

# APPENDIX F. WATER CHEMISTRY VALIDATION REPORT

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**EcoChem, INC.**  
Environmental Data Quality

**DATA QUALITY EVALUATION  
PORTLAND HARBOR  
LAMPREY AMMOCOETE TOXICITY STUDY**

**Prepared for:**

Integral Consulting  
7900 SE 28<sup>th</sup> Street, Suite 300  
Mercer Island, Washington 98040

Integral Project: B01-01-58C

**Prepared by:**

EcoChem, Inc.  
710 Second Avenue, Suite 660  
Seattle, Washington 98104

EcoChem Project: C22119-1

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**Approved for Release:**

  
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Eric Strout  
Project Manager/Technical Director  
**EcoChem, Inc.**

# 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)* (Integral 2004); *Addendum 7: Round 3 Chemical Analysis of Lamprey Ammonoete Toxicity Test Water* (Integral 2006), and *National Functional Guidelines for Organic and/or Inorganic Data Review* (USEPA 1994, 1999 & 2002).

Data qualifier definitions, reason codes, and validation criteria are included as **Appendix A**. 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 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, herbicide, phenol fractions)
- Detection limits
- Instrument performance (initial calibration, continuing calibration, tuning, sensitivity and degradation)

The 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:

The following numbers of water samples were analyzed for aniline, pentachlorophenol, gamma-BHC (Lindane), diazinon, naphthalene, copper, and general chemistry parameters. The water samples represented different concentrations of these analytes at different time periods after dosing, as part of the lamprey ammocoete toxicity test study. Columbia Analytical Services (CAS) completed the analyses.

| Analysis               | EPA Method    | Number of Water Samples |
|------------------------|---------------|-------------------------|
| Aniline                | 8270C         | 31                      |
| Pentachlorophenol      | 8151 modified | 32                      |
| Gamma-BHC (Lindane)    | 8081A         | 35                      |
| Diazinon               | 8141A         | 32                      |
| Naphthalene            | 8260B         | 63                      |
| Copper                 | 200.8         | 64                      |
| Total Suspended Solids | SM 2540D      | 19                      |
| Total Organic Carbon   | 415.1         | 19                      |
| Ammonia (as N)         | SM 4500       | 19                      |

In addition, one soil sample (NAS#1135G Waupaca Materials) was analyzed for chlorinated herbicides and pesticides as part of this study. CAS also completed these analyses.

The data for the water samples were generally acceptable. Seven results (2.3% of all water 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 data for the soil sample were acceptable with the following exception. One result for dinoseb (2.6% of soil results) was rejected. Rejected data must not be used for any 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 water samples, and were greater than 97% complete for the soil sample.

### Holding Times and Sample Preservation

All samples were extracted and analyzed within the specified holding times.

## **Instrument Performance**

### ***Calibrations***

Initial and continuing calibrations were completed at the proper frequency. No qualifiers were required due to calibration outliers.

### ***Endrin/DDT Breakdown***

Breakdown evaluation mixtures were completed with the pesticide analyses at the proper frequency to measure percent breakdown. All percent breakdown values met the acceptance criteria.

## **Laboratory 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.

Copper, ammonia and total organic carbon (TOC) were detected in laboratory blanks. One result for copper, three results for ammonia, and two results for TOC (2.0% of all water results) were qualified as not detected based on laboratory blank contamination.

Gamma-BHC (Lindane) and diazinon were also detected in laboratory blanks. The concentrations in the associated samples were greater than the action levels. No qualifiers were necessary. All other laboratory blanks were free of contamination.

## **Accuracy**

### ***Surrogate Compound Recovery***

Surrogate compounds were added to all required samples. The surrogates were either not recovered or the recovery values were outside the control limits in the analyses for gamma-BHC (Lindane) and naphthalene. No qualifiers were required for surrogate outliers. All other surrogate recovery values were acceptable.

### ***Matrix Spike Recovery***

Matrix spike (MS) analyses were not performed with some data sets. For these sets, laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses were used to assess accuracy and precision. One reporting limit for dinoseb (2.6% of all soil results) was rejected with potential very low bias in the associated soil sample, based on an MS outlier. Also, the water MS recovery value was outside the control limits in the analysis for pentachlorophenol. No qualifier was required for this MS outlier. All other MS recovery values were acceptable.

