A complete listing of outliers is documented in the validation worksheets.

SDG K0800270: Sample LW3-G676-B - C1-phenanthrenes/anthracenes and C1-fluoranthenes/pyrenes.

SDG K0800450: Sample LW3-C690-E - C2-naphthalene, C3-naphthalene, and C4-naphthalene

SDG K0800516: Sample LW3-C662-B - C1-phenanthrenes/anthracenes

Calculation Verification

SDGs K0711175, K0711709, K0712101, K0800325, & K0800516: Calculation verifications were performed on these SDG. No calculation errors were found. A discrepancy was noted, as discussed in the **Surrogate Compounds** section. No further action was necessary.

IV. OVERALL ASSESSMENT

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

Data were estimated based on MS/MSD recovery and precision outliers. Data were estimated due to interference from an unidentified peak, and due to the calibration range being exceeded.

Data were also qualified as not detected based on contamination in the associated laboratory and field blanks.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3B Sediments
Chlorophenols by Method SW8151 (Modified)

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

SDG	Number of Samples	Validation Level
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K0711423	27 Sediment	Summary
K0711577	4 Sediment	Summary
K0711706	19 Sediment	Summary
K0711709	10 Sediment	Full
K0711828	13 Sediment	Summary
K0711830	20 Sediment	Summary
K0712050	15 Sediment	Summary
K0712101	9 Sediment	Full
K0712106	9 Sediment	Summary
K0712149	6 Sediment	Summary
K0800158	28 Sediment	Summary
K0800270	21 Sediment	Summary
K0800325	13 Sediment	Full
K0800450	27 Sediment	Summary
K0800487	22 Sediment	Summary
K0800516	14 Sediment	Full

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

A complete (100%) verification of the electronic data deliverable (EDD) results was performed by comparison to the hardcopy 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 | Laboratory Control Samples (LCS/LCSD) |
| | Initial Calibration (ICAL) | 1 | Field Replicates and Splits |
| | Continuing Calibration (CCAL) | 1 | Target Analyte List |
| 2 | Laboratory Blanks | | Reporting Limits |
| 1 | Field Blanks | 2 | Compound Identification |
| | Surrogate Compounds | 1 | Calculation Verification |
| 1 | Matrix Spikes/Matrix Spike Duplicates (MS/MSD) | | |

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

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

Holding Times and Sample Receipt

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

Laboratory Blanks

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

Method blanks were analyzed at the appropriate frequency. For the analytical batches noted below, chlorophenols were reported in the method blank. A summary of contaminant levels, associated samples, and action levels is provided in the data validation worksheets.

SDG K0712101: 2,4,6-trichlorophenol (7 results), pentachlorophenol (8 results)

SDG K0800516: 2,4,6-trichlorophenol (1 result), pentachlorophenol (1 result)

Field Blanks

The phenol fraction of the rinsate blanks were analyzed using the standard SVOC methodology, SW8270C. The SVOC and phenol analytes for all rinsate blanks are discussed in the SVOC report.

Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed at the required frequency. All MS/MSD recovery values were within the specified control limits, with the exceptions noted below. If the percent recovery (%R) outlier was due to the presence of high levels of the target analyte present in the parent sample, no action was taken. If the outliers indicated a potential high bias, only the associated positive results were estimated (J-8). If the outliers indicated a potential low bias, results and reporting limits were estimated (J/UJ-8). If the recovery value was less than 10%, the reporting limits were rejected (R-8). No action was taken if only one outlier (in the MS or MSD analysis) was present. A summary of outliers is provided in the data validation worksheets.

All MS/MSD relative percent difference (RPD) values were within the specified control limits, with the exceptions noted below. For RPD outliers, positive results for the affected compounds were qualified as estimated (J-9) in the parent sample.

SDG K0710893: In the MS/MSD performed using Sample LW3-UG02A, the MS %R value for 2,4,6-trichlorophenol was less than the lower control limit.

SDG K0711220: In the MS/MSD performed using Sample LW3-G699, the MS %R value for 2,4,6-trichlorophenol was less than the lower control limit.

SDGs K0711320 & K0711423: In the MS/MSD performed using Sample LW3-G717, the MSD %R value for 2,4,6-trichlorophenol was less than the lower control limit.

SDG K0711577: In the MS/MSD performed using a batched QC Sample, the MSD %R value for 2,4,6-trichlorophenol was less than the lower control limit.

SDG K0711706: In the MS/MSD performed using Sample LW3-C604-B, the MSD %R value for 2,4,6-trichlorophenol was less than the lower control limit. Also, the 2,4,6-trichlorophenol RPD value was greater than the control limit. No qualifier was applied as there was no positive result for this compound in the parent sample.

Laboratory Control Samples

SDG K0711220: The %R value for 2,4,6-trichlorophenol was less than the control limits in the laboratory control sample (LCS) extracted on 12/13/07. Reporting limits for this analyte were qualified as estimated (UJ-10) in all associated samples.

Field Replicates

Two types of field replicates were submitted, field replicates (co-located samples) and field splits. Replicate sample sets are listed below. Field splits were compared to the parent sample results. Field replicates were compared to the average of the field split and parent sample result.

The following acceptance criteria were used to evaluate precision: the RPD control limit is 50% for results greater than five times the reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and replicate must be less than two times the RL.

No data were qualified based on field replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

SDG K0710859: Samples LW3-UG03A-2 (field replicate) and LW3-UG03A-3 (field split) were submitted with parent Sample LW3-UG03A-1. Samples LW3-G637-2 (field replicate) and LW3-G637-3 (field split) were submitted with parent Sample LW3-G637-1. All RPD values and/or absolute differences met the acceptance criteria.

SDG K0710866: The data for one set of field replicates was submitted: Samples LW3-G612-1 and LW3-G612-2. All RPD and/or absolute difference values were within control limits.

SDG K0711067: Samples LW3-G684-2 (field replicate) and LW3-G684-3 (field split) were submitted with parent Sample LW3-G684-1. All RPD and/or absolute difference values were within control limits.

SDG K0711172: One pair of samples, LW3-G627-1 & LW3-627-2 were submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711174: Samples LW3-M002-2 (field replicate) and LW3-M002-3 (field split) were submitted with parent Sample LW3-M002-1. The RPD and/or absolute difference values were within control limits.

SDG K0711174 & K0711320: One pair of samples, LW3-G641-1 (submitted in SDG K0711174) & LW3-G641-2 (in K0711320) was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711175: One pair of samples, LW3-G659-1 & LW3-G659-2, was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711220: One pair of samples, LW3-UG02E-1 & LW3-UG02E-2, was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711252: One pair of samples, LW3-G732-1 & LW3-G732-2 was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711423: Samples LW3-G692-2 (field replicate) and LW3-G692-3 (field split) were submitted with parent Sample LW3-G692-1. All RPD and/or absolute difference values were within control limits.

A second field replicate set (LW3-G759-1 & LW3-G759-2) was also submitted. All RPD and/or absolute difference values were within control limits.

SDG K0711706: The data for two field replicate sets (LW3-MC001-B1 & LW3-MC001-B2 and LW3-MC001-C1 & LW3-MC001-C2) were submitted. All RPD and/or absolute difference values were within control limits.

SDG K0711709: Samples LW3-C601-B2 (field replicate) and LW3-C601-B3 (field split) were submitted with parent Sample LW3-C601-B1. For the comparison of the field split to the parent

sample, the pentachlorophenol RPD value was greater than the control limit. For the field replicate, all RPD and/or absolute difference values were within control limits.

SDG K0711828 & K0711830: The data for two sets of field precision samples were submitted. Samples LW3-C644-B2 (field replicate) and LW3-C644-B3 (field split) were submitted with parent Sample LW3-C644-B1, and Samples LW3-C644-C2 (field replicate) and LW3-C644-C3 (field split) were submitted with parent Sample LW3-C644-C1. All RPD and/or absolute difference values were within control limits.

SDG K0800158: The data for three sets of field precision samples were submitted. Samples LW3-C773-B2 (field replicate) and LW3-C773-B3 (field split) were submitted with parent Sample LW3-C773-B1, Samples LW3-C773-C2 (field replicate) and LW3-C773-C3 (field split) were submitted with parent Sample LW3-C773-C1, and LW3-C773-D2 (field replicate) and LW3-C773-D3 (field split) were submitted with parent Sample LW3-C773-D1. All RPD and/or absolute difference values were within control limits.

SDG K0800270 & K0800325: The following field replicates were submitted (all parent samples were reported in SDG K0800270, all field replicates (sample ID ends in 2) and splits (ends in 3) were reported in SDG K0800325):

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C748-B1	LW3-C748-B3	LW3-C748-B2
LW3-C748-C1	LW3-C748-C3	LW3-C748-C2
LW3-C748-D1	LW3-C748-D3	LW3-C748-D2
LW3-C764-B1		LW3-C764-B2
LW3-C764-C1		LW3-C764-C2
LW3-C764-D1		LW3-C764-D2

For both the field split and field replicate precision assessments, all RPD and/or absolute difference values were within control limits.

Target Analyte List

It was noted by the laboratory that 2,3,5,6-tetrachlorophenol could not be separated from 2,3,4,6-tetrachlorophenol.

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). Refer to the data validation worksheets for a detailed list of these outliers.

SDG K0710859:

- Sample LW3-G637-2 2,3,5,6-tetrachlorophenol (NJ-3)

- Sample LW3-G654 2,4,6-trichlorophenol (NJ-3)
- 7 results for pentachlorophenol (NJ-3)

SDG K0710866:

- Sample LW3-G628 pentachlorophenol (J-3)
- Sample LW3-G612-2 pentachlorophenol (NJ-3)

SDG K0710893:

- Sample LW3-G693 2,3,5,6-tetrachlorophenol (J-3)
- Samples LW3-G740 & LW3-G744 2,3,5,6-tetrachlorophenol (NJ-3)
- Sample LW3-G694 2,4,6-trichlorophenol (NJ-3)
- 3 results for pentachlorophenol (NJ-3)

SDG K0711067:

- 4 results for pentachlorophenol (J-3)
- 5 results for pentachlorophenol (NJ-3)

SDG K0711172:

- 4 results for pentachlorophenol (NJ-3)
- Sample LW3-G618 2,3,5,6-tetrachlorophenol (J-3)
- Samples LW3-G629 & LW3-G631 2,3,5,6-tetrachlorophenol (NJ-3)

SDG K0711174:

- Sample LW3-MG006 pentachlorophenol (J-3) and 2,3,5,6-tetrachlorophenol (NJ-3)
- Sample LW3-G634 2,3,4,5-tetrachlorophenol (J-3) and 2,4,6-trichlorophenol (NJ-3)

SDG K0711175:

- Sample LW3-G644 pentachlorophenol (J-3)
- Sample LW3-G647 2,4,6-trichlorophenol (J-3)

SDG K0711220:

- LW3-UG11C pentachlorophenol (NJ-3)

SDG K0711252:

- 3 results for pentachlorophenol (J-3)
- Sample LW3-G715 pentachlorophenol (NJ-3)

SDG K0711320:

- Samples LW3-MG004 & LW3-G660 pentachlorophenol (J-3)

SDG K0711423:

- Sample LW3-G743 pentachlorophenol (J-3)
- 13 results for pentachlorophenol (NJ-3)

SDG K0711577:

- Sample LW3-G771 2,3,4,5-tetrachlorophenol (J-3) and pentachlorophenol (NJ-3)

SDG K0711706:

- Sample LW3-C605-C pentachlorophenol (J-3)
- Sample LW3-C600-B 2,4,6-trichlorophenol (J-3)

SDG K0711709:

- 3 results for pentachlorophenol (NJ-3)
- Sample LW3-C606-C 2,4,5-trichlorophenol (NJ-3)

SDG K0711828:

- Sample LW3-C652-D pentachlorophenol (J-3)

SDG K0711830:

- 4 pentachlorophenol results (NJ-3)
- Sample LW3-C613-C 2,4,5-trichlorophenol (J-3)
- Samples LW3-C615-B & LW3-C630-C 2,4,5-trichlorophenol (NJ-3)
- Sample LW3-C634-B 2,3,5,6-tetrachlorophenol (J-3)

SDG K0712050:

- Sample LW3-C712-B pentachlorophenol (J-3)
- Samples LW3-C701-B & LW3-C686-C pentachlorophenol (NJ-3)
- Sample LW3-C704-D 2,4,6-trichlorophenol (J-3)
- Sample LW3-C704-B 2,4,6-trichlorophenol (NJ-3)
- Sample LW3-C701-B 2,3,5,6-tetrachlorophenol (NJ-3)

SDG K0712101:

- Sample LW3-MC008-D 2,4,5-trichlorophenol (NJ-3)
- Sample LW30C602-D: 2,4,5-trichlorophenol, 2,3,4,5-tetrachlorophenol, pentachlorophenol (NJ-3)

SDG K0712106:

- Samples LW3-C695-B & LW3-C714-B pentachlorophenol (J-3)

- Sample LW3-C695-E 2,3,4,5-tetrachlorophenol (NJ-3)
- Samples LW3-C733-B & LW3-C742-B pentachlorophenol (NJ-3)

SDG K0712149:

- Samples LW3-C743-D & LW3-C749-B pentachlorophenol (J-3)

SDG K0800158:

- 5 results for pentachlorophenol (J-3)
- 6 results for pentachlorophenol (NJ-3)
- 3 results for 2,3,5,6-tetrachlorophenol (NJ-3)
- Sample LW3-C762-C 2,4,6-trichlorophenol (NJ-3)
- Sample LW3-C773-D2 2,4,6-trichlorophenol (J-3)

SDG K0800270:

- Samples LW3-C676-B & LW3-C757-B pentachlorophenol (J-3)
- Sample LW3-C764-D1 2,4,5-trichlorophenol (J-3); 2,3,5,6-tetrachlorophenol and pentachlorophenol (NJ-3)

SDG K0800325:

- Samples LW3-C783-B & LW3-C783-C 2,4,5-trichlorophenol (J-3)
- Sample LW3-C748-C3 2,4,6-trichlorophenol (J-3)
- Sample LW3-C783-B pentachlorophenol (J-3)
- Sample LW3-C764-D2 2,3,5,6-tetrachlorophenol (J-3)

SDG K0800450:

- Samples LW3-C690-C & LW3-C702-C 2,4,6-trichlorophenol (J-3)
- Sample LW3-C665-B 2,4,6-trichlorophenol (NJ-3)
- Samples LW3-C688-D & LW3-C690-E 2,4,5-trichlorophenol (J-3)
- Samples LW3-C688-F & LW3-C690-D 2,4,5-trichlorophenol (NJ-3)
- Sample LW3-C688-D 2,3,4,5-tetrachlorophenol (NJ-3)
- Samples LW3-C626-D & LW3-C690-D 2,3,5,6-tetrachlorophenol (J-3)
- 5 results for 2,3,5,6-tetrachlorophenol (NJ-3)
- Sample LW3-C703-D pentachlorophenol (J-3)
- 4 results for pentachlorophenol (NJ-3)

SDG K0800487:

- Sample LW3-C703-C pentachlorophenol (NJ-3)

- Sample LW3-C706-D 2,3,5,6-tetrachlorophenol (NJ-3) & pentachlorophenol (J-3)
- Sample LW3-C708-B 2,3,4,5-tetrachlorophenol & 2,3,5,6-tetrachlorophenol (J-3)

SDG K0800516:

- Samples LW3-C738-B, LW3-C739-D, LW3-C651-B pentachlorophenol (J-3)
- Sample LW3-C738-C 2,4,6-trichlorophenol (J-3) & 2,3,5,6-tetrachlorophenol (NJ-3)
- Samples LW3-C739-C & LW3-C738-B pentachlorophenol (NJ-3)
- Sample LW3-C651-B 2,3,5,6-tetrachlorophenol (J-3)

Reporting Limits

Several RLs were elevated by the laboratory due to interferences or dilution analyses. In some cases the elevated RL was greater than the QAPP specified method reporting limit (MRL).

Calculation Verification

SDGs K0711175, K0711709, K0712101, K0800325, K0800516: Calculation verifications were performed on these SDGs. 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.

Data were qualified as estimated or tentatively identified because the confirmation criteria were exceeded. Data were also estimated due to an LCS %R outlier. Data were qualified as not detected based on laboratory blank contamination.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3B Sediments
Pesticides by Method SW8081A

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

SDG	Number of Samples	Validation Level
K0710859	20 Sediment	Summary
K0710866	12 Sediment	Summary
K0710893	24 Sediment & 2 Equipment Blank	Summary
K0711067	19 Sediment	Summary
K0711172	20 Sediment	Full
K0711174	21 Sediment & 1 Equipment Blank	Summary
K0711175	12 Sediment & 2 Equipment Blank	Summary
K0711220	23 Sediment & 1 Equipment Blank	Summary
K0711252	19 Sediment & 2 Equipment Blank	Summary
K0711320	4 Sediment	Summary
K0711423	27 Sediment & 2 Equipment Blank	Summary
K0711577	4 Sediment	Summary
K0711706	19 Sediment & 1 Equipment Blank	Summary
K0711709	10 Sediment	Summary
K0711828	13 Sediment & 2 Equipment Blank	Summary
K0711830	20 Sediment	Summary
K0712050	15 Sediment & 1 Equipment Blank	Summary
K0712101	9 Sediment	Full
K0712106	9 Sediment & 1 Equipment Blank	Summary
K0712149	6 Sediment	Summary
K0800158	28 Sediment & 2 Equipment Blank	Screening
K0800270	21 Sediment & 1 Equipment Blank	Summary
K0800325	13 Sediment & 1 Equipment Blank	Full
K0800450	27 Sediment	Summary
K0800487	22 Sediment	Summary
K0800516	14 Sediment & 1 Equipment Blank	Full

I. DATA PACKAGE COMPLETENESS

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

SDG K0800270: Two DDT/Endrin breakdown standards were not included in the data package. The laboratory was contacted and the missing standards were submitted 3/26/08.

SDG K0800450: One DDT/Endrin breakdown standard was not included in the data package. The missing standard was located in SDG K0800325. No further action was necessary.

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 (MS/MSD) |
| | DDT/Endrin Breakdown | 2 | Laboratory Control Samples (LCS) |
| | Initial Calibration (ICAL) | 1 | Field Replicates |
| 1 | Continuing Calibration (CCAL) | 1 | Reporting Limits |
| 2 | Laboratory Blanks | 2 | Compound Identification |
| 1 | Field Blanks | 1 | Calculation Verification (full validation only) |
| 1 | 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

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

SDG K0711423: Several analytes in Samples LW3-G776 and LW3-G778 were reported from re-analyses that 2 and 4 days, respectively, outside the 40 day extract holding time. As the initial analyses were performed within the extract holding time and the results from the re-extraction were similar, no qualifiers were assigned.

Continuing Calibration (CCAL)

The percent difference (%D) values for various compounds were outside the control limits on the secondary column (DB-35MS) in many of the CCAL. In all cases, the %D values on the primary column (DB-XLB) were acceptable. Since the secondary column is only used for compound identification confirmation, no action was taken for the following %D outliers:

SDG K0710859: The %D values for methoxychlor and endrin were greater than the upper control limit in one CCAL.

SDG K0710866: The methoxychlor %D values were greater than the control limit in two CCAL.

SDG K0710893: The %D values for alpha BHC, gamma BHC, and heptachlor were greater than the upper control limit in three CCAL.

SDG K0711423, K0711577: The %D values for methoxychlor was greater than the upper control limit (DB-35MS) in one CCAL.

SDG K0711709: The %D values for aldrin, beta BHC, delta BHC, and/or heptachlor were greater than the upper control limit in all five CCAL.

SDG K0711830: The %D values for aldrin, beta BHC, delta BHC, and/or heptachlor were greater than the upper control limit in five of 14 CCAL.

SDG K0712101: The %D values for several analytes were greater than the upper control limit in two CCAL.

SDG K0712149: The %D values for aldrin and delta BHC were greater than the upper control limit for the secondary column (DB-35MS) in two of the six CCAL.

SDG K0800325: The methoxychlor %D value was greater than the control limit in one CCAL.

SDG K0800450: The %D values for several analytes were greater than the upper control limit in eight CCAL.

SDG K0800487: The %D values for several analytes were greater than the upper control limit in six CCAL.

SDG K0800516: The %D values for several analytes were greater than the upper control limit in two CCAL.

Laboratory Blanks

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

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

SDG K0710859: oxychlordan (3 results), 2,4'-DDE (4 results)

SDG K0710866: oxychlordan (2 results), 2,4'-DDE (1 result)

SDG K0710893: oxychlordan and 2,4'-DDE (2 results), 4,4'-DDD and delta-BHC (1 result)

SDG K0711174 (aqueous): 2,4'-DDT (1 result)

SDG K0711174 (sediment): beta-BHC (2 results), delta-BHC (3 results), hexachlorobutadiene (1 result)

SDG K0711175 (aqueous): 2,4'-DDT (2 results)

SDG K0711220: 2,4'-DDT (1 result - EB), hexachlorobutadiene (1 result)

SDG K0711252 (aqueous): 4,4'-DDT (1 result), 2,4'-DDT (2 results)

SDG K0711252 (sediment): 4,4'-DDD (1 result)

SDG K0711423 (aqueous): 4,4'-DDT (1 result), 2,4'-DDT (2 results)

SDG K0711706: (aqueous): 2,4'-DDT (1 result)

SDG K0711828 (aqueous): 2,4'-DDT (2 results)

SDG K0711830: heptachlor epoxide (1 result), endosulfan sulfate (2 results)

SDG K0712050: 4,4'-DDT (1 result), 2,4'-DDT (1 result)

SDG K0712101: oxychlordan (2 results), trans-nonachlor (3 results)

SDG K0712106 (aqueous): 4,4'-DDT (1 result), 2,4'-DDT (1 result)

SDG K0712106 (sediment): beta-BHC (1 result), delta-BHC (1 result)

SDG K0800158 (aqueous): 2,4'-DDT (1 result)

SDG K0800158 (sediment): heptachlor epoxide (2 results)

SDG K0800270 (aqueous): 2,4'-DDT (1 result)

SDG K0800270 (sediment): hexachlorobutadiene (1 result)

SDG K0800325 (aqueous): 2,4'-DDT (1 result)

SDG K0800450 (sediment): 4,4'-DDD (1 result)

SDG K0800450 (aqueous): 2,4'-DDT (1 result)

SDG K0800516 (aqueous): 2,4'-DDT (1 result)

Field Blanks

SDG K0710893: Two equipment blanks were submitted. Hexachlorobutadiene was detected in Sample LW3-SAG901 and positive results for gamma-BHC and hexachlorobutadiene were detected in Sample LW3-UG901. All associated results were greater than the action levels, no qualifiers were applied.

SDG K0711174: One equipment blank was submitted, LW3-SAG902. No positive results remained in this blank after qualifiers based on the laboratory blank were assigned.

SDG K0711175: Two equipment blanks were submitted. A positive result for gamma-BHC remained in Sample LW3-SAG903 after qualifiers based on the laboratory blank were assigned. Gamma-BHC was not detected in any associated samples, no qualifiers were applied. No

positive results remained in Sample LW3-SAG904 after qualifiers based on the laboratory blank were assigned.

SDG K0711220: One equipment blank was submitted, LW3-SAG901. No positive results remained after qualifiers based on the laboratory blank were assigned.

SDG K0711252: Two equipment blanks were submitted, LW3-SAG905 and LW3-SAG906. No positive results remained in either blank after laboratory blank qualifiers were assigned.

SDG K0711423: Two equipment blanks were submitted, LW3-SAG907 and LW3-SAG908. No positive results remained in either blank after laboratory blank qualifiers were assigned.

SDG K0711706: One equipment blank was submitted, LW3-MC901. After qualification for method blank contamination, a positive result for oxychlordane remained. Oxychlordane was detected in the associated samples at concentrations greater than the action level, no qualifiers were applied.

SDG K0711828: Two equipment blanks were submitted, LW3-SAC901 and LW3-SAC902. No positive results remained in either blank after qualifiers based on the method blank were assigned.

SDG K0712050: One equipment blank was submitted, LW3-SAC903. No positive results remained in this blank after qualifiers based on the laboratory blank were assigned.

SDG K0712106: One equipment blank was submitted, LW3-SAC904. After qualifiers based on method blank contamination were issued, no positive results were detected in this blank.

SDG K0800158: Two equipment blanks were submitted, LW3-SAC905 and LW3-SAC906. After qualification for method blank contamination, positive results for beta-BHC remained in both field blanks. Beta-BHC was either not detected in the associated samples or present at concentrations greater than the action level, no qualifiers were applied.

SDG K0800270: One equipment blank was submitted, LW3-SAC907. After qualification for method blank contamination, a positive result for beta-BHC remained. Beta-BHC was either not detected in the associated samples or present at concentrations greater than the action level, no qualifiers were applied.

SDG K0800325: One equipment blank was submitted, LW3-SAC908. After qualification for method blank contamination, a positive result for cis-chlordane remained. Cis-chlordane was either not detected in the associated samples or was present at concentrations greater than the action level; no qualifiers were applied.

SDG K0800450: One equipment blank was submitted, LW3-SAC908. After qualification for method blank contamination, a positive result for alpha-chlordane remained. Alpha-chlordane was detected in the associated samples at concentrations greater than the action level, no qualifiers were applied.

SDG K0800516: One equipment blank was submitted, LW3-SAC909. After qualification for method blank contamination, no positive results remained in this blank. No action was necessary.

Surrogate Compounds

SDG K0710859: The percent recovery (%R) values for tetrachloro-m-xylene and decachlorobiphenyl were less than the lower control limit in a laboratory control sample duplicate (LCSD). The LCSD and the associated samples were re-extracted and re-analyzed with acceptable results. Qualifiers are not issued to QC samples, so no action was taken.

SDG K0711174: The %R values for tetrachloro-m-xylene was less than the lower control limit in Sample LW3-MG010. The recovery of decachlorobiphenyl was within control limits; no qualifiers were applied.

SDG K0712050: The %R values for tetrachloro-m-xylene were less than the lower control limit in Samples LW3-C704-B, LW3-C704-C, and LW3-C704-D. The decachlorobiphenyl recovery was within control limits; no qualifiers were applied.

SDG K0712106: The %R value for tetrachloro-m-xylene was less than the lower control limit in a batch QC MS sample. The decachlorobiphenyl recovery was acceptable; no action was taken.

SDG K0800450: The %R value for tetrachloro-m-xylene was greater than the upper control limit in Sample LW3-C690-E. The recovery of decachlorobiphenyl was within control limits; no qualifiers were applied.

SDG K0800487: The %R values for tetrachloro-m-xylene and decachlorobiphenyl were greater than the upper control limits in Samples LW3-C708-B and LW3-C708-C. Extracts for both samples were diluted (50x) prior to analysis. No qualification was required.

SDG K0800516: The %R value decachlorobiphenyl was greater than the upper control limit in Sample LW3-C651-B. The %R value for tetrachloro-m-xylene was within control limits; no qualifiers were applied. The %R values for both surrogates were outside of control limits in Samples LW3-C662-C & LW3-C662-D. Both sample extracts were diluted 100x prior to analysis. No qualifiers were applied.

Matrix Spikes/Matrix Spike Duplicates

SDG K0710859: The %R value for 4,4'-DDT was greater than the upper control limit in the matrix spike (MS) performed on Sample LW3-G663. As the %R value in the matrix spike duplicate (MSD) was acceptable no qualifiers were required. The 4,4'-DDD relative percent difference (RPD) value was greater than the control limit. The positive 4,4'-DDD result in the parent sample was estimated (J-9).

SDG K0710866: The %R value for 4,4'-DDT was less than the lower control limit in the MSD performed on Sample LW3-G612-2. As the %R value in the MS was acceptable no qualifiers were required. Aldrin was not recovered in the MS and not detected in the parent sample; the aldrin reporting limit was rejected (R-8).

The RPD values for 4,4'-DDD and 4,4'-DDT were greater than the control limit in this MS/MSD set. These compounds were detected in the parent sample and the reported results were estimated (J-9). The RPD value for hexachloroethane was greater than the control limit in the MS/MSD

performed on Sample LW3-G622. This compound was not detected in the parent sample and reporting limits were not affected; no qualifiers were required.

SDG K0710893: Only an MS was submitted for the equipment blanks. Precision was assessed from the LCS/LCSD analysis. For the sediments, a MS/MSD set using Sample LW3-UG02A was submitted. The RPD value for hexachloroethane exceeded the control limit; this analyte was not detected in the parent sample, no qualifiers were applied.

A batch sediment MS/MSD set was also submitted. Several %R and RPD values exceeded the control limits in the batch MS/MSD analyses. No qualifiers were applied as the parent sample was not from this SDG.

SDG K0711067: The RPD value for cis-nonachlor was greater than the control limit in the batch QC MS/MSD. As the parent sample was not a part of this SDG no qualifiers were required.

SDG K0711172: The %R value for 4,4'-DDT was less than the lower control limit in the MS/MSD performed on Sample LW3-G614. The positive result for this compound was estimated (J-8). The MSD %R value and the RPD value for cis-nonachlor were greater than the control limit in the MS/MSD performed on Sample LW3-G624. This compound was not detected in the parent sample and reporting limits were not affected; no qualifiers were required.

The RPD value for hexachloroethane was greater than the control limit in the batch MS/MSD set. As the parent sample was not a part of this SDG no qualifiers were required.

SDGs K0711174, K0711175, K0711252, K0711423, K0712050, K0712101, K0800158, & K0800325: No MS/MSD analyses were submitted. Precision was assessed from the laboratory control sample/laboratory control sample duplicate (LCS/LCSD).

SDG K0711220: The RPD value for hexachloroethane was greater than the control limit in the MS/MSD performed on Sample LW3-G681; this compound was not detected in the parent sample and reporting limits were not affected. No qualifiers were required.

SDG K0711252: Multiple %R values and RPD values were outside controls limit in the batch QC MS/MSD. As the parent sample was not a part of this SDG no qualifiers were required. The outliers are documented in the data validation worksheets.

SDG K0711320: The RPD values for cis-nonachlor, trans-nonachlor, and oxychlordan were greater than the control limit in the batch QC MS/MSD. As the parent sample was not a part of this SDG no qualifiers were required.

SDG K0711423: The %R value for hexachloroethane was less than 10% in the MSD performed on Sample LW3-G717. As the %R value in the MS was acceptable the hexachloroethane reporting limit in the parent sample was estimated (UJ-8) rather than rejected. The RPD value for hexachloroethane was also greater than the control limit in this MS/MSD. As this compound was not detected in the parent sample no qualifiers based on the RPD outlier were assigned.

A batch sediment MS/MSD set was also submitted. The %R values for toxaphene were greater than the upper control limit, and the RPD value for hexachloroethane was greater than the upper

control limit in the batch QC MS/MSD. As the parent sample was not a part of this SDG no qualifiers were required.

SDG K0711577: MS/MSD analyses were performed using Sample LW3-G783. The MS/MSD set was originally extracted on 1/11/08; however, the MS/MSD set was re-extracted on 2/12/08. A subset of the analytes were reported from each of the two sets.

For the MS/MSD set extracted 1/11/08, the %R values for toxaphene were greater than the upper control limit. This analyte was not detected in the parent sample and no qualifiers were applied. The 1/11/08 MS %R values for hexachloroethane and hexachlorobutadiene were less than the lower control limit and the RPD values exceeded the control limit. These analytes were reported from the re-extraction on 2/12/08, so no qualifiers were required. The 2/12/08 RPD value for hexachloroethane exceeded the control limit. This analyte was not detected in the parent sample; no qualifiers were applied.

SDG K0711706: The RPD value for hexachloroethane exceeded the control limit in the MS/MSD using Sample LW3-MC006-B; this compound was not detected in the parent sample, no qualifiers were assigned.

SDG K0711709: The toxaphene MSD %R value was greater than the control limit in the batch QC MS/MSD. As the parent sample was not a part of this SDG no qualifiers were required.

SDG K0711830: The MSD %R value for toxaphene was greater than the control limit in the MS/MSD using Sample LW3-C615-B; this compound was not detected in the parent sample, no qualifiers were assigned.

The hexachloroethane RPD value exceeded the control limit in the Sample LW3-C634-B MS/MSD set; this compound was not detected in the parent sample, no qualifiers were assigned.

SDG K0712050: The hexachloroethane MS %R value was less than the lower control limit in the LW3-C704-B MS/MSD set. The RPD value was greater than the control limit. Hexachloroethane was not detected in the parent sample, the reporting limit was estimated (UJ-8).

SDG K0712106: The RPD values for oxychlordan, cis-nonachlor, and trans-nonachlor were outside the control limits in the MS/MSD using Sample LW3-C714-B; oxychlordan and cis-nonachlor were not detected in the parent sample, no qualifiers were assigned. The positive result for trans-nonachlor was estimated (J-9) in the parent sample.

The MS %R value for hexachloroethane was less than 10% in the batch QC MS/MSD. As the parent sample was not a part of this SDG no qualifiers were required.

SDG K0712149: The RPD values for oxychlordan, cis-nonachlor, and trans-nonachlor were outside the control limits in the batch QC MS/MSD. As the parent sample was not a part of this SDG no qualifiers were required.

SDG K0800158: MS/MSD analyses were performed using Sample LW3-C760-C. The RPD values for gamma-chlordane and 2,4'-DDT exceeded the 40% control limit. Positive results for these analytes were qualified as estimated (J-9) in the parent sample. The RPD values for

beta-endosulfan and 2,4'-DDD exceeded the 40% control limit. These analytes were not detected in the parent sample; no qualifiers were required.

SDG K0800270: Two sets of MS/MSD were submitted. The RPD values for four compounds were greater than the control limit in the batch QC MS/MSD. As the parent sample was not a part of this SDG no qualifiers were required.

For the MS/MSD performed on Sample LW3-C757-C, the %R values for gamma-chlordane and toxaphene were greater than the upper control limit in both MS and MSD, and the %R values for alpha-chlordane, 4,4'-DDD, and 2,4'-DDT were greater than the upper control limits in the MS only. The result for gamma-chlordane was estimated (J-8) in the parent sample. Toxaphene was not detected in the parent sample and reporting limits were determined to be unaffected. Qualifiers were not assigned for single %R outliers. Also, the RPD values for delta-BHC, endosulfan I, alpha-chlordane, 4,4'-DDE, hexachloroethane, and 2,4'-DDT exceeded the control limit. Positive results for alpha-chlordane, 4,4'-DDE, and 2,4'-DDT were estimated (J-9). The analytes associated with the remaining outliers were not detected in the parent sample and no further qualifiers were required.

SDG K0800450: The RPD values for 4,4'-DDT and endrin ketone were greater than the control limit in the MS/MSD performed on Sample LW3-C679-D. The result for 4,4'-DDT was estimated (J-9) in the parent sample. Endrin ketone was not detected in the parent sample and reporting limits were unaffected; no qualifiers were required.

SDG K0800487: Multiple %R values and RPD values were outside controls limit in the batch QC MS/MSD. As the parent sample was not a part of this SDG no qualifiers were required. The outliers are documented in the data validation worksheets.

SDG K0800516: MS/MSD analyses were performed using Sample LW3-C738-B. There was no recovery for 4,4'-DDD in the MSD, but the recovery was within control limits in the MS. The MS %R value for 4,4'-DDT was less than the lower control limit, but within control limits in the MSD. No qualifiers were required for these single outliers. The %R values for toxaphene were greater than the upper control limit. Toxaphene was not detected in the parent sample; no qualifiers were required. The RPD value for 4,4'-DDD was outside control limits. The result for 4,4'-DDD was estimated (J-9) in the parent sample.

Laboratory Control Sample/Laboratory Control Sample Duplicate

SDGs K0710859, K0710983: The %R value for endrin aldehyde was less than the lower control limit in the LCS. Endrin aldehyde was estimated (UJ-10) in the six associated samples.

SDG K0711252: The %R value for hexachlorobenzene was less than the lower control limit in the LCS. Hexachlorobenzene was estimated (UJ-10) in the single associated sample.

SDG K0711577: The %R values for hexachloroethane and hexachlorobutadiene were less than the lower control limits in the sediment LCS extracted 1/11/08. All results for these pesticides were reported from the 2/12/08 extraction batch. No qualifiers were required.

SDG K0711706: The %R values for hexachlorobenzene and mirex were less than the lower control limit in the sediment LCS extracted on 1/15/08. Hexachlorobenzene and mirex were estimated (UJ-10) in the associated sample. The RPD value for 4,4'-DDD was outside control limits in the aqueous LCS/LCSD; this analyte was not detected in the associated sample, no further qualifiers were applied.