### ***Laboratory Control Sample Recovery***

LCS/LCSD analyses met the criteria for frequency of analysis. One reporting limit for dinoseb (2.6% of all soil results) was rejected with potential very low bias in the associated soil sample, based on an LCS outlier. All other LCS/LCSD recovery values were acceptable.

## **Precision**

LCS/LCSD and MS/MSD analyses were evaluated for laboratory precision. Six results for gamma-BHC (Lindane) (2.0% of all water results) were estimated, based on an LCS/LCSD precision outlier. All other relative percent difference (RPD) values were acceptable.

## **Compound Identification**

The results from the two analytical columns (as appropriate per the analytical method) were compared for agreement. 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). If the RPD value was greater than 60%, the result was qualified as a tentative identification (NJ). One result for diazinon (0.33% of all water results) was qualified as estimated (J).

## **Method Detection Limits and Method Reporting Limits**

With the exception of the copper analyses, most of the samples were analyzed at dilution factors ranging from 5x to 50,000x. Detection/reporting limits were adjusted accordingly.

## **Field Quality Control Samples**

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

### ***Trip Blanks***

One trip blank was associated with the naphthalene analyses. No target analytes were detected in the trip blank.

### ***Field Replicate Samples***

Replicate water samples were received and analyzed. For Samples NAS# 1369G Day 3 – 4.0 mg/L Lindane and NAS#1371G Day 3 – 4.0 mg/L Lindane Dupl, the RPD value exceeded the acceptance criterion, at 81.4%. All other replicate precision values were acceptable.



**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.

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|    |                                                                                                                                                                                                                                                                           |
|----|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 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

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|    |                                                                                   |
|----|-----------------------------------------------------------------------------------|
| 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                                                         |

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

| VALIDATION QC ELEMENT                               | ACCEPTANCE CRITERIA                                                                     | ACTION                                                                                                                                                                                | REASON CODE |
|-----------------------------------------------------|-----------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------|
| Cooler/Storage Temperature and Preservation         | Aqueous/Soil/Sediment <4°C<br>Tissues <-10°C<br>Water: HCl to pH < 2                    | J(+)/UJ(-) if greater than 6 deg. C (EcoChem PJ)                                                                                                                                      | 1           |
| Hold Time                                           | Waters: 14 days preserved<br>7 Days: unpreserved (for aromatics)<br><br>Solids: 14 Days | J(+)/UJ(-) if hold times exceeded<br>If exceeded by > 3X HT: J(+)/R(-) (EcoChem PJ)                                                                                                   | 1           |
| Tuning                                              | BFB<br>Beginning of each 12 hour period<br>Method acceptance criteria                   | R(+/-) all analytes in all samples associated with the tune                                                                                                                           | 5A          |
| Initial Calibration (Minimum 5 stds.)               | RRF > 0.05                                                                              | J(+)/R(-) if RRF < 0.05                                                                                                                                                               | 5A          |
|                                                     | %RSD < 30%                                                                              | (EcoChem PJ, see TM-06)<br>J(+) if %RSD > 30%                                                                                                                                         | 5A          |
| Continuing Calibration (Prior to each 12 hr. shift) | RRF > 0.05                                                                              | (EcoChem PJ, see TM-06)<br>If MDL= reporting limit:<br>J(+)/R(-) if RRF < 0.05<br><br>If reporting limit > MDL:<br>note in worksheet if RRF <0.05                                     | 5B          |
|                                                     | %D <25%                                                                                 | (EcoChem PJ, see TM-06)<br>If > +/-90%: J+/-R-<br>If -90% to -26%: J+ (high bias)<br>If 26% to 90%: J+/UJ- (low bias)                                                                 | 5B          |
| Method Blank                                        | One per matrix per batch<br>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           |
| Storage Blank                                       | One per SDG<br><QL                                                                      | U(+) the specific analyte(s) results in all assoc.samples using the 5x or 10x rule                                                                                                    | 7           |
| Trip Blank                                          | Frequency as per project QAPP                                                           | Same as method blank for positive results remaining in trip blank after method blank qualifiers are assigned                                                                          | 18          |
| Field Blanks                                        | No results > QL                                                                         | Apply 5X/10X rule; U(+) < action level                                                                                                                                                | 6           |
| MS/MSD (recovery)                                   | One per matrix per batch<br>Use method acceptance criteria                              | Qualify parent only unless other QC indicates systematic problems:<br>J(+) if both %R > UCL<br>J(+)/UJ(-) if both %R < LCL<br>J(+)/R(-) if both %R < 10%<br>PJ if only one %R outlier | 8           |
| MS/MSD (RPD)                                        | One per matrix per batch<br>Use method acceptance criteria                              | J(+) if RPD > CL                                                                                                                                                                      | 9           |