SDG K0711828: The %R values for hexachlorobenzene and mirex were less than the lower control limit in the LCS/LCSD set. Results for hexachlorobenzene and mirex were estimated (UJ-10) in the associated samples.

SDG K0712050: The RPD values for 15 of the 40 spiked analytes were greater than the control limit in the aqueous LCS/LCSD. None of the analytes associated with RPD outliers were detected in the rinsate blank and no qualifiers were required. In the sediment LCD the %R value for hexachlorobenzene was less than the lower control limit. Hexachlorobenzene was estimated (J/UJ-10) in the associated samples.

SDG K0712106: In the sediment LCS the %R value for hexachlorobenzene was less than the lower control limit. Reporting limits for hexachlorobenzene were estimated (UJ-10) in the associated samples.

In the aqueous LCS/LCSD the RPD values for 15 analytes were outside control limits. These analytes were not detected in the associated sample, no qualifiers were applied. The outliers are documented in the validation worksheets.

SDG K0800516: The RPD values for aldrin and alpha-endosulfan were greater than the control limit in the aqueous LCS/LCSD. These compounds were not detected in the associated equipment blank; no action was necessary.

Field Replicates

Two types of field replicates were submitted, field replicates (co-located samples) and field splits. Replicate sample sets are listed below. Field splits were compared to the parent sample results. Field replicates were compared to the average of the field split and parent sample result.

The following acceptance criteria were used to evaluate precision: the RPD control limit is 50% for results greater than five times the reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and replicate must be less than two times the RL. No data were qualified based on field replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

SDG K0710859: Samples LW3-UG03A-2 (field replicate) and LW3-UG03A-3 (field split) were submitted with parent Sample LW3-UG03A-1. Samples LW3-G637-2 (field replicate) and LW3-G637-3 (field split) were submitted with parent Sample LW3-G637-1. For the comparison of the field splits to the parent samples, the 4,4'-DDT RPD values were greater than the control limit in both sets. All other RPD values and/or absolute differences met the acceptance criteria.

SDG K0710866: The data for one set of field replicates was submitted: Samples LW3-G612-1 and LW3-G612-2. All absolute difference values were within control limits.

SDG K0711067: Samples LW3-G684-2 (field replicate) and LW3-G684-3 (field split) were submitted with parent Sample LW3-G684-1. For the comparison of the field split to the parent sample, the RPD or absolute difference values for endosulfan I, 4,4'-DDT, cis-nonachlor, and aldrin were greater than control limits. For the field replicate, the RPD and/or absolute difference values for heptachlor epoxide, endosulfan II, 4,4'-DDT, and endrin ketone were outside the control limits.

SDG K0711172: One pair of samples, LW3-G627-1 & LW3-627-2 were submitted as field replicates. The RPD values for 2,4'-DDD and 4,4'-DDT exceeded the control limits. All other RPD and/or absolute difference values were within control limits.

SDG K0711174: Samples LW3-M002-2 (field replicate) and LW3-M002-3 (field split) were submitted with parent Sample LW3-M002-1. For the comparison of the field split to the parent sample, the RPD values for five compounds were outside control limits. For the field replicate, the RPD values for six compounds were outside the control limits.

SDG K0711174 & K0711320: One pair of samples, LW3-G641-1 (submitted in SDG K0711174) & LW3-G641-2 (in K0711320) was submitted as field replicates. The RPD values for 4,4'-DDE and 4,4'-DDT exceeded the control limits.

SDG K0711175: One pair of samples, LW3-G659-1 & LW3-G659-2, was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711220: One pair of samples, LW3-UG02E-1 & LW3-UG02E-2, was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711252: One pair of samples, LW3-G732-1 & LW3-G732-2 was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711423: Samples LW3-G692-2 (field replicate) and LW3-G692-3 (field split) were submitted with parent Sample LW3-G692-1. For the comparison of the field split to the parent sample, the 2,4'-DDT RPD value was greater than the control limit. For the field replicate, the absolute differences for heptachlor and 2,4'-DDD and the RPD value for 2,4'-DDT were greater than the control limits.

A second field replicate set (LW3-G759-1 & LW3-G759-2) was also submitted. The absolute differences for 2,4'-DDT and hexachlorobutadiene were greater than the control limit.

SDG K0711706: The data for two field replicate sets (LW3-MC001-B1 & LW3-MC001-B2 and LW3-MC001-C1 & LW3-MC001-C2) were submitted. For Samples LW3-MC001-B1 & LW3-MC001-B2, the absolute difference for five analytes were outside control limits. The outliers are documented in the validation worksheets. For Samples LW3-MC001-C1 & LW3-MC001-C2, the RPD and/or absolute difference values were within control limits.

SDG K0711709: Samples LW3-C601-B2 (field replicate) and LW3-C601-B3 (field split) were submitted with parent Sample LW3-C601-B1. For the comparison of the field split to the parent sample, the RPD values for heptachlor and 4,4'-DDT exceeded the control limits. For the field

replicate, the RPD values for heptachlor, methoxychlor, 2,4'-DDT, and 4,4'-DDT exceeded the control limits.

SDG K0711828 & K0711830: The data for two sets of field precision samples were submitted. Samples LW3-C644-B2 (field replicate) and LW3-C644-B3 (field split) were submitted with parent Sample LW3-C644-B1, and Samples LW3-C644-C2 (field replicate) and LW3-C644-C3 (field split) were submitted with parent Sample LW3-C644-C1. For the comparison of the field splits to the parent samples, the absolute difference for hexachloroethane was outside control limits between Samples LW3-C644-C1 & LW3-C644-C3. For the field replicates, all RPD and/or absolute difference values were within control limits.

SDG K0800158: The following field precision samples were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C773-B1	LW3-C773-B3	LW3-C773-B2
LW3-C773-C1	LW3-C773-C3	LW3-C773-C2
LW3-C773-D1	LW3-C773-D3	LW3-C773-D2

The following outliers were noted:

Replicates	RPD > 50%	Diff > 2x RL
LW3-C773-B1 and LW3-C773-B3	trans-chlordane, 4,4'-DDE, and 4,4'-DDD	4,4'-DDT
LW3-C773-C1/C3 and LW3-C773-C2	trans-chlordane, 4,4'-DDD, 4,4'-DDT, 2,4'-DDD, trans-nonachlor, and 2,4'-DDT	cis-chlordane and beta-endosulfan
LW3-C773-C1 and LW3-C773-C3	trans-chlordane, 4,4'-DDE, 4,4'-DDT, 2,4'-DDD, and 2,4'-DDT	cis-chlordane, beta-endosulfan, and trans-nonachlor
LW3-C773-D1/D3 and LW3-C773-D2	4,4'-DDD	
LW3-C773-D1 and LW3-C773-D2	trans-chlordane, 4,4'-DDD, methoxychlor, 2,4'-DDD, and 2,4'-DDT	endrin

SDG K0800270 & K0800325: The following field replicates were submitted (all parent samples were reported in SDG K0800270, all field replicates (sample ID ends in 2) and splits (ends in 3) were reported in SDG K0800325):

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C748-B1	LW3-C748-B3	LW3-C748-B2
LW3-C748-C1	LW3-C748-C3	LW3-C748-C2
LW3-C748-D1	LW3-C748-D3	LW3-C748-D2
LW3-C764-B1		LW3-C764-B2
LW3-C764-C1		LW3-C764-C2
LW3-C764-D1		LW3-C764-D2

The following outliers were noted:

Replicates	RPD > 50%	Diff > 2x RL
LW3-C748-B1 and LW3-C748-B3		heptachlor epoxide
LW3-C748-C1/C3 and LW3-C748-C2		4,4'-DDE, 4,4'-DDD, and 2,4'-DDD
LW3-C748-C1 and LW3-C748-C3		trans-chlordane, 4,4'-DDE, 4,4'-DDD, and 2,4'-DDD
LW3-C748-D1 and LW3-C748-D3		4,4'-DDE
LW3-C764-B1 and LW3-C764-B2	2,4'-DDT	
LW3-C764-C1 and LW3-C764-C2		aldrin, trans-chlordane, and 2,4'-DDD
LW3-C764-D1 and LW3-C764-D2		trans-chlordane and beta-endosulfan

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 resulting 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-3). Refer to the data validation worksheets for a detailed list of these outliers.

SDG K0710859: 50 results were estimated (J-3) and 27 results were qualified as tentatively identified (NJ-3).

SDG K0710893: 89 results were estimated (J-3) and 37 results were qualified as tentatively identified (NJ-3).

SDG K0710866: 23 results were estimated (J-3) and 14 results were qualified as tentatively identified (NJ-3).

SDG K0710893: 2 results were qualified as tentatively identified (NJ-3).

SDG K0711067: 50 results were estimated (J-3) and 14 results were qualified as tentatively identified (NJ-3).

SDG K0711172: 46 results were estimated (J-3) and 25 results were qualified as tentatively identified (NJ-3).

SDG K0711174: 65 results were estimated (J-3) and 45 results were qualified as tentatively identified (NJ-3).

SDG K0711175: 33 results were estimated (J-3) and 18 results were qualified as tentatively identified (NJ-3).

SDG K0711220: 51 results were estimated (J-3) and 26 results were qualified as tentatively identified (NJ-3).

SDG K0711252: 28 results were estimated (J-3) and 25 results were qualified as tentatively identified (NJ-3).

SDG K0711320: 12 results were estimated (J-3) and three results were qualified as tentatively identified (NJ-3).

SDG K0711423: 55 results were estimated (J-3) and 44 results were qualified as tentatively identified (NJ-3).

SDG K0711577: 12 results were estimated (J-3) and eight results were qualified as tentatively identified (NJ-3).

SDG K0711706: 33 results were estimated (J-3) and 29 results were qualified as tentatively identified (NJ-3).

SDG K0711709: 19 results were estimated (J-3) and 18 results were qualified as tentatively identified (NJ-3).

SDG K0711828: 10 results were estimated (J-3) and nine results were qualified as tentatively identified (NJ-3).

SDG K0711830: 41 results were estimated (J-3) and 34 results were qualified as tentatively identified (NJ-3).

SDG K0712050: 30 results were estimated (J-3) and 39 results were qualified as tentatively identified (NJ-3).

SDG K0712106: 21 results were estimated (J-3) and 16 results were qualified as tentatively identified (NJ-3).

SDG K0712101: 14 results were estimated (J-3) and 12 results were qualified as tentatively identified (NJ-3).

SDG K0712149: 11 results were estimated (J-3) and 11 results were qualified as tentatively identified (NJ-3).

SDG K0800158 (field blanks): One result was estimated (J-3) and one result was qualified as tentatively identified (NJ-3).

SDG K0800158 (sediment): 70 results were estimated (J-3) and 58 results were qualified as tentatively identified (NJ-3).

SDG K0800270: 38 results were estimated (J-3) and 17 results were qualified as tentatively identified (NJ-3).

SDG K0800325: 32 results were estimated (J-3) and 29 results were qualified as tentatively identified (NJ-3).

SDG K0800450: 59 results were estimated (J-3) and 37 results were qualified as tentatively identified (NJ-3).

SDG K0800487: 49 results were estimated (J-3) and 34 results were qualified as tentatively identified (NJ-3).

SDG K0800516: 31 results were estimated (J-3) and 22 results were qualified as tentatively identified (NJ-3).

Reporting Limits

Several reporting limits (RLs) were elevated by the laboratory due to interferences or dilution analyses. In some cases the elevated RL was greater than the QAPP specified method reporting limit (MRL).

Calculation Verification

SDGs K0711172, K0712101, K0800325, & K0800516: Calculation verifications were performed on these 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, MS/MSD, and LCS/LCSD %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the RPD values for the LCS/LCSD, MS/MSD, and field replicate analyses, with the exceptions noted above.

Data were estimated due to LCS and MS/MSD %R outliers and MS/MSD RPD outliers. Data were qualified as estimated and/or estimated and tentatively identified because the confirmation criteria were not met. Data were qualified as not detected due to contamination in the associated laboratory blanks. Field replicate outliers were noted.

Data were rejected due to very low (<10%) MS/MSD %R values. Rejected data should not be used for any purpose.

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

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3B Sediments
PCB Aroclors by Method SW8082

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

SDG	Number of Samples	Validation Level
K0710859	20 Sediment	Summary
K0710866	12 Sediment	Summary
K0710893	24 Sediment & 2 Equipment Blank	Summary
K0711067	19 Sediment	Summary
K0711172	20 Sediment	Summary
K0711174	21 Sediment & 1 Equipment Blank	Full
K0711175	12 Sediment & 2 Equipment Blank	Summary
K0711220	23 Sediment & 1 Equipment Blank	Summary
K0711252	19 Sediment & 2 Equipment Blank	Summary
K0711320	4 Sediment	Summary
K0711423	27 Sediment & 2 Equipment Blank	Summary
K0711577	4 Sediment	Summary
K0711706	19 Sediment & 1 Equipment Blank	Summary
K0711709	10 Sediment	Full
K0711828	13 Sediment & 2 Equipment Blank	Summary
K0711830	20 Sediment	Summary
K0712050	15 Sediment & 1 Equipment Blank	Summary
K0712101	9 Sediment	Full
K0712106	9 Sediment & 1 Equipment Blank	Summary
K0712149	6 Sediments	Summary
K0800158	28 Sediment & 2 Equipment Blank	Summary
K0800270	21 Sediment & 1 Equipment Blank	Summary
K0800325	13 Sediment & 1 Equipment Blank	Full
K0800450	27 Sediment	Summary
K0800487	22 Sediment	Summary
K0800516	14 Sediment & 1 Equipment Blank	Full

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

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

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

- | | | | |
|---|--|---|--|
| 2 | Holding Times and Sample Receipt
Instrument Performance Check
Initial Calibration (ICAL) | 2 | Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
Laboratory Control Samples (LCS) |
| 1 | Continuing Calibration (CCAL)
Laboratory Blanks | 1 | Field Replicates |
| 1 | Field Blanks | 1 | Reporting Limits |
| 2 | Surrogate Compounds | 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

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

SDG K0711174: Several of the sample names used by the laboratory did not match those on the chain of custody. No explanation was provided, however the sample names used by the laboratory match the sample names in Table A6-1 of the QAPP, with the exception of LW3-G641-1. No action was taken, other than to note the discrepancy.

Chain-of-Custody Name	Laboratory Name
LW3-633	LW3-G633
LW3-634	LW3-G634
LW3-635	LW3-G635
LW3-636	LW3-G636
LW3-639	LW3-G639
LW3-641	LW3-G641-1

SDG K0711709: Samples LW3-C606-B, LW3-C606-C, and LW3-C610-B were analyzed one day outside the 40 day holding time for sample extracts. All positive results and reporting limits were estimated (J/UJ-1) in these samples.

SDG K0711830: Ten samples were analyzed 41 days after extraction, one day outside the holding time for extracts. All positive results and reporting limits for these samples were estimated (J/UJ-1). A summary of outliers is provided in the data validation worksheets.

Continuing Calibration (CCAL)

SDG K0711172: The percent difference (%D) values for decachlorobiphenyl and Aroclors 1016 and 1260 were greater than the upper control limit for the primary column (DB-3MS) in one CCAL. As the %D values for these analytes were within the control limits on the secondary column (DB-XLB), no qualifiers were required.

SDG K0711830: The %D values for Aroclors 1016 and 1260 were greater than the upper control limit for the primary column (DB-3MS) in one CCAL. As the %D values for these analytes were within the control limits on the secondary column (DB-XLB), no qualifiers were required.

Field Blanks

The following field (equipment rinsate) blanks were analyzed with the sediment samples. No target analytes were detected in any field blank.

SDG K0710893: Two equipment blanks were submitted, LW3-SAG901 and LW3-UG901.

SDG K0711174: One equipment blank was submitted, LW3-SAG902.

SDG K0711175: Two equipment blanks were submitted, LW3-SAG903 and LW3-SAG904.

SDG K0711220: One equipment blank was submitted, LW3-MG901.

SDG K0711252: Two equipment blanks were submitted, LW3-SAG905 and LW3-UG906.

SDG K0711423: Two equipment blanks were submitted, LW3-SAG907 and LW3-SAG908.

SDG K0711706: One equipment blank was submitted, LW3-MC901.

SDG K0711828: Two equipment blanks were submitted, LW3-SAC901 and LW3-SAC902.

SDG K0712050: One equipment blank was submitted, LW3-SAC903.

SDG K0712106: One equipment blank was submitted, LW3-SAC904.

SDG K0800158: Two equipment blanks were submitted, LW3-SAC905 and LW3-SAC906.

SDG K0800270: One equipment blank was submitted, LW3-SAC907.

SDG K0800325: One equipment blank was submitted, LW3-SAC908.

SDG K0800516: One equipment blank was submitted, LW3-SAC909.

Surrogate Compounds

SDG K0800516: The percent recovery (%R) value for decachlorobiphenyl in Sample LW3-C622-C was greater than the upper control limit of 141%, at 187%. The positive results for Aroclors 1242 and 1260 were estimated (J-13) in this sample. The %R value for decachlorobiphenyl in Sample LW3-C622-D was less than the lower control limit of 33%, at 29%. Aroclors were not detected in this sample and all reporting limits were estimated (UJ-13).

Matrix Spike/Matrix Spike Duplicate

SDG K0710893: Only an matrix spike (MS) was submitted for the equipment blanks. Precision was assessed from the laboratory control sample/laboratory control sample duplicate (LCS/LCSD).

SDGs K0711174, K0711175, K0711220, K0711252, K0711423, K0711706, K0711828, K0800325: No matrix spike/matrix spike duplicate (MS/MSD) were submitted with the equipment blank analyses. Precision and accuracy were assessed from the LCS/LCSD.

SDG K0711252: The %R and relative percent difference (RPD) values for Aroclors 1016 and 1260 were outside the control limits in the batch QC MS/MSD submitted with this SDG. As the parent sample was not a part of this SDG no qualifiers were required.

SDG K0711272: The Aroclor 1260 %R values were less than the lower control limit in the MS/MSD performed on Sample LW3-G614. The Aroclor 1260 result was estimated (J-8) in the parent sample.

SDG K0800270: The %R values for Aroclor 1016 were greater than the upper control limit in the MS/MSD performed on Sample LW3-C757-C. This Aroclor was not detected in the parent sample; no qualifiers were applied.

Laboratory Control Sample/Laboratory Control Sample Duplicate

SDGs K0712050 & K0712106: The LCS %R value for Aroclor 1016 was greater than the upper control limit in the LCS/LCSD; no qualifiers were applied for this single outlier. The %R values for Aroclor 1260 were greater than the upper control limit in the LCS/LCSD; this Aroclor was not detected in the associated sample, no qualifiers were applied.

Field Replicates

Two types of field replicates were submitted, field replicates (co-located samples) and field splits. Replicate sample sets are listed below. Field splits were compared to the parent sample results. Field replicates were compared to the average of the field split and parent sample result.

The following acceptance criteria were used to evaluate precision: the RPD control limit is 50% for results greater than five times the reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and replicate must be less than two times the RL. No data

were qualified based on field replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

SDG K0710859: Samples LW3-UG03A-2 (field replicate) and LW3-UG03A-3 (field split) were submitted with parent Sample LW3-UG03A-1. Samples LW3-G637-2 (field replicate) and LW3-G637-3 (field split) were submitted with parent Sample LW3-G637-1. All RPD values and/or absolute differences met the acceptance criteria.

SDG K0710866: The data for one set of field replicates was submitted: Samples LW3-G612-1 and LW3-G612-2. The Aroclor 1254 absolute difference value was greater than the control limit. All other RPD or absolute difference values were within control limits.

SDG K0711067: Samples LW3-G684-2 (field replicate) and LW3-G684-3 (field split) were submitted with parent Sample LW3-G684-1. For the comparison of the field replicate to the average of the values from the parent and split samples, the Aroclors 1242, 1254, 1260, and 1268 RPD values were greater than control limits. For the field split, all RPD and/or absolute difference values were within control limits.

SDG K0711172: One pair of samples, LW3-G627-1 & LW3-627-2 were submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711174: Samples LW3-M002-2 (field replicate) and LW3-M002-3 (field split) were submitted with parent Sample LW3-M002-1. For the comparison of the field split to the parent sample, the Aroclor 1254 and Aroclor 1260 absolute difference values were greater than the control limit. For the field replicate, the Aroclor 1254 and Aroclor 1260 absolute difference values were greater than the control limit. All other RPD and/or absolute difference values were acceptable.

SDG K0711174 & K0711320: One pair of samples, LW3-G641-1 (submitted in SDG K0711174) & LW3-G641-2 (in K0711320) was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711175: One pair of samples, LW3-G659-1 & LW3-G659-2, was submitted as field replicates. No target analytes were detected in either sample, field precision was acceptable.

SDG K0711220: One pair of samples, LW3-UG02E-1 & LW3-UG02E-2, was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711252: One pair of samples, LW3-G732-1 & LW3-G732-2 was submitted as field replicates. No target analytes were detected in either sample, field precision was acceptable.

SDG K0711423: Samples LW3-G692-2 (field replicate) and LW3-G692-3 (field split) were submitted with parent Sample LW3-G692-1. For the comparison of the field split to the parent sample, the Aroclor 1254 absolute difference value was greater than the control limit. For the field replicate, the Aroclor 1254 and Aroclor 1260 absolute difference values were greater than the control limits. All other RPD and/or absolute difference values were within control limits.

A second field replicate set (LW3-G759-1 & LW3-G759-2) was also submitted. The RPD and/or absolute difference values were within control limits.

SDG K0711706: The data for two pairs of field replicate samples were submitted. For Samples LW3-MC001-B1 & LW3-MC001-B2 the absolute difference value for Aroclor 1254 exceeded the control limit. For Samples LW3-MC001-C1 & LW3-MC001-C2, all RPD and/or absolute difference values were within control limits.

SDG K0711709: Samples LW3-C601-B2 (field replicate) and LW3-C601-B3 (field split) were submitted with parent Sample LW3-C601-B1. The RPD and/or absolute difference values were within control limits.

SDG K0711828 & K0711830: The data for two sets of field precision samples were submitted. Samples LW3-C644-B2 (field replicate) and LW3-C644-B3 (field split) were submitted with parent Sample LW3-C644-B1, and Samples LW3-C644-C2 (field replicate) and LW3-C644-C3 (field split) were submitted with parent Sample LW3-C644-C1. No target analytes were detected in any sample, field precision was acceptable.

SDG K0800158: The data for three sets of field precision samples were submitted.

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C773-B1	LW3-C773-B3	LW3-C773-B2
LW3-C773-C1	LW3-C773-C3	LW3-C773-C2
LW3-C773-D1	LW3-C773-D3	LW3-C773-D2

The following outliers were noted:

Replicates	RPD > 50%	Diff > 2x RL
LW3-C773-B1/B3 and LW3-C773-B2	Aroclor 1254, Aroclor 1260	
LW3-C773-D1/D3 and LW3-C773-D2	Aroclor 1242	
LW3-C773-B1 and LW3-C773-B3	Aroclor 1254, Aroclor 1260	
LW3-C773-C1 and LW3-C773-C3	Aroclor 1254, Aroclor 1260	
LW3-C773-D1 and LW3-C773-D3	Aroclor 1242	

SDG K0800270 & K0800325: The following field replicates were submitted (all parent samples were reported in SDG K0800270, all field replicates (sample ID ends in 2) and splits (ends in 3) were reported in SDG K0800325):

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C748-B1	LW3-C748-B3	LW3-C748-B2
LW3-C748-C1	LW3-C748-C3	LW3-C748-C2
LW3-C748-D1	LW3-C748-D3	LW3-C748-D2
LW3-C764-B1		LW3-C764-B2
LW3-C764-C1		LW3-C764-C2
LW3-C764-D1		LW3-C764-D2

The following outliers were noted:

Replicates	RPD > 50%	Diff > 2x RL
LW3-C748-B1 and LW3-C748-B3	Aroclor 1254	
LW3-C748-B1/B3 and LW3-C748-B2	Aroclor 1254 and Aroclor 1260	

LW3-C748-C1 and LW3-C748-C3	Aroclors 1242, 1254, and 1260
LW3-C748-C1/C3 and LW3-C748-C2	Aroclors 1242, 1254, and 1260
LW3-C764-B1 and LW3-C764-B2	Aroclor 1260
LW3-C764-C1 and LW3-C764-C2	Aroclor 1260
LW3-C764-D1 and LW3-C764-D2	Aroclor 1254 and Aroclor 1260

Reporting Limits

SDGs K0710893, K0711172, K0711174, K0711175, K0711252, K0711423, K0711577, K0711706, K0711828, K0711830, K0712050, K0800158, K0800270, K0800325, K0800450, K0800487, K0800516: Several RLs were elevated by the laboratory due to interferences. In some cases the elevated RL was greater than the QAPP specified method reporting limit (MRL).

SDG K0711423: Samples LW3-G776 (5x) and LW3-G777 (10x) were analyzed at dilution. Reporting limits were elevated accordingly.

SDG K0800487: The following samples were analyzed at 5x dilutions; reporting limits were adjusted accordingly: LW3-C703-B, LW3-C703-C, LW3-C706-D, LW3-C708-B, LW3-C708-C, LW3-C708-D, LW3-C724-B, LW3-C664-B, LW3-C721-B, LW3-C721-C, and LW3-C721-D.

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 resulting 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-3). Refer to the data validation worksheets for a detailed list of these outliers.

SDG K0710859: 6 results were qualified as estimated (J-3).

SDG K0710866: 5 results were qualified as estimated (J-3) and 1 result was qualified as tentatively identified (NJ-3).

SDG K0710893: 13 results were qualified as estimated (J-3) and 2 results were qualified as tentatively identified (NJ-3).

SDG K0711067: 12 results were qualified as estimated (J-3) and 1 result was qualified as tentatively identified (NJ-3).

SDG K0711172: 17 results were qualified as estimated (J-3) and 3 results were qualified as tentatively identified (NJ-3).

SDG K0711174: 16 results were qualified as estimated (J-3) and 6 results were qualified as tentatively identified (NJ-3).

SDG K0711175: 3 results were qualified as estimated (J-3).

SDG K0711220: 12 results were qualified as estimated (J-3) and 2 results were qualified as tentatively identified (NJ-3). Positive values for Aroclor 1221 in Sample LW3-G669 and for Aroclor 1242 in Sample LW3-G668 were reported, however these Aroclors were not detected on the confirmation column. When a result is not confirmed the Aroclor should be reported as not-detected, for this reason these Aroclors in these samples were qualified as not detected (U-14).

SDG K0711252: 1 result was qualified as estimated (J-3).

SDG K0711320: 1 result was qualified as estimated (J-3) and 1 result was qualified as tentatively identified (NJ-3).

SDG K0711423: 6 results were qualified as estimated (J-3) and 2 results were qualified as tentatively identified (NJ-3).

SDG K0711577: 2 results were qualified as estimated (J-3) and 2 results were qualified as tentatively identified (NJ-3).

SDG K0711706: 7 results were qualified as estimated (J-3) and 4 results were qualified as tentatively identified (NJ-3).

SDG K0711709: 8 results were qualified as estimated (J-3) and 2 results were qualified as tentatively identified (NJ-3).

SDG K0712101: 5 results were qualified as estimated (J-3).

SDG K0711828: 1 result was qualified as estimated (J-3) and 1 result was qualified as tentatively identified (NJ-3).

SDG K0711830: 5 results were qualified as estimated (J-3) and 20 results were qualified as tentatively identified (NJ-3).

SDG K0712050: 16 results were qualified as estimated (J-3) and 3 results were qualified as tentatively identified (NJ-3).

SDG K0712106: 12 results were qualified as estimated (J-3) and 1 result was qualified as tentatively identified (NJ-3).

SDG K0712149: 4 results were qualified as estimated (J-3).

SDG K0800158: 33 results were qualified as estimated (J-3) and 1 result was qualified as tentatively identified (NJ-3).

SDG K0800270: 12 results were qualified as estimated (J-3) and 2 results were qualified as tentatively identified (NJ-3).

SDG K0800325: 7 results were qualified as estimated (J-3).

SDG K0800450: 8 results were qualified as estimated (J-3) and 5 results were qualified as tentatively identified (NJ-3).

SDG K0800487: 12 results were qualified as estimated (J-3) and 1 result was qualified as tentatively identified (NJ-3).

SDG K0800516: 1 result was qualified as tentatively identified (NJ-3).

Calculation Verification

SDGs K0711174, K0711709, K0712101, K0800325, K0800516: Calculation verifications were performed on these SDG. No calculation errors were found.

SDG K0800325: For the calibration RRF summary forms, the headings for the RRF values were mis-labeled, incorrectly identifying the column that the RRF values were associated with. The RRF values were verified from the raw data, and the correct RRF values were used in the calculations. No further action was taken.

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 RPD values from the LCS/LCSD, MS/MSD, and field replicates, with the exceptions noted above.

Data were estimated due to surrogate and MS/MSD %R outliers and holding time violations. Data were qualified as estimated and/or estimated and tentatively identified because the confirmation criteria were not met. Data were qualified as not detected due to lack of confirmation.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
R3B Sediments
PCB Congeners by EPA Method 1668A

This report documents the review of analytical data from the analyses of sediment samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Vista Analytical Laboratory, Inc., El Dorado Hills, California.

SDG	Number of Samples	Validation Level
29972	16 Sediment & 2 Equipment Blank	Summary
30010	19 Sediment	Summary
30011	6 Sediment & 3 Equipment Blank	Full
30050	14 Sediment & 5 Equipment Blank	Summary
30051	18 Sediment	Summary
30092	22 Sediment & 2 Equipment Blank	Summary
30131	18 Sediment & 1 Equipment Blank	Summary
30164	31 Sediment	Full
30187	26 Sediment	Summary

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

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

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

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

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

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

Holding Times and Sample Receipt

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

Laboratory Blanks

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times the concentration detected in the blank. If a contaminant is detected in an associated field sample and the concentration is less than the action level, the result is qualified U-7 at the reported concentration to indicate an elevation of the reporting limit. No action is taken if the sample result is greater than the action level, or for non-detected results.

Laboratory (method) blanks were analyzed at the appropriate frequency. Various target analytes were detected in all method blanks. The detected analytes, concentrations, associated samples, and action levels are documented in the data validation worksheets. However, only the following analytes required qualification in one or more samples in the associated laboratory data sets:

SDG 29972: PCB77 (two results) and PCB81 (seven results)

SDG 30010: PCB77 (two results)

SDG 30011: PCB77 (one result) and PCB81 (one result)

SDG 30050 (aqueous): PCB105 (two results) and PCB106/118 (three results)

SDG 30050 (sediment): PCB77 (two results)

SDG 30051: PCB77 (five results)

SDG 30092 (aqueous): PCB105 (one result) and PCB106/118 (one result)

SDG 30092 (sediment): PCB77 (four results)

SDG 30164: PCB77 (twelve results) and PCB81 (nine results)

SDG 30187: PCB77 (six results)

Field Blanks

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

The following analytes were detected in the field blanks. In all cases, the analytes were either not detected in the associated samples, or were present at concentrations greater than the action level. No data were qualified based on equipment rinsate blank contamination.

SDG 29972: Two equipment blanks were submitted, LW3-SAG901 & LW3-UG901. PCB77 was detected in LW3-SAG901, no target analytes were detected in LW3-UG901.

SDG 30011: Three equipment blanks were submitted. Positive results for eight PCB congeners were detected in LW3-SAG902 and for 23 PCB congeners in LW3-SAG903. A positive result for PCB77 was detected in LW3-SAG904.

SDG 30050: Five equipment blanks were submitted. PCB77 was detected in LW3-SAC905 and LW3-SAC908. Seven PCB congeners were detected in LW3-SAG906 and 11 congeners were detected in LW3-SAG907. No target analytes were detected in LW3-MG901.

SDG 30092: Two equipment blanks were submitted. PCB77 was detected in LW3-SAC901 and PCB77, PCB110, PCB156, and PCB167 were detected in LW3-SAC902.

SDG 30131: One equipment blank was submitted. PCB77 was detected in LW3-SAC903.

Matrix Spike/Matrix Spike Duplicate

No matrix spike/matrix spike duplicate (MS/MSD) sets were performed. Accuracy was assessed using labeled compound recoveries and ongoing precision and recovery (OPR) samples. Precision was assessed from the laboratory duplicates and field replicates.

Laboratory Duplicates

Duplicate sample pairs are listed below. The following acceptance criteria were applied: the relative percent difference (RPD) control limit is 50% for results greater than five times the RL. For results less than five times the reporting limit, the absolute difference between the sample and duplicate must be less than two times the RL.

Precision outliers are documented in the data validation worksheets. For precision outliers, the analyte is estimated (J-9) in the parent sample and duplicate.

SDG 29972: Duplicate analysis was performed on Sample LW3-G675. The RPD values for 59 analytes exceeded the acceptance criteria.

SDG 30010: Duplicate analysis was performed on Sample LW3-G618. The RPD values for 24 analytes exceeded the acceptance criteria.

SDG 30011: No laboratory duplicate analysis was submitted.

SDG 30050: Duplicate analysis was performed on Sample LW3-G692-2. The RPD values for 10 analytes exceeded the acceptance criteria.

SDG 30051: Duplicate analysis was performed on Sample LW3-G676. The reported values for PCB1 exceeded the acceptance criteria.

SDG 30092: Duplicate analysis was performed on Sample LW3-C644-C2. All reported values met the acceptance criteria.

SDG 30131: Duplicate analysis was performed on Sample LW3-C608-B. All reported values met the acceptance criteria.

SDG 30164: Duplicate analyses were performed on Samples LW3-C778-C and LW3-C703-D. All reported values met the acceptance criteria.

SDG 30187: Duplicate analyses were performed on Samples LW3-C708-B and LW3-C739-C. For Sample LW3-C708-B, the RPD value for PCB81 exceeded the acceptance criteria. For Sample LW3-C739-C, the RPD values for 36 analytes exceeded the acceptance criteria.

Field Replicates

Two types of field QC samples were submitted: field replicates (co-located samples) and field splits. Sample sets are listed below. Field splits were compared to the parent sample results. Field replicates were compared to the average of the field split and parent sample result.

The following acceptance criteria were used to evaluate precision: the RPD control limit is 50% for results greater than five times the RL. For results less than five times the RL, the absolute difference between the sample and replicate must be less than two times the RL. No data were qualified based on field replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

SDG 29972: Samples LW3-UG03A-2 (field replicate) and LW3-UG03A-3 (field split) were submitted with parent Sample LW3-UG03A-1. All RPD values and/or absolute differences met the acceptance criteria.

SDG 30011: Samples LW3-G684-2 (field replicate) and LW3-G684-3 (field split) were submitted with parent Sample LW3-G684-1. For Samples LW3-G684-1 and LW3-G684-3, the RPD values for 17 PCB congeners and homologues exceeded the control limit. Sample LW3-C684-2 was compared to the average results of Samples LW3-G684-1 and LW3-G684-3 and the RPD values for PCB44, PCB82, PCB97, and PCB105 exceeded the control limits.

SDG 30050: Samples LW3-G692-2 (field replicate) and LW3-G692-3 (field split) were submitted with parent Sample LW3-G692-1. All RPD values and/or absolute differences met the acceptance criteria.

SDG 30051: Two sets of field replicates were submitted. The absolute differences for PCB40 and total monochlorobiphenyls did not meet the acceptance criteria for Samples LW3-G732-1 and LW3-G732-2. All RPD values and/or absolute differences met the acceptance criteria for Samples LW3-UG02E-1 and LW3-UG02E-2.

SDG 30092: One set of sample and field replicate, LW3-C601-B1 & LW3-C601-B2, and two sets of sample, field replicate, and field split, Samples LW3-C644-B1, LW3-C644-B2, & LW3-C644-B3, and LW3-C644-C1, LW3-C644-C2, & LW3-C644-C3, were submitted. All RPD values and/or absolute differences met the acceptance criteria.

Reporting Limits

For most samples, the laboratory reported specific toxic PCB congener (PCB77, PCB81, PCB105, PCB106/118, PCB114, PCB123, PCB126, PCB156, PCB157, PCB167, PCB169, and PCB189) results using sample-specific reporting limits determined by the sample signal-to-noise ratios. A laboratory flag (*) was applied to indicate this. All other PCB congener results were reported to the method reporting limit.

SDG 29972: PCB105, PCB106/118, PCB114, PCB123, and PCB126 were reported to the sample-specific reporting limits in Sample LW3-UG901, however the laboratory did not apply the * flag to these results. No action was taken other than to note this discrepancy.

Compound Identification

The laboratory noted chemical interferences affecting the identification of one or more PCB results in several samples. These were flagged ("I") by the laboratory, and the reporting limits were raised accordingly. These reporting limits were qualified as estimated (UJ-14).

SDG 30050: Since the reporting limits for PCB77 and PCB157 were flagged "I" by the laboratory, the Total TetraCB and Total HexaCB results were also "I" flagged. However, these results are the sum of the positive results for the congeners in those chlorination ranges, and are not affected by the "I" flagged reporting limits. No action was taken.

SDG 30131: Since the reporting limit for PCB77 was flagged "I" by the laboratory, the Total TetraCB result was also "I" flagged in Sample LW3-GCA11E-C00. However, this result is the sum of the positive results for the congeners in this chlorination range, and is not affected by the "I" flagged reporting limit. No action was taken.

SDG 30187: The results for the associated homologue groups (and Total PCB) were also "I" flagged. However, these values represent a sum of the positive results for the congeners in this chlorination range, and are not affected by the "I" flagged reporting limit. No action was taken.

Calculation Verification

SDG 30011: Calculation verifications were performed on this SDG. No errors were found.

SDG 30164: The quantitation summary sheets were not included for the lowest level calibration standard. Recalculations were performed using peak areas taken from the chromatograms.

IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the labeled compound and OPR %R values. Precision was acceptable as demonstrated by the laboratory duplicate and field replicate RPD values, with the exceptions noted above.

Data were estimated due to chemical interferences and laboratory precision outliers. Data detection limits were elevated based on laboratory blank results. . Field replicate and split precision outliers were noted.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3B Sediments
Dioxin/Furan Compounds by EPA 1613 ver. B

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

SDG	Number of Samples	Validation Level
K0710859	6 Sediment	Summary
K0710866	7 Sediment	Summary
K0710893	3 Sediment & 2 Equipment Blank	Full
K0711067	1 Sediment	Summary
K0711172	6 Sediment	Summary
K0711174	3 Sediment & 1 Equipment Blank	Summary
K0711175	1 Sediment & 2 Equipment Blank	Full
K0711220	17 Sediment & 1 Equipment Blank	Summary
K0711252	3 Sediment & 2 Equipment Blank	Summary
K0711423	5 Sediment & 2 Equipment Blank	Summary
K0711828	3 Sediment & 2 Equipment Blank	Summary
K0711830	3 Sediment	Summary
K0712050	4 Sediment & 1 Equipment Blank	Summary
K0800450	13 Sediment	Summary
K0800487	4 Sediment	Summary
K0800516	4 Sediment	Full

I. DATA PACKAGE COMPLETENESS

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

The majority of the closing calibration standards from the DB-5 column were not included in the data packages. A closing calibration is not required by EPA Method 1613 version B. As all of the submitted calibration standards were acceptable no action was taken.

SDG K0800516: One of the continuing calibration (CCAL) and both of the initial calibrations (ICAL) were not included in the data package. The raw data were found in SDG K0800487 and these data were used in the validation.

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

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

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

Holding Times and Sample Receipt

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

Laboratory Blanks

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times the concentration 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.

Laboratory (method) blanks were analyzed at the appropriate frequency. Various target analytes were detected in the method blanks. The detected analytes, concentrations, associated samples, and action levels are documented in the data validation worksheets. However, however only the following analytes required qualification in one or more samples in the associated laboratory data sets:

SDG K0710893: OCDD (2 results)

SDG K0711174 (aqueous): OCDD (1 result)

SDG K0711175 (aqueous): OCDD, total heptachlorodioxins (2 results each)

SDG K0711220: OCDD (1 result EB)

SDG K0711828: 1,2,3,4,6,7,8-HpCDD (3 results), OCDD (4 results), OCDF (1 result), total heptachlorodioxins (4 results), total heptachlorofurans (1 result)

SDG K0711830: 1,2,3,4,6,7,8-HpCDD (2 results), OCDD (3 results), total heptachlorodioxins (2 results)

SDG K0712050: 1,2,3,4,6,7,8-HpCDD, OCDD, total heptachlorodioxins (1 result each)

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 K0710893: Two equipment blanks were submitted, LW3-SAG901 and LW3-UG901. No positive results remained in these blanks after qualifiers based on laboratory blanks were assigned.

SDG K0711252: Two equipment blanks were submitted, LW3-SAG905 and LW3-UG906. A positive result for OCDD was detected in both samples. No qualifiers were required since the OCDD results in the associated sediment samples exceeded the blank action levels.

SDG K0711174: One equipment blank was submitted, LW3-SAG902. No positive results remained in this blank after qualifiers based on laboratory blanks were assigned.

SDG K0711175: Two equipment blanks were submitted, LW3-SAG903 and LW3-SAG904. No positive results remained in either blank after qualifiers based on laboratory blanks were assigned.

SDG K0711220: One equipment blank was submitted, LW3-MG901. No positive results remained in this blank after qualifiers based on laboratory blanks were assigned.

SDG K0711252: Two equipment blanks were submitted, LW3-SAG905 and LW3-SAG906. Positive results for OCDD were detected in both blanks. No qualifiers were required since the results for these analytes in the associated sediment samples exceeded the blank action levels.

SDG K0711423: Two equipment blanks were submitted, LW3-SAG907 and LW3-SAG908. A positive result for OCDD was detected in both samples and a positive result for total heptachlorodioxins was detected in LW3-SAG907. No qualifiers were required since the results for these analytes in the associated sediment samples exceeded the blank action levels.

SDG K0711828: Two equipment blanks were submitted, LW3-SAC901 and LW3-SAC902. No positive results remained in either blank after qualifiers based on laboratory blanks were assigned.

SDG K0712050: One equipment blank was submitted, LW3-SAC903. No positive results remained in this blank after qualifiers based on laboratory blanks were assigned.

Labeled Compounds

SDG K0711220: The percent recovery (%R) value for $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDF was less than the lower control limit of 29%, at 21% in Sample LW3-UG02C-R. This congener was not detected in the associated sample and the reporting limit was estimated (UJ-13).

Matrix Spike/Matrix Spike Duplicate

With the exception of SDG K0710893, no matrix spike/matrix spike duplicate (MS/MSD) sets were performed. Accuracy and precision were assessed using labeled compound recoveries, ongoing precision and recovery (OPR) samples, and OPR duplicate samples.

Field Replicates

Two types of field replicates were submitted, field replicates (co-located samples) and field splits. Replicate sample sets are listed below. Field splits were compared to the parent sample results. Field replicates were compared to the average of the field split and parent sample result.

The following acceptance criteria were used to evaluate precision: the relative percent difference (RPD) control limit is 50% for results greater than five times the reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and replicate must be less than two times the RL. No data were qualified based on field replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

SDG K0710859: Samples LW3-UG03A-2 (field replicate) and LW3-UG03A-3 (field split) were submitted with parent Sample LW3-UG03A-1. For the comparison of the field split to the parent sample, the RPD value for OCDD exceeded the control limit. An absolute difference value for total hepta-dioxins exceeded the control limit. All other RPD values and/or absolute differences met the acceptance criteria.

SDG K0710866: The data for one set of field replicates was submitted: Samples LW3-G612-1 and LW3-G612-2. All RPD and/or absolute difference values were within control limits.

SDG K0711220: One pair of samples, LW3-UG02E-1 & LW3-UG02E-2, was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711423: Samples LW3-G692-2 (field replicate) and LW3-G692-3 (field split) were submitted with parent Sample LW3-G692-1. For the comparison of the field split to the parent sample, the RPD values for 1,2,3,4,6,7,8-HpCDD, OCDD, and total hepta-dioxins were greater than the control limit. For the field replicate, all RPD and/or absolute difference values were within control limits.

SDGs K0711828 & K0711830 Field Splits: Two sets of field splits were submitted. Samples LW3-C644-B1 (from SDG K0711830) and LW3-C644-B3 (from SDG K0711828) and Samples LW3-C644-C1 (from SDG K0711830) and LW3-C644-C3 (from SDG K0711828). All absolute differences met the acceptance criteria described above.

SDGs K0711828 & K0711830 Field Replicates: Two sets of field replicates were submitted. Sample LW3-LW3-C644-B2 (from SDG K0711830) was compared to the average results of Samples LW3-C644-B1 (from SDG K0711830) and LW3-C644-B3 (from SDG K0711828). Sample LW3-C644-C2 (from SDG K0711828) was compared to the average results of Samples LW3-C644-C1 (from SDG K0711830) and LW3-C644-C3 (from SDG K0711828). All absolute differences met the acceptance criteria described above.

Laboratory Duplicates

No laboratory duplicate analyses were performed. Precision was assessed from the OPR/OPR duplicate analyses.

Compound Identification

The laboratory assigned K-flags to numerous values when a peak was detected but did not meet quantitation criteria, therefore the reported values cannot be considered as positive identification for these analytes. These results were considered potential false positives or "estimated maximum possible concentrations" and were qualified as not detected (U-21) at the reported values. Laboratory blank values with K flags were considered as not detected.

All results for 2,3,7,8-TCDF were confirmed on a DB-225 column as required by the method. Although the 2,3,7,8-TCDF results from both columns were reported in the raw data, only the results from the DB-225 column were reported in the EDD. No action was necessary.

The laboratory used an "E" flag to indicate when reported results (usually OCDD or OCDF) were at concentrations greater than the linear range of the instrument calibration. These samples were usually not reanalyzed at dilutions. Since results greater than the linear range could have a potential low bias, all "E" flagged results were qualified as estimated (J-20), as noted below.

SDG K0710859: OCDD in Sample LW3-G671

SDG K0712050: 1,2,3,4,6,7,8-HpCDD and OCDD in Sample LW3-C678-B, and OCDD in Sample LW3-C678-C

SDG K0800450: OCDD in Samples LW3-C690-E and LW3-C679-D

SDG K0800487: OCDD in Sample LW3-C721-D

SDG K0800516: OCDD in Sample LW3-C739-E

Reporting Limits

The QAPP specified method reporting limits and method detection limits were not met by the laboratory for most compounds. Full details are included in the validation worksheets.

Calculation Verification

SDG K0710893, K0711175, & K0800516: Calculation verifications were performed on these SDG. No calculation errors were found.

IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the labeled compound, and OPR/OPR duplicate %R values. Precision was acceptable as demonstrated by the RPD values for the OPR/OPR duplicate, and field replicate analyses, with the above exceptions.

Data were estimated due to results exceeding the linear range of the calibration and a labeled compound recovery outlier. Data were qualified as not detected due to laboratory blank contamination and ion ratio criteria outliers. Field replicate outliers were noted.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3B Sediments
Diesel and Residual Range Hydrocarbons by Method NWTPH-Dx

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

SDG	Number of Samples	Validation Level
K0710859	20 Sediment	Summary
K0710866	12 Sediment	Summary
K0710893	24 Sediment & 2 Equipment Blank	Summary
K0711067	19 Sediment	Summary
K0711172	20 Sediment	Summary
K0711174	21 Sediment & 1 Equipment Blank	Summary
K0711175	12 Sediment & 2 Equipment Blank	Full
K0711220	23 Sediment & 1 Equipment Blank	Summary
K0711252	19 Sediment & 2 Equipment Blank	Summary
K0711320	4 Sediment	Summary
K0711423	27 Sediment & 2 Equipment Blank	Summary
K0711577	4 Sediment	Summary
K0711706	19 Sediment & 1 Equipment Blank	Summary
K0711709	10 Sediment	Full
K0711828	13 Sediment & 2 Equipment Blank	Summary
K0711830	20 Sediment	Summary
K0712050	15 Sediment & 1 Equipment Blank	Summary
K0712101	9 Sediment	Full
K0712106	9 Sediment & 1 Equipment Blank	Summary
K0712149	6 Sediment	Summary
K0800158	28 Sediment & 2 Equipment Blank	Summary
K0800270	21 Sediment & 1 Equipment Blank	Summary
K0800325	13 Sediment & 1 Equipment Blank	Full
K0800450	27 Sediment	Summary
K0800487	22 Sediment	Summary
K0800516	14 Sediment & 1 Equipment Blank	Full

I. DATA PACKAGE COMPLETENESS

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

All samples were silica gel treated by the laboratory prior to analysis. Both sets of data were reported.

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/LCSD)
	GC/MS Instrument Performance Check	2 Laboratory Duplicates
	Initial Calibration (ICAL)	1 Field Replicates
	Continuing Calibration (CCAL)	Reporting Limits
2	Laboratory Blanks	2 Compound Identification
1	Field Blanks	1 Calculation Verification
2	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

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

Laboratory Blanks

To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times the concentration 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.

Laboratory (method) blanks were analyzed at the appropriate frequency. DRO and RRO were detected in some of the method blanks. The detected analytes, concentrations, associated samples, and action levels are documented in the data validation worksheets. However, however only the following analytes required qualification in one or more samples in the associated laboratory data sets:

SDG K0710859: DRO (2 results), RRO (1 result)

SDG K0710859 (silica gel treated): DRO (2 results), RRO (1 result)

SDG K0710893: DRO (2 results), RRO (2 results)

SDG K0711172 (silica gel treated): DRO (1 result)

SDG K0711174: DRO (1 result)

SDG K0711174 (silica gel treated): DRO (1 result)
SDG K0711175: DRO (5 results)
SDG K0711175 (silica gel treated): DRO (5 results)
SDG K0711220 (silica gel treated): DRO (2 results)
SDG K0711252 (silica gel treated): DRO (1 result)
SDG K0711320: RRO (1 result)
SDG K0711320 (silica gel treated): RRO (1 result)
SDG K0711423 (silica gel treated): DRO (2 results); RRO (3 results)
SDG K0711706: RRO (1 result)
SDG K0711706 (silica gel treated): DRO (8 results); RRO (9 results)
SDG K0711709: DRO (1 result); RRO (2 results)
SDG K0711709 (silica gel treated): DRO (1 result); RRO (1 result)
SDG K0711828: RRO (10 results)
SDG K0711828 (silica gel treated): RRO (10 results)
SDG K0711830: RRO (1 result)
SDG K0711830 (silica gel treated): RRO (3 results)
SDG K0712050: DRO (1 result); RRO (3 results)
SDG K0712050 (silica gel treated): RRO (1 result)
SDG K0712106 (aqueous): DRO (1 result); RRO (1 result)
SDG K0712149 (silica gel treated): RRO (3 results)
SDG K0800158: RRO (2 results)
SDG K0800270: RRO (3 results)
SDG K0800270 (silica gel treated): RRO (3 results)
SDG K0800516: DRO (1 result) RRO (2 results)

Field Blanks

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

The field (equipment rinsate) blanks and any detected analytes are listed below. In all cases where DRO or RRO was detected, the concentrations in the associated samples were greater than the action levels, and no data were qualified based on rinsate blank contamination.

SDG K0710893: Two rinsate blanks were reported, LW3-SAG901 and LW3-UG901. After qualifiers based on method blank contamination were issued, there were no positive results for DRO or RRO in either of these blanks.

SDG K0711174: One rinsate blank was reported, LW3-SAG902. A positive result for DRO was reported in this blank.

SDG K0711175: Two rinsate blanks were reported, LW3-SAG903 & LW3-SAG904. Positive results for DRO were reported in both blanks.

SDG K0711220: One rinsate blank was reported, LW3-MG901. A positive result for DRO was reported in this blank.

SDG K0711252: Two rinsate blanks were reported, LW3-SAG905 & LW3-SAG906. Positive results for DRO and RRO were reported in the first blank, while a positive result for DRO was reported in the second blank.

SDG K0711423: Two rinsate blanks were reported, LW3-SAG907 & LW3-SAG908. Positive results for DRO and RRO were reported in the first blank, while a positive result for DRO was reported in the second blank.

SDG K0711706: One rinsate blank was reported, LW3-MC901. A positive result for DRO was reported in this blank.

SDG K0711828: Two rinsate blanks were reported, LW3-SAC901 & LW3-SAC902. Positive results for DRO were reported in both of these blanks.

SDG K0712050: One rinsate blank was reported, LW3-SAC903. After method blank contamination qualifiers were applied, there were no positive results for DRO or RRO in this sample.

SDG K0712106: One rinsate blank was reported, LW3-SAC904. After method blank contamination qualifiers were applied, there were no positive results for DRO or RRO in this sample.

SDG K0800158: Two rinsate blanks were reported, LW3-SAC905 & LW3-SAC906. Positive results for DRO were reported in both blanks.

SDG K0800270: One rinsate blank was reported, LW3-SAC907. A positive result for DRO was reported in this blank.

SDG K0800325: One rinsate blank was reported, LW3-SAC908. A positive result for DRO was reported in this blank.

SDG K0800516: One rinsate blank was reported, LW3-SAC909. A positive result for DRO was reported in this blank.

Surrogate Compounds

SDG K0800487: The percent recovery (%R) values for n-triacontane were greater than the upper control limit in Samples LW3-C708-C and LW3-C708-C (silica gel treated). The %R values for o-terphenyl were within control limits for both samples. Both samples were diluted prior to analysis. No qualifiers were applied.

SDG K0800516: The %R values for o-terphenyl and n-triacontane were greater than the upper control limits in Samples LW3-C662-C and LW3-C662-C (silica gel treated). The positive results were qualified as estimated (J-13) in both samples.

Laboratory Duplicates

The following acceptance criteria were used to evaluate precision: the relative percent difference (RPD) control limit is 30% for any results greater than the reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and replicate must be less than two times the RL. All laboratory duplicate precision was acceptable with the following exceptions:

SDG K0711174: The laboratory duplicate RPD value for DRO was greater than the control limit in Sample LW3-MG006. The DRO result was estimated (J-9) in the parent sample.

SDG K0711175: The laboratory duplicate RPD value for DRO and RRO were greater than the control limit in Sample LW3-G650. The DRO and RRO results were estimated (J-9) in the parent sample.

Field Replicates

Two types of field replicates were submitted, field replicates (co-located samples) and field splits. Replicate sample sets were always sampled in groups of three and are listed below. Field splits were compared to the parent sample results. Field replicates were compared to the average of the field split and parent sample result.

The following acceptance criteria were used to evaluate precision: the RPD control limit is 50% for results greater than five times the reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and replicate must be less than two times the RL. No data were qualified based on field replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

SDG K0710859: Samples LW3-UG03A-2 (field replicate) and LW3-UG03A-3 (field split) were submitted with parent Sample LW3-UG03A-1. Samples LW3-G637-2 (field replicate) and LW3-G637-3 (field split) were submitted with parent Sample LW3-G637-1. All RPD values and/or absolute differences met the acceptance criteria.

SDG K0710866: The data for one set of field replicates was submitted: Samples LW3-G612-1 and LW3-G612-2. All RPD and/or absolute difference values were within control limits.

SDG K0711067: Samples LW3-G684-2 (field replicate) and LW3-G684-3 (field split) were submitted with parent Sample LW3-G684-1. All RPD and/or absolute difference values were within control limits.

SDG K0711172: One pair of samples, LW3-G627-1 & LW3-627-2 were submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711174: Samples LW3-M002-2 (field replicate) and LW3-M002-3 (field split) were submitted with parent Sample LW3-M002-1. The RPD and/or absolute difference values were within control limits.

SDG K0711174 & K0711320: One pair of samples, LW3-G641-1 (submitted in SDG K0711174) & LW3-G641-2 (in K0711320) was submitted as field replicates. The absolute difference values for DRO and RRO were greater than the control limits in the regular analysis. The absolute difference value for DRO was greater than the control limit in the silica gel treated analysis.

SDG K0711175: One pair of samples, LW3-G659-1 & LW3-G659-2, was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711220: One pair of samples, LW3-UG02E-1 & LW3-UG02E-2, was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711252: One pair of samples, LW3-G732-1 & LW3-G732-2 was submitted as field replicates. The RPD and/or absolute difference values were within control limits.

SDG K0711423: Samples LW3-G692-2 (field replicate) and LW3-G692-3 (field split) were submitted with parent Sample LW3-G692-1. A second field replicate set (LW3-G759-1 & LW3-G759-2) was also submitted. The RPD and/or absolute difference values were within control limits.

SDG K0711706: The data for two field replicate sets (LW3-MC001-B1 & LW3-MC001-B2 and LW3-MC001-C1 & LW3-MC001-C2) were submitted. All RPD and/or absolute difference values were within control limits.

SDG K0711709: Samples LW3-C601-B2 (field replicate) and LW3-C601-B3 (field split) were submitted with parent Sample LW3-C601-B1. The RPD and/or absolute difference values were within control limits.

SDG K0711828 & K0711830: The data for two sets of field precision samples were submitted. Samples LW3-C644-B2 (field replicate) and LW3-C644-B3 (field split) were submitted with parent Sample LW3-C644-B1, and Samples LW3-C644-C2 (field replicate) and LW3-C644-C3 (field split) were submitted with parent Sample LW3-C644-C1. All RPD and/or absolute difference values were within control limits.

SDG K0800158: The data for three sets of field precision samples were submitted. Samples LW3-C773-B2 (field replicate) and LW3-C773-B3 (field split) were submitted with parent Sample LW3-C773-B1, Samples LW3-C773-C2 (field replicate) and LW3-C773-C3 (field split) were submitted with parent Sample LW3-C773-C1, and LW3-C773-D2 (field replicate) and

LW3-C773-D3 (field split) were submitted with parent Sample LW3-C773-D1. The absolute difference values for DRO/RRO and silica gel treated DRO/RRO were greater than the control limit for the comparison of LW3-C773-B1 to LW3-C773-B3. All other RPD and/or absolute difference values were within control limits.

SDG K0800270 & K0800325: The following field replicates were submitted (all parent samples were reported in SDG K0800270, all field replicates (sample ID ends in 2) and splits (ends in 3) were reported in SDG K0800325):

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C748-B1	LW3-C748-B3	LW3-C748-B2
LW3-C748-C1	LW3-C748-C3	LW3-C748-C2
LW3-C748-D1	LW3-C748-D3	LW3-C748-D2
LW3-C764-B1		LW3-C764-B2
LW3-C764-C1		LW3-C764-C2
LW3-C764-D1		LW3-C764-D2

The following outliers were noted: for Samples LW3-C748-C1 & LW3-C748-C3, the absolute difference values for DRO and silica gel treated DRO were greater than the control limit. All other RPD and/or absolute difference values were within control limits.

Compound Identification

For many samples, the chromatographic patterns from the samples did not match the patterns of the DRO and RRO standards used during the instrument calibration. For these samples, the laboratory reported DRO and/or RRO as detected based on elution times, but flagged the results to indicate the pattern mismatch. Since the reported concentrations may be biased high (due to interference) or low (due to weathering or other factors), the results were estimated (J-2). The following results were qualified:

SDG K0710859: DRO – 10 samples. RRO – 13 samples

SDG K0710859 (silica gel treated): DRO – 4 samples. RRO – 3 samples

SDG K0710866: DRO – 7 samples. RRO – 12 samples

SDG K0710866 (silica gel treated): DRO – 2 samples. RRO – 2 samples

SDG K0710893: DRO – 7 samples. RRO – 23 samples

SDG K0710893 (silica gel treated): DRO – 5 samples. RRO – 2 samples

SDG K0711067: DRO – 16 samples. RRO – 18 samples

SDG K0711067 (silica gel treated): DRO – 4 samples. RRO – 4 samples

SDG K0711172: DRO – 18 samples. RRO – 18 samples

SDG K0711172 (silica gel treated): DRO – 6 samples. RRO – 2 samples

SDG K0711174: DRO – 19 samples. RRO – 19 samples

SDG K0711174 (silica gel treated): DRO – 9 samples. RRO – 7 samples

SDG K0711175: DRO – 7 samples. RRO – 5 samples
SDG K0711175 (silica gel treated): DRO – 4 samples. RRO – 1 sample
SDG K0711220: DRO – 15 samples. RRO – 17 samples
SDG K0711220 (silica gel treated): DRO – 2 samples. RRO – 2 samples
SDG K0711252: DRO – 13 samples. RRO – 18 samples
SDG K0711252 (silica gel treated): DRO – 1 sample. RRO – 2 samples
SDG K0711320: DRO – 2 samples. RRO – 2 samples
SDG K0711320 (silica gel treated): DRO – 2 samples. RRO – 1 sample
SDG K0711423: DRO – 19 samples. RRO – 23 samples
SDG K0711423 (silica gel treated): DRO – 11 samples. RRO – 10 samples
SDG K0711577: DRO – 3 samples. RRO – 4 samples
SDG K0711577 (silica gel treated): DRO – 3 samples. RRO – 3 samples
SDG K0711706: DRO – 11 samples. RRO – 9 samples
SDG K0711706 (silica gel treated): DRO – 9 samples. RRO – 9 samples
SDG K0711709: DRO – 8 samples. RRO – 8 samples
SDG K0711709 (silica gel treated): DRO – 7 samples. RRO – 7 samples
SDG K0711828: DRO – 4 samples. RRO – 2 samples
SDG K0711828 (silica gel treated): DRO – 2 samples. RRO – 1 sample
SDG K0711830: DRO – 13 samples. RRO – 13 samples
SDG K0711830 (silica gel treated): DRO – 13 samples. RRO – 10 samples
SDG K0712050: DRO – 13 samples. RRO – 13 samples
SDG K0712050 (silica gel treated): DRO – 13 samples. RRO – 13 samples
SDG K0712101: DRO – 4 samples. RRO – 4 samples
SDG K0712101 (silica gel treated): DRO – 4 samples. RRO – 4 samples
SDG K0712106: DRO – 9 samples. RRO – 8 samples
SDG K0712106 (silica gel treated): DRO – 9 samples. RRO – 8 samples
SDG K0712149: DRO – 3 samples. RRO – 3 samples
SDG K0712149 (silica gel treated): DRO – 3 samples. RRO – 2 samples
SDG K0800158: DRO – 25 samples. RRO – 27 samples
SDG K0800158 (silica gel treated): DRO – 22 samples. RRO – 23 samples
SDG K0800270: DRO – 8 samples. RRO – 8 samples
SDG K0800270 (silica gel treated): DRO – 8 samples. RRO – 8 samples

SDG K0800325: DRO – 10 samples. RRO – 10 samples

SDG K0800325 (silica gel treated): DRO – 10 samples. RRO – 10 samples

SDG K0800450: DRO – 21 samples. RRO – 21 samples

SDG K0800450 (silica gel treated): DRO – 20 samples. RRO – 19 samples

SDG K0800487: DRO – 15 samples. RRO – 15 samples

SDG K0800487 (silica gel treated): DRO – 15 samples. RRO – 13 samples

SDG K0800516: DRO – 13 samples. RRO – 12 samples

SDG K0800516 (silica gel treated): DRO – 13 samples. RRO – 12 samples

Calculation Verification

SDGs K0711175, K0711709, K0712101, K0800325, & K0800516: Calculation verifications were performed on these SDGs. 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 and laboratory control sample %R values, with the exception noted above. Precision was also acceptable as demonstrated by the laboratory duplicate and field replicate RPD values, with the exceptions noted above.

Data were estimated based on chromatographic pattern mismatches, surrogate recovery outliers, and laboratory duplicate RPD outliers. Data were also qualified as not detected based on contamination in the associated method blank.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3B Sediments
Metals by Methods SW6010, SW6020, and SW7470/7471A

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

SDG	Number of Samples	Validation Level
K0710859	20 Sediment	Summary
K0710866	12 Sediment	Summary
K0710893	24 Sediment & 2 Equipment Blank	Summary
K0711067	19 Sediment	Summary
K0711172	20 Sediment	Summary
K0711174	21 Sediment & 1 Equipment Blank	Summary
K0711175	12 Sediment & 2 Equipment Blank	Full
K0711220	23 Sediment & 1 Equipment Blank	Summary
K0711252	19 Sediment & 2 Equipment Blank	Summary
K0711320	4 Sediment	Summary
K0711423	27 Sediment & 2 Equipment Blank	Summary
K0711577	4 Sediment	Summary
K0711706	19 Sediment & 1 Equipment Blank	Summary
K0711709	10 Sediment	Full
K0711828	13 Sediment & 2 Equipment Blank	Summary
K0711830	20 Sediment	Summary
K0712050	15 Sediment & 1 Equipment Blank	Summary
K0712101	9 Sediment	Full
K0712106	9 Sediment & 1 Equipment Blank	Summary
K0712149	6 Sediment	Summary
K0800158	28 Sediment & 2 Equipment Blank	Summary
K0800270	21 Sediment & 1 Equipment Blank	Summary
K0800325	13 Sediment & 1 Equipment Blank	Full
K0800450	27 Sediment	Summary
K0800487	22 Sediment	Summary
K0800516	14 Sediment & 1 Equipment Blank	Full

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

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

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

- | | | | |
|---|---|---|---|
| 1 | Technical Holding Times and Sample Preservation | 2 | Laboratory Duplicates |
| | Initial Calibration | | Interference Check Samples |
| | Calibration Verification | 2 | Serial Dilutions |
| 2 | CRDL Standards | 2 | ICPMS Internal Standards |
| 2 | Laboratory Blanks | 1 | Field Replicates |
| 2 | Field Blanks | 1 | Reported Results |
| | Laboratory Control Samples | 1 | Calculation Verification (Full validation only) |
| 2 | Matrix Spike 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

Several coolers were received at the laboratory with internal temperatures outside the recommended temperature range of 4°C ±2° (the range of recorded temperature was -0.9° to 7.9 °C). The temperature outliers did not impact data quality and no data were qualified.

Contract Required Detection Limit Standard

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

SDG K0710893: Nickel – high bias

SDG K0712101: Copper – high bias

SDGs K0711706, K071182, K0712050 & K0712106 (aqueous): Silver – low bias (R-14).

SDG K0800158: Antimony, copper – high bias

Laboratory Blanks

Various analytes were detected in the method and instrument blanks at levels greater than the method detection limits (MDL). To evaluate the effect on the sample data, action levels of five

times 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.

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

The following analytes were qualified in one or more samples based on laboratory blanks:

SDG K0710893: *aqueous:* chromium - low bias (J-7)

SDG K0711174: *aqueous:* aluminum, antimony, cadmium, nickel, silver, zinc - not detected (U-7)
sediment: cadmium – not detected (U-7)

SDG K0711175: *aqueous:* nickel - not detected (U-7); nickel-low bias (J-7)
sediment: cadmium – not detected (U-7)

SDG K0711220: *aqueous:* zinc - not detected (U-7); aluminum, chromium - low bias (UJ-7)

SDG K0711252: *aqueous:* aluminum - not detected (U-7); arsenic - low bias (UJ-7)

SDG K0711423: *aqueous:* aluminum, nickel, zinc - not detected (U-7)

SDG K0711706: *aqueous:* zinc - not detected (U-7)

SDG K0711828: *aqueous:* arsenic, mercury - low bias (UJ-7)

SDG K0712050: *aqueous:* aluminum, zinc - not detected (U-7); mercury - low bias (UJ-7)

SDG K0712101: *sediment:* antimony - low bias (J-7)

SDG K0712106: *aqueous:* zinc - not detected (U-7); mercury - low bias (UJ-7)

SDG K0800158: *aqueous:* aluminum, chromium, copper, lead, silver, zinc - not detected (U-7);
arsenic - low bias (UJ-7)

SDG K0800270: *aqueous:* aluminum, chromium, copper, lead, silver, zinc - not detected (U-7);
mercury - low bias (UJ-7)

SDG K0800325: *aqueous:* mercury - low bias (UJ-7)
sediment: cadmium – not detected (U-7)

SDG K0800450: *sediment:* cadmium – not detected (U-7), antimony – low bias (J-7)

SDG K0800516: *aqueous:* lead – not detected (U-7); copper – low bias (J-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). Field blanks and outliers are noted below. With the exception of the field blank in SDG K0800325, the concentrations in the associated samples were greater than the action levels and no data were qualified based on field blank contamination.

SDG K0710893: Two field blanks, LW3-UG901 & LW3-SAG901, were submitted. After qualifiers based on laboratory blank contamination were issued, results remained for the following analytes:

- LW3-UG901: aluminum, chromium, copper, lead, nickel, silver, and zinc
- LW3-SAG901: aluminum, chromium, copper, nickel, and zinc

SDG K0711174: One field blank, LW3-SAG902, was submitted. After qualifiers based on laboratory blank contamination were issued, a positive result remained for chromium.

SDG K0711175: Two field blanks, LW3-SAG903 & LW3-SAG904, were submitted. After qualifiers based on laboratory blank contamination were issued, results remained for the following analytes:

- LW3-SAG903 - aluminum, chromium, copper, lead, nickel, zinc
- LW3-SAG904 - aluminum, chromium, copper, lead, zinc

SDG K0711220: One field blank, LW3-MG901, was submitted. After qualifiers based on laboratory blank contamination were issued, positive results remained for aluminum and copper.

SDG K0711252: Two field blanks, LW3-SAG905 & LW3-SAG906, were submitted. Selenium was reported for these samples, although it is not a target analyte. After qualifiers based on laboratory blank contamination were issued, results remained for the following analytes:

- LW3-SAG905 - aluminum, chromium, copper, lead, nickel, zinc
- LW3-SAG906 - chromium, copper, lead, nickel, zinc

SDG K0711423: Two field blanks, LW3-SAG907 & LW3-SAG908, were submitted. After qualifiers based on laboratory blank contamination were issued, positive results remained for the following analytes:

- LW3-SAG907: chromium, copper, lead, and zinc
- LW3-SAG908: chromium and copper

SDG K0711706: One field blank, LW3-MC901, was submitted. After qualifiers based on laboratory blank contamination were issued, positive results remained for the following analytes: aluminum, chromium, copper, and nickel.

SDG K0711828: Two field blanks, LW3-SAC901 & LW3-SAC902, were submitted. After qualifiers based on laboratory blank contamination were issued, the following positive results remained:

- LW3-SAC901 - aluminum, chromium, copper, lead, nickel, zinc
- LW3-SAC902 - aluminum, chromium, copper, nickel, zinc

SDG K0712050: One field blank, LW3-SAC903, was submitted. After qualifiers based on laboratory blank contamination were issued, positive results remained for chromium and nickel.

SDG K0712106: One field blank, LW3-SAC904, was submitted. After qualifiers based on laboratory blank contamination were issued, positive results remained for the following analytes: aluminum, chromium, copper, lead, and nickel.

SDG K0800158: Two field blanks, LW3-SAC905 & LW3-SAC906, were submitted. After qualifiers based on laboratory blank contamination were issued, the following positive results remained:

- LW3-SAC905 - aluminum, zinc
- LW3-SAC906 - cadmium, chromium

SDG K0800270: One field blank, LW3-SAC907, was submitted. After qualifiers based on laboratory blank contamination were issued, no positive results remained.

SDG K0800325: One field blank, LW3-SAC908, was submitted. After qualifiers based on laboratory blank contamination were issued, results remained for chromium, aluminum, copper, lead, nickel, and zinc. The chromium result (85.6 µg/L) was high enough so that the action level was greater than the concentration in all associated samples. All chromium results were qualified as not detected at the reported concentration (U-6).

SDG K0800516: One field blank, LW3-SAC909, was submitted. After qualifiers based on laboratory blank contamination were issued, positive results remained for the following analytes: aluminum, chromium, copper, nickel, and zinc.

Matrix Spike Samples

A matrix spike sample (MS) was 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 laboratory control limits, with the exceptions noted below. For antimony and chromium, the default control limits of 70%-130% were used to evaluate the matrix spike recoveries. For %R values greater than the upper control limit, the associated positive results were estimated (J-8) to indicate a possible high bias. No action was taken for non-detects. For %R values less than the lower control limit, the associated positive results and non-detects were qualified as estimated (J/UJ-8) to indicate a possible low bias. The following outliers were noted:

SDG K0710859: QC Sample LW3-G788 - antimony (54.9%) – low bias.

SDG K0710866: QC Sample LW3-G612-1 – antimony (32%) – low bias

SDG K0711067: QC Sample LW3-G763 – antimony (33%) – low bias

SDG K0710893: QC Sample LW3-G674 – antimony (44.0%) – low bias
QC Sample LW3-UG03C – antimony (27.0%) – low bias

SDG K0711172: QC Sample LW3-G624 – antimony (26.6%) – low bias
QC Sample LW3-G614 – antimony (27.0%) – low bias

SDG K0711174: QC Sample LW3-MG006 – antimony (44.1%) – low bias
Batch QC1 – antimony (66.1%) – low bias

SDG K0711175: QC Sample LW3-G650 – antimony (66.2%) – low bias

SDG K0711220: QC Sample LW3-G669 - antimony (48.0%)– low bias
QC Sample LW3-G681B – antimony (30.9%) – low bias

SDG K0711252: QC Sample LW3-G697 – antimony (30.1%) – low bias

SDG K0711320: Batch QC Sample – antimony (30.9%) – low bias

SDG K0711423: QC Sample LW3-G717 – antimony (33.4%) – low bias
QC Sample LW3-G613 – nickel (78.3%), antimony (33.6%) – low bias

SDG K0711577: QC Sample LW3-G783 – antimony (40.0%) – low bias

SDG K0711706: QC Sample LW3-MC006-B – antimony (32.0%) – low bias;
QC Sample LW3-C604-B – antimony (26.0%) – low bias

SDG K0711709: QC Sample LW3-C609-B – antimony (29.6%) – low bias

SDG K0711828: QC Sample LW3-C644-C2 – antimony (55.8%) – low bias

SDG K0711830: QC Sample LW3-C615-B – antimony (29.1%) – low bias
QC Sample LW3-C634-B – antimony (26.7%) – low bias

SDG K0712101: Batch QC Sample – antimony (50.9%), mercury (67.8%) – low bias

SDG K0712149: Batch QC Sample – antimony (22.0%) – low bias

SDG K0712050: QC Sample LW3-C704-B – antimony (36.9%) – low bias

SDG K0712106: QC Sample LW3-C714-B – antimony (31.4%) – low bias

SDG K0800158: QC Sample LW3-C760-C – antimony (37.6%) – low bias
QC Sample LW3-C773-B3 – antimony (37.4%) – low bias

SDG K0800270: QC Sample LW3-C757 – antimony (36.2%) – low bias
QC Sample LW3-C779 – antimony (48.7%) – low bias

SDG K0800325: QC Sample LW3-C748-D2 – antimony (50.5%) – low bias

SDG K0800450: QC Sample LW3-C782-A – antimony (55.2%) – low bias
QC Sample LW3-C679-B– antimony (29.3%) – low bias

SDG K0800487: QC Sample LW3-C703-D – antimony (41.0%) – low bias
Batch QC Sample – antimony (37.4%) – low bias

SDG K0800516: QC Sample LW3-C738-B – antimony (31.7%) – low bias

Laboratory Duplicates

Laboratory duplicate relative percent difference (RPD) values were used to evaluate precision. The RPD values were within the control limit of 30% for sample results greater than five times the reporting limit (for results less than five times the reporting limit, the difference was less than twice the reporting limit) with the exceptions noted below.

For RPD or difference values exceeding the control limits, associated positive results and non-detects were qualified as estimated (J/UJ-9).

SDG K0710893: QC Sample LW3-G674 - arsenic (32.0%)

SDG K0711067: QC Sample LW3-G763 - lead (34%)

SDG K0711172: QC Sample LW3-G614 - chromium (20.2%); parent only qualified

SDG K0711577: QC Sample LW3-G783 - mercury (39.6)

SDG K0711706: QC Sample LW3-MC006-B - arsenic (32%); parent sample only qualified

SDG K0711709: QC Sample LW3-C609-B - silver (30.4%)

SDG K0712101: Batch QC Sample - cadmium (57.1%)

SDG K0712050: QC Sample LW3-C704-B - mercury (44.8%)

SDG K0800270 (sediment): QC Sample LW3-C757 - mercury (33.7%)
Batch QC – mercury (44.3%)

SDG K0800270 (aqueous): QC Sample LW3-SAC907 - lead (diff > RL)

SDG K0800325: QC Sample LW3-C783-B – mercury (44.9%)
QC Sample LW3-C748-D2 - aluminum (30.8%)

SDG K0800487: Batch QC Sample - antimony (diff > 2x RL)

Field Replicates

Two types of field replicates were submitted, field replicates (co-located samples) and field splits. Field splits were compared to the parent sample results. Field replicates were compared to the average of the field split and parent sample results, or in the absence of a split sample, just the parent sample result.

The following acceptance criteria were used to evaluate precision: the RPD control limit is 50% for results greater than five times the reporting limit (RL). For results less than five times the RL, the difference between the sample and replicate must be less than two times the RL.

No data were qualified based on field replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

SDG K0710859: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-G637-1	LW3-G637-3	LW3-G637-2
LW3-UG03A-1	LW3-UG03A-3	LW3-UG03A-2

All field precision criteria were met.

SDG K0710866: One set of field replicates, Samples LW3-G612-1 and LW3-G612-2 were submitted with this SDG. All field precision criteria were met.

SDG K0711067: One set of field replicates and splits, Samples LW3-G684-1, LW3-G684-2, and LW3-G684-3, were submitted with this SDG. All field precision criteria were met.

SDG K0711172: One set of field replicates, Samples LW3-G627-1 and LW3-G627-2, were submitted with this SDG. All field precision criteria were met.

SDG K0711174: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-MG002-1	LW3-MG002-3	LW3-MG002-2

All field precision criteria were acceptable.

SDG K0711175: One set of field replicates, Samples LW3-G732-1, and LW3-G732-2, were submitted with this SDG. All field precision criteria were met.

SDG K0711220: One set of field replicates, Samples LW3-UG02E-1 and LW3-UG02E-2, were submitted with this SDG. All field precision criteria were met.

SDG K0711252: One set of field replicates, Samples LW3-G659-1 and LW3-G659-2, were submitted with this SDG. All field precision criteria were met.

SDG K0711320: Field duplicate sample LW3-G641-2 was submitted in this SDG. The parent sample, LW3-G641-1, was submitted in SDG K0711174. The RPD values for cadmium (54.1%), mercury (71.4%), and silver (76.2%) exceeded the control limit.

SDG K0711423: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-G759-1		LW3-G759-2
LW3-G692-1	LW3-G692-3	LW3-G692-2

All field precision criteria were met.

SDG K0711706: Two sets of field replicates were submitted with this SDG: LW3-MC001-B1 & LW3-MC001-B2 and LW3-MC001-C1 & LW3-MC001-C2. All field precision criteria were met.

SDG K0711709: One set of field replicates and splits were submitted: LW3-C601-B1, LW3-C601-B2 & LW3-C601-B3. All field precision criteria were met.

SDGs K0711828 & K0711830: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C644-B1	LW3-C644-B3	LW3-C644-B2
LW3-C644-C1	LW3-C644-C3	LW3-C644-C2

All field precision criteria were met.

SDG K0800158: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C773-B1	LW3-C773-B3	LW3-C773-B2
LW3-C773-C1	LW3-C773-C3	LW3-C773-C2
LW3-C773-D1	LW3-C773-D3	LW3-C773-D2

The following outliers were noted:

Replicates	RPD > 50%	Diff > 2x RL
LW3-C773-B1 and LW3-C773-B3	lead, silver	mercury
LW3-C773-C1 and LW3-C773-C3	lead	
LW3-C773-D1 and LW3-C773-D3	cadmium, zinc	

SDG K0800270 & K0800325: The following field replicates were submitted (all parent samples were reported in SDG K0800270, all field replicates (sample ID ends in 2) and splits (ends in 3) were reported in SDG K0800325):

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C748-B1	LW3-C748-B3	LW3-C748-B2
LW3-C748-C1	LW3-C748-C3	LW3-C748-C2
LW3-C748-D1	LW3-C748-D3	LW3-C748-D2
LW3-C764-B1		LW3-C764-B2
LW3-C764-C1		LW3-C764-C2
LW3-C764-D1		LW3-C764-D2

The following outliers were noted:

Replicates	RPD > 50%	Diff > 2x RL
LW3-C748-C1 and LW3-C748-C3	lead, silver	cadmium, mercury
LW3-C748-C1/C3 and LW3-C748-C2	antimony, lead	
LW3-C748-D1 and LW3-C748-D3	lead, zinc	antimony, nickel, silver
LW3-C748-D1/D3 and LW3-C748-D2	lead	antimony
LW3-C764-C1 and LW3-C764-C2	lead	
LW3-C764-D1 and LW3-C764-D2		mercury

ICP-MS Internal Standards

SDG K0711172: For the analyses performed on January 30, 2008, the recovery for the internal standard Lutetium 175 was greater than the upper control limit of 125% for Sample LW3-G618. The lead result was qualified as estimated (J-19) in this sample.

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:

SDG K0710859: LW3-G671 - copper (13%), lead (10.3%), nickel (14%)

SDG K0710866: Batch QC - cadmium (15%)

SDG K0711172: LW3-G624 - cadmium (21%)

SDG K0711220: LW3-G669 - chromium (15%)

SDG K0711252: LW3-G697 - cadmium (20%)

SDG K0711830: LW3-C634-B - zinc (12.4%)

SDG K0712106: LW3-C714-B – nickel (18.0%), copper (13.0%), lead (11.0%), zinc (11.8%)

SDG K0800516: LW3-C738-B – nickel (12%)

Reported Results

The concentrations for aluminum and zinc were often high enough to necessitate quantification by EPA method 6010B (ICP) rather than EPA method 6020 (ICP-MS)

SDGs K0710893, K0711220, K0711320 & K0711577: The QAPP calls for method reporting limits (MRL) of 2.0 mg/kg for aluminum, the laboratory reports 2.5 mg/kg for this analyte.

SDG K0711067: The QAPP calls for MRL of 2.0 mg/kg for aluminum, the laboratory reports 2.5 mg/kg for this analyte.

The laboratory analyzed more analytes than listed on the COC, cadmium and lead were analyzed for all samples.

SDG K0711174: The QAPP calls for MRL of 2.0 mg/kg for aluminum, the laboratory reports 7.0 mg/kg and 10.0 mg/kg for this analyte.

SDG K0712101: The QAPP calls for MRL of 2.0 mg/kg for aluminum, 0 and 0.5 mg/kg for zinc. The laboratory reports 5 mg/kg and 2.0 mg/kg respectively for these analytes.

SDGs K0711172, K0711175 & K0711252: The QAPP calls for MRL of 2.0 mg/kg for aluminum, 0.1 mg/kg for arsenic and 0.5 mg/kg for zinc. The laboratory reports 10.0 mg/kg, 0.5 mg/kg, and 2.0 mg/kg respectively for these analytes.

SDGs K0711706, K0711709, K0711828, K0711830, K0712050, K0712106, K0712149, K0800158, K0800270, K0800325 & K0800450: The QAPP calls for MRL of 2.0 mg/kg for aluminum and 0.5 mg/kg for zinc. The laboratory reports 5.0 mg/kg and 1.0 mg/kg respectively for these analytes.

SDGs K0800270 & K0800325: For the field blank analyses, two sets of arsenic results were reported, one from a standard digestion and one from a pre-concentrated digestion used to obtain a lower detection limit. The laboratory control sample recovery for the pre-concentration prep was greater than the upper control limit due to an apparent spiking error. The MRL from the standard digestion met the QAPP requirements, therefore these results should be used. Results from the pre-concentrated analyses were labeled do-not-report (DNR-11).

Calculation Verification

SDGs K0711175, K0711709, K0712101, K0800325 & K0800516: Several results were verified by recalculation from the raw data. No calculation or transcription errors were found.

IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the laboratory control sample and MS recoveries, with the exceptions noted above. Precision was also acceptable as demonstrated by the RPD values for the laboratory duplicate and field replicate analyses, with the exceptions previously noted.

Data were rejected based on CRDL % R outliers. Data were labeled DNR to indicate which result should be used from multiple reported results.

Data were estimated based on CRDL % R, MS %R, laboratory duplicate RPD, serial dilution %D, and internal standard %R outliers. Data were qualified as not detected based on blank contamination; data were estimated based on low bias indicated by negative blank results.

Data that has been rejected or labeled DNR should not be used for any purpose. All other data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Portland Harbor RI/FS
Round 3B Sediments
General Chemistry Parameters

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

SDG	Number of Samples	Validation Level
K0710859	20 Sediment	Summary
K0710866	12 Sediment	Summary
K0710893	24 Sediment	Summary
K0711067	19 Sediment	Summary
K0711172	20 Sediment	Summary
K0711174	21 Sediment	Summary
K0711175	12 Sediment	Full
K0711220	23 Sediment	Summary
K0711252	19 Sediment	Summary
K0711320	4 Sediment	Summary
K0711423	27 Sediment	Summary
K0711577	4 Sediment	Summary
K0711706	19 Sediment	Summary
K0711709	10 Sediment	Full
K0711828	13 Sediment	Summary
K0711830	20 Sediment	Summary
K0712050	15 Sediment	Summary
K0712101	9 Sediment	Full
K0712106	9 Sediment	Summary
K0712149	6 Sediment	Summary
K0800158	28 Sediment	Summary
K0800270	21 Sediment	Summary
K0800325	13 Sediment	Full
K0800450	27 Sediment	Summary
K0800487	22 Sediment	Summary
K0800516	14 Sediment	Full

The analytical tests that were performed are summarized below:

Parameter	Method
Total Solids	160.3
Total Organic Carbon (TOC)	PSEP
Grain Size	PSEP
Specific Gravity	ASTM D854
Ammonia as Nitrogen*	350.1
Total Sulfide*	9030

* SDG K0710859, K0710866, K0710893, K0711067, K0711172, and K0711423 only

I. DATA PACKAGE COMPLETENESS

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

II. EDD TO HARDCOPY VERIFICATION

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

III. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

2	Holding Times and Sample Preservation	Matrix Spike (MS)
	Initial Calibration	2 Laboratory Replicates
	Calibration Verification	1 Field Replicates
1	Laboratory Blanks	Reporting Limits
	Field Blanks	1 Calculation Verification (Full validation only)
	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 -0.9° to 7.9°C. These temperature outliers did not impact data quality and no qualifiers were required.

SDG K0710859, K0710866, and K0711067: All samples were analyzed for ammonia and sulfide beyond the 7 day holding time. The samples were extracted within the holding time, therefore no action was taken.

SDG K0710893: All samples were analyzed for ammonia and sulfide beyond the 7 day holding time. All of the ammonia and some of the sulfide samples were extracted within the holding time, therefore no action was taken.

Sixteen samples were extracted and analyzed for sulfide beyond the 7 day holding time; these results were qualified as estimated (J/UJ-1).

SDG K0711172: All samples were analyzed for ammonia beyond the 7 day holding time, however they were extracted within 7 days so no action was taken. All samples were extracted and analyzed for sulfide beyond the 7 day holding time; these results were estimated (J/UJ-1).

SDG K0711174: Sample LW3-MG006 was analyzed for TOC four days past the 28 day holding time. The TOC result for this sample was estimated (J-1).

SDG K0800516: TOC and total solids analyses were not requested for Samples LW3-C738-B through LW3-C651-C on the COC. All samples were analyzed for these two parameters. No corrective action was necessary.

Laboratory Blanks

SDG K0800450: The total organic carbon (TOC) concentration for one continuing calibration blank was greater than the method detection limit (MDL). To evaluate the effect on the sample data, an action level of five times (5x) the blank concentration was established. Associated results were greater than the action limit; no qualification of data was necessary.

Laboratory Replicates

Laboratory duplicate samples were performed at the frequency of one per batch. The relative percent difference (RPD) values were within the control limit of 20%, except as noted below. (Note: for grain size, the laboratory performed triplicate analyses. A percent relative standard deviation [%RSD] limit of 20% was used for all fractions with sieve size results of at least 5%). For RPD outliers, all associated samples are qualified (J/UJ-9). If the %RSD value for a grain size fraction was greater than the control limit, the results for the parent sample, duplicate, and triplicate were qualified (J-9).

The following outliers were noted:

SDG K0710859: Sample LW3-UG03A-3 - ammonia (27%)

SDG K0711172: Sample LW3-G614 - coarse silt (34%), >9 phi clay (39%)

SDG K0711706: Sample LW3-MC006-B - fine silt (21%)

SDG K0711830: Sample LW3-C634-B - very fine silt (39%)

SDG K0800158: Sample LW3-C760-C – fine gravel (27%), medium silt (38%), very fine silt (32%), >9 phi clay (22%)

SDG K0800325: Sample LW3-C748-C2 - coarse silt (71.2%), medium silt (32.5%)

SDG K0800450: Sample LW3-C782-C - medium gravel (25%)

SDG K0800487: Sample LW3-C706-C - 8-9 phi clay (22%)
Sample LW3-C708-B - coarse silt (20.2%), 8-9 phi clay (24%)

Field Replicates

Two types of field replicates were submitted, field replicates (co-located samples) and field splits. Field splits were compared to the parent sample results. Field replicates were compared to the average of the field split and parent sample results, or in the absence of a split sample, just the parent sample result.

The following acceptance criteria were used to evaluate precision: the RPD control limit is 50% for results greater than five times the reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and replicate must be less than two times the RL. No data were qualified based on field replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

SDG K0710859: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-G637-1	LW3-G637-3	LW3-G637-2
LW3-UG03A-1	LW3-UG03A-3	LW3-UG03A-2

All precision criteria were met for Samples LW3-G637-1, LW3-G637-2, and LW3-G637-3.

Although the precision was acceptable between Samples LW3-UG03A-2 and LW3-UG03A-3, the results for the parent sample (LW3-UG03A-1) were not consistent with the results from the split and duplicate samples. The RPD values for medium sand, medium silt, and fine silt were greater than the control limit for the field replicate and average of the field split/parent sample. The RPD values for medium sand through very fine silt, and > 9 phi clay were greater than the control limits for samples LW3-UG03A-1 and LW3-UG03A-3.

SDG K0710866: One set of field replicates, Samples LW3-G612-1 and LW3-G612-2, were submitted with this SDG. All field precision criteria were met.

SDG K0711067: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-G684-1	LW3-G684-3	LW3-G684-2

For sulfide, the differences between the sample and field split, and between the field replicate and the average of the sample and split were greater than twice the RL.

SDG K0711172: One set of field replicates, Samples LW3-G627-1 and LW3-G627-2, were submitted. The RPD value for fine silt was greater than 50%. Also, the difference between the parent and duplicate results for sulfide was greater than two times the reporting limit.

SDG K0711174: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-MG002-1	LW3-MG002-3	LW3-MG002-2

The RPD value for clay > 9Phi was greater than the control limit for Samples LW3-MG002-1 and LW3-MG002-3.

SDG K0711175: One set of field replicates, Samples LW3-G659-1 and LW3-G659-2, were submitted with this SDG. All field precision criteria were met.

SDG K0711220: One set of field replicates, Samples LW3-UG02E-1 and LW3-UG02E-2, were submitted with this SDG. All field precision criteria were met.

SDG K0711252: One set of field replicates, Samples LW3-G732-1 and LW3-G732-2, were submitted with this SDG. All field precision criteria were met.

SDG K0711320: Field duplicate sample LW3-G641-2 was submitted in this SDG. The parent sample, LW3-G641-1, was submitted in SDG K0711174. The RPD values for total organic carbon (53.9%), course sand (154.2%), medium sand (146.0%), very fine sand (64.8%), coarse silt (99.6%), medium silt (75.3%), fine silt (74.0%), 8-9 phi clay (136.4%), and >9 phi clay (81.0%) exceeded the control limit.

SDG K0711423: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-G759-1		LW3-G759-2
LW3-G692-1	LW3-G692-3	LW3-G692-2

All field precision criteria were met.

SDG K0711706: Two sets of field replicates were submitted with this SDG: LW3-MC001-B1 & LW3-MC001-B2 and LW3-MC001-C1 & LW3-MC001-C2. All field precision criteria were met.

SDG K0711709: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C601-B1	LW3-C601-B3	LW3-C601-B2

All field precision criteria were met.

SDGs K0711828 & K0711830: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C644-B1	LW3-C644-B3	LW3-C644-B2
LW3-C644-C1	LW3-C644-C3	LW3-C644-C2

All field precision criteria were met.

SDG K0800158: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C773-B1	LW3-C773-B3	LW3-C773-B2
LW3-C773-C1	LW3-C773-C3	LW3-C773-C2
LW3-C773-D1	LW3-C773-D3	LW3-C773-D2

The following outliers were noted:

Replicates	RPD > 50%
LW3-C773-B1 and LW3-C773-B3	fine sand, TOC
LW3-C748-B1/B3 and LW3-C748-B2	course silt
LW3-C773-D1 and LW3-C773-D3	coarse silt, medium sand

SDGs K0800270 & K0800325: The following field replicates were submitted:

Parent Sample ID	Split Sample ID	Replicate Sample ID
LW3-C748-B1	LW3-C748-B3	LW3-C748-B2
LW3-C748-C1	LW3-C748-C3	LW3-C748-C2
LW3-C748-D1	LW3-C748-D3	LW3-C748-D2
LW3-C764-B1		LW3-C764-B2
LW3-C764-C1		LW3-C764-C2
LW3-C764-D1		LW3-C764-D2

The following outliers were noted:

Replicates	RPD > 50%	Diff > 2x RL
LW3-C748-C1 and LW3-C748-C3	coarse sand, fine sand, medium silt, very fine sand	TOC
LW3-C748-C1/C3 and LW3-C748-C2	coarse sand, coarse silt, fine sand, TOC	
LW3-C748-D1/D3 and LW3-C748-D2		TOC
LW3-C764-B1 and LW3-C764-B2	coarse sand, fine silt, medium sand, very fine sand	
LW3-C764-C1 and LW3-C764-C2	coarse silt, fine silt, medium sand, medium silt, very fine sand, TOC	
LW3-C764-D1 and LW3-C764-D2	medium sand	

Calculation Verification

SDGs K0711175, K0711709, K0712101, K0800325, & K0800516: Several results were verified by recalculation from the raw data. No calculation errors were found.

SDG K0800516: The value for fine gravel for Sample LW3-C651-C Triplicate was incorrect in the EDD. The result was corrected and no further action was taken.

IV. OVERALL ASSESSMENT

As determined by this evaluation, the laboratory followed the specified analytical methods. The laboratory replicate RPD and %RSD values indicated acceptable precision, with the exceptions noted above. Accuracy was also acceptable, as demonstrated by the matrix spike and laboratory control sample recoveries.

Data were qualified based on exceeded holding times and laboratory replicate % RSD outliers.

All data, as qualified, are acceptable for use.

ROUND 3B SEDIMENTS
Validation of Additional PAH Results

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ACRONYMS AND ABBREVIATIONS

GIS	geographic information system
SOQ	statement of qualification
VOC	volatile organic compound
PAH	polycyclic aromatic hydrocarbon
QAPP	quality assurance project plan
EcoChem	EcoChem Inc.
Integral	Integral Consulting Inc.
SDG	sample delivery group
QA	quality assurance
QC	quality control
GC/MS	gas chromatography/mass spectrometry
ICAL	initial calibration
CCAL	continuing calibration
MS/MSD	matrix spike/matrix spike duplicate
LCS/LCSD	laboratory control sample/laboratory control sample duplicate
MDL	method detection limit
MRL	method reporting limit
EDD	electronic data deliverable
RSD	relative standard deviation
%R	percent recovery
RPD	relative percent difference

1 INTRODUCTION

Sediment samples analyzed for alkylated polynuclear aromatic hydrocarbons (PAHs) were intended to have benzo(e)pyrene and perylene included in the target compound list; however, these compounds were inadvertently omitted from the quality assurance project plan (QAPP) and therefore were not reported by the laboratory. The laboratory, Columbia Analytical Services, includes these compounds in their calibration mixtures and was able to reprocess the data files and provide results for these compounds. The data were reported in separate data packages containing results for only these two compounds.

The initial alkylated PAH data was validated by EcoChem Inc (EcoChem). In order to expedite the validation of this additional data, the validation was performed in-house by Integral Consulting Inc (Integral). Where possible the results of the validation performed by EcoChem were used so as to not duplicate effort. The results of this validation apply only to the benzo(e)pyrene and perylene data.

The sample delivery groups (SDGs) reviewed are listed below:

SDG	Number of Samples	Validation Level
K0710859	20 Sediment Grabs	Summary
K0710866	12 Sediment Grabs	Summary
K0710893	24 Sediment Grabs	Summary
K0711067	19 Sediment Grabs	Summary
K0711172	20 Sediment Grabs	Summary
K0711174	21 Sediment Grabs	Summary
K0711175	12 Sediment Grabs	Full
K0711220	23 Sediment Grabs	Summary
K0711252	19 Sediment Grabs	Summary
K0711320	4 Sediment Grabs	Summary
K0711423	27 Sediment Grabs	Summary
K0711577	4 Sediment Grabs	Summary
K0711706	6 Sediment Cores	Summary
K0711709	9 Sediment Cores	Full
K0711828	11 Sediment Cores	Summary
K0711830	17 Sediment Cores	Summary
K0712050	7 Sediment Cores	Summary
K0712101	6 Sediment Cores	Full
K0712106	8 Sediment Cores	Summary

SDG	Number of Samples	Validation Level
K0712149	2 Sediment Cores	Summary
K0800158	13 Sediment Cores	Summary
K0800270	7 Sediment Cores	Summary
K0800325	10 Sediment Cores	Summary
K0800450	12 Sediment Cores	Summary
K0800487	14 Sediment Cores	Summary
K0800516	8 Sediment Cores	Full

2 BASIS OF VALIDATION

The data validations performed by both EcoChem and Integral used guidance specified by *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (EPA 1999) and *Guidance on Environmental Data Verification and Validation* (EPA 2002). The method used for the PAH analysis, SW8270C-SIM, is a non-Contract Laboratory Program method and modifications to the guidance documents were made to reflect the quality assurance/quality control (QA/QC) requirements of method.

3 PARAMETERS REVIEWED BY ECOCHEM

The data reported in the additional data packages were the results of reprocessing the electronic files from the PAH analyses. Therefore, information pertaining to sample receipt and sample extraction applies to the initial data as well as the additional data. The QC parameters assessed from the EcoChem data validation reports are listed below.

QC Parameter	Finding
Data Package Completeness	Acceptable
Sample Receipt and Chain-of-Custody	See discussion below
Holding Times	Acceptable
GC/MS Instrument Performance Checks	Acceptable
Surrogate Compounds	See discussion below

3.1 SAMPLE RECEIPT AND HOLDING TIMES

EcoChem noted that many of the sample coolers were received by the laboratory with temperatures slightly outside the control limits of 2°C to 6°C. Cooler temperatures at the time of receipt ranged from -0.8 to 7.9°C. EcoChem found *“These temperature outliers did not impact data quality and no action was taken.”* No qualifiers were applied to the additional PAH data for cooler receipt temperatures.

3.2 SURROGATE COMPOUNDS

K08000516: EcoChem noted several surrogate outliers; however, all outliers were the result of sample dilution and no qualifiers were required. No qualifiers were applied to the additional PAH data for surrogate results.

4 PARAMETERS REVIEWED BY INTEGRAL CONSULTING

The QC parameters reviewed by Integral are listed below. When qualifiers were assigned by EcoChem a reason code was also assigned to indicate which QC parameter resulted in the qualifier. The same reason codes used by EcoChem were used for the additional PAH data in order to keep the data validation process consistent.

QC Parameter	Finding
Initial Calibration (ICAL)	Acceptable
Continuing Calibration (CCAL)	Acceptable
Laboratory Blanks	Qualifiers issued, see discussion below
Matrix Spikes/Matrix Spike Duplicates (MS/MSD)	Qualifiers issued, see discussion below
Laboratory Control Samples (LCS/LCSD)	See discussion below
Field Replicates	See discussion below
Internal Standards	Acceptable
Target Compound List	Acceptable
Reporting Limits (MDL and MRL)	Qualifier issued, see discussion below
Compound Identification	Acceptable
Calculation Verification (Full Validation only)	Acceptable
EDD Verification – 100% of results	Acceptable

4.1 LABORATORY BLANKS

K0711220, K0712101, K0711320, K0711577, & K0800516: Benzo(e)pyrene was detected in the laboratory blanks associated with these SDGs. Benzo(e)pyrene was either not detected in the associated samples or the concentration was greater than 5 times the blank concentration and no qualifiers were required.

K0711423: Benzo(e)pyrene was detected in the laboratory blank for batch KWG0713541. The concentration of benzo(e)pyrene in LW3-G779 was less than 5 times the blank concentration and was qualified as not-detected (U-7).

K0711830: Benzo(e)pyrene was detected in the laboratory blank for batch KWG0714018. The concentrations of benzo(e)pyrene in LW3-C636-B and LW3-C644-B2 were less than 5 times the blank concentration and were qualified as not-detected (U-7).

K0800450: Benzo(e)pyrene was detected in the laboratory blank for batch KWG0800660. The concentration of benzo(e)pyrene in LW3-C645-C was less than 5 times the blank concentration and was qualified as not-detected (U-7).

4.2 MATRIX SPIKES/MATRIX SPIKE DUPLICATES (MS/MSD)

The spiking solution used by the laboratory for the MS/MSD for most SDGs did not contain benzo(e)pyrene or perylene. For these PAHs, PAH compounds with the same molecular weight (252.32) and similar structure to benzo(e)pyrene and perylene were used to assess precision and accuracy. These PAH compounds are benzo(b)fluoranthene, benzo(k)fluoranthene, and benzo(a)pyrene. Unless otherwise noted below, the recoveries of these PAHs were acceptable.

In addition, the unspiked MS/MSD constituted triplicate analyses of the samples for benzo(e)pyrene and perylene. The concentrations of these PAHs in the parent sample, MS, and MSD were evaluated against control limits of 40% relative standard deviation (RSD) for results greater than 5 times the method reporting limit (MRL). For results less than 5 times the MRL, the absolute difference between results should be less than twice the MRL. Unless otherwise noted below, precision was acceptable.

K0711175: Benzo(e)pyrene and perylene were not included in the spiking solution as described above. EcoChem noted that the percent recoveries (%R) for benzo(b)fluoranthene and benzo(a)pyrene were greater than the upper control limits in either the MS or the MSD, but not both, and no qualifiers were assigned to benzo(e)pyrene or perylene.

K0711423: The %RSD for benzo(e)pyrene in the original sample and the MS and MSD samples (i.e., without spiked benzo(e)pyrene) was greater than the control limit in the MS/MSD performed on LW3-G717. The result for benzo(e)pyrene was estimated (J-9) in this sample.

K0711577: Benzo(e)pyrene and perylene were not included in the spiking solution as described above. EcoChem noted several %R outliers, and benzo(k)fluoranthene was estimated on this basis. The %R values for benzo(b)fluoranthene and benzo(a)pyrene were less than the lower control limits; however, the concentrations of these compounds in the parent sample, LW3-G783, were greater than 4 times the amount spiked and no qualifiers were assigned because these spike results were not meaningful. Similarly, no qualifiers were assigned to benzo(e)pyrene and perylene based on recoveries. The %RSD for both benzo(e)pyrene and perylene were greater than 40%, and the results for these compounds were estimated (J-9) in LW3-G783 based on precision outliers.

K0712106: The %RSD for benzo(e)pyrene was greater than the control limit in the MS/MSD performed on LW3-C714-B. The result for benzo(e)pyrene was estimated (J-9) in this sample.

K0800158: The absolute difference for benzo(e)pyrene was greater than the control limit in the MS/MSD performed on LW3-C760-C. The result for benzo(e)pyrene was estimated (J-9) in this sample.

K0800325: The absolute differences for benzo(e)pyrene and perylene were greater than the control limit in the MS/MSD performed on LW3-C748-D2. The results for these compounds were estimated (J-9) in this sample.

K0800450: The absolute difference for perylene was greater than the control limit in the MS/MSD performed on LW3-C645-C. The result for perylene was estimated (J-9) in this sample.

K08000516: Benzo(e)pyrene and perylene were not included in the spiking solution as described above. EcoChem noted that the recoveries for all compounds were outside the control limits but that the concentrations of all compounds in the parent sample, LW3-C662-D, were greater than 4 times the amount spiked, and no qualifiers were required. The concentrations of benzo(e)pyrene and perylene were similarly not qualified based on spike recoveries. The %RSDs for both benzo(e)pyrene and perylene were greater than 40%, and the results for these compounds were estimated (J-9) in LW3-C662-D based on precision outliers.

K0711828, K0712101, & K0712149: No MS/MSDs were submitted with these SDG due to limited sample volumes. Laboratory accuracy and precision were assessed from the LCS/LCSD analyses.

K0711709, K0712050, & K0800270: The MS/MSD analyses for these SDG were not evaluated as they were performed on samples that were not analyzed for alkylated PAHs. For organics analyses, qualifiers based on MS/MSD outliers are only assigned to the parent sample and therefore an evaluation of the MS/MSD would not have provided meaningful information.

4.3 LABORATORY CONTROL SAMPLES (LCS/LCSD)

As noted above, the spiking solution used by the laboratory for most SDGs did not contain benzo(e)pyrene or perylene, and precision and accuracy were assessed based on the recoveries of PAH compounds with similar molecular weight and structure.

K0710866: The %R for perylene in the LCSD was less than the lower control limit. The LCS %R was acceptable and no qualifiers were assigned.

K0710893: The %R for perylene in the LCSD for batch KWG0712902 was less than the lower control limit. The LCS %R was acceptable and no qualifiers were assigned.

K0711828: Benzo(e)pyrene and perylene were not spiked. EcoChem noted multiple %R outliers in the LCSD; however, no qualifiers were assigned because no systematic performance problems were evident.

4.4 FIELD REPLICATES

Field replicates (identified by the suffix “-2” in the sample ID) and field splits (identified by the suffix “-3” in the sample ID) were submitted. The parent sample (identified by the suffix of “1” in the sample ID) was compared to the field replicate and field replicates were compared to the field split using the following criteria: the relative percent difference (RPD) for results greater than 5 times the MRL should be less than 50%; for results less than 5 times the MRL the absolute difference should be less than 2 times the MRL. The RPD values are provided in Table D-8 of Appendix D of this report.

Data are not qualified based on field precision outliers.

K07010859: Samples LW3-G637-1, LW3-G637-2, & LW3-G637-3 and LW3-UG03A-1, LW3-UG03A-2, & LW3-UG03A-3 were submitted as field replicates/splits and the above control limits were met.

K0710866: Samples LW3-G612-1 and LW3-G612-2 were submitted as replicates and the above control limits were met.

K0711067: Samples LW3-G684-1, LW3-G684-2, and LW3-G684-3 were submitted as field replicates/splits and the above control limits were met.

K0711172: Samples LW3-G627-1 and LW3-G627-2 were submitted as replicates and the above control limits were met.

K0711174 & K0711320: Samples LW3-MG002-1, LW3-MG002-2, and LW3-MG002-3 were submitted as field replicates/splits. The relative percent difference (RPD) value for perylene was greater than the control limit between the parent and replicate samples. Samples LW3-G641-1 and LW3-G641-2 were submitted as replicates, and the RPD values for benzo(e)pyrene and perylene were greater than the control limit.

K0711175: Samples LW3-G659-1 and LW3-G659-2 were submitted as replicates and absolute difference of benzo(e)pyrene exceeded the control limit.

K0711220: Samples LW3-UG02E-1 and LW3-UG02E-2 were submitted as replicates and the above control limits were met.

K0711252: Samples LW3-G732-1 and LW3-G732-2 were submitted as replicates and the above control limits were met.

K0711423: Samples LW3-G692-1, LW3-G692-2, and LW3-G692-3 were submitted as field replicates/splits. The RPD values for benzo(e)pyrene and perylene were greater than the control limit between the replicate and split samples. Samples LW3-G759-1 and LW3-G759-2 were

submitted as replicates, and the RPD values for benzo(e)pyrene and perylene were greater than the control limit .

K0711709: Samples LW3-C601-B1, LW3-G601-B2, and LW3-C601-B3 were submitted as field replicates/splits and the above control limits were met.

K0711828 & K0711830: Samples LW3-C644-B1, LW3-G644-B2, LW3-C644-B3 and LW3-C644-C1, LW3-G644-C2, & LW3-C644-C3 were submitted as field replicates/splits and the above control limits were met.

K0800270 & K0800325: The following samples in this SDG were field replicates or splits:

- LW3-C748-B1, LW3-C748-B2, LW3-C748-B3
- LW3-C748-C1, LW3-C748-C2, LW3-C748-C3
- LW3-C748-D1, LW3-C748-D2, LW3-C748-D3.

The RPD values for benzo(e)pyrene and perylene were greater than the control limit between samples LW3-C748-C1 and LW3-C748-C2.

4.5 METHOD REPORTING LIMITS (MRL)

There are no method reporting limits (MRLs) listed in the QAPP for benzo(e)pyrene or perylene. The MRL of 5 µg/kg for the other target PAHs was met. Some samples were analyzed at dilution, resulting in elevated reporting limits. Both benzo(e)pyrene and perylene were detected in all samples analyzed at dilution, and therefore the elevated reporting limits have no effect on data quality.

K0711174: The result for benzo(e)pyrene in LW3-G639 was greater than the linear range of the calibration and no dilution was performed. This result was estimated (J-20).

5 SUMMARY

The results are acceptable for use as qualified.

6 REFERENCES

EPA. 1999. USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review. EPA-540/R-99/008. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC.

EPA. 2002. Guidance on Environmental Data Verification and Data Validation. EPA QA/G-8. EPA/240/R-02/004. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, DC.