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

| VALIDATION QC ELEMENT                       | ACCEPTANCE CRITERIA                                                                                                                                                                                                          | ACTION                                                                                                                               | REASON CODE        |
|---------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------|--------------------|
| Lab Duplicate                               | <p><b>Results &gt;5X reporting limit (RL):</b><br/> RPD criteria: use QAPP limits, all matrices</p> <p><b>Results &lt;5X RL:</b><br/> Solid: Absolute difference &lt; 2X RL<br/> Aqueous: Absolute difference &lt; 1X RL</p> | J(+)/UJ(-) if outside limits                                                                                                         | 9                  |
| LCS<br><i>low conc. H2O VOA</i>             | <p>One per lab batch<br/> Within method control limits</p>                                                                                                                                                                   | <p>J(+) assoc. compd if &gt; UCL<br/> J(+)/R(-) assoc. compd if &lt; LCL<br/> J(+)/R(-) all compds if half are &lt; LCL</p>          | 10                 |
| LCS<br><i>regular VOA (H2O &amp; solid)</i> | <p>One per lab batch<br/> Lab or method control limits</p>                                                                                                                                                                   | <p>J(+) if %R &gt; UCL J(+)/UJ(-) if %R &lt; LCL<br/> J(+)/R(-) if %R &lt; 10% (EcoChem PJ)</p>                                      | 10                 |
| LCS/LCSD<br><i>(if required)</i>            | <p>One set per matrix and batch of 20 samples<br/> RPD &lt; 35%</p>                                                                                                                                                          | J(+) assoc. compd. in all samples                                                                                                    | 9                  |
| Surrogates                                  | <p>Added to all samples<br/> Within method control limits</p>                                                                                                                                                                | <p>J(+) if %R &gt; UCL<br/> J(+)/UJ(-) if %R &lt; LCL but &gt; 10% (see PJ1)<br/> J(+)/R(-) if &lt; 10%</p>                          | 13                 |
| Internal Standards                          | <p>Added to all samples<br/> Acceptable Range: IS area 50% to 200% of CCAL area<br/> RT within 30 seconds of CC RT</p>                                                                                                       | <p>J(+) if &gt; 200%<br/> J(+)/UJ(-) if &lt; 50%<br/> J(+)/R(-) if &lt; 25%<br/> R T &gt; 30 seconds, narrate and Notify PM</p>      | 19                 |
| Field Duplicates                            | <p><b>Results &gt;5X reporting limit (RL):</b><br/> RPD &lt; 50% (all matrices, QAPP specified)</p> <p><b>Results &lt;5X RL:</b><br/> Solid: Absolute difference &lt; 2X RL<br/> Aqueous: Absolute difference &lt; 1X RL</p> | Narrate: do not qualify                                                                                                              | na                 |
| TICs                                        | <p>Major ions (&gt;10%) in reference must be present in sample; intensities agree within 20%; check identification</p>                                                                                                       | <p>R(+) common laboratory contaminants<br/> R(+) target compounds from other fractions<br/> See Technical Director for ID issues</p> | 14                 |
| Quantitation/<br>Identification             | <p>RRT within 0.06 of standard RRT<br/> Ion relative intensity within 20% of standard<br/> All ions in std. at &gt; 10% intensity must be present in sample</p>                                                              | See Technical Director if outliers                                                                                                   | 14<br>21 (false +) |

**PJ1** No action if there are 4+ surrogates and only 1 outlier.

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<br>Tissues <-10°C                                                        | J(+)/UJ(-) if greater than 6 deg. C<br>(EcoChem PJ)                                                                                                                                                                                                   | 1           |
| Holding Time                                           | Water: 7 days from collection<br>Soil: 14 days from collection<br>Analysis: 40 days from extraction | <u>Water:</u><br>J(+)/UJ(-) if ext. > 7 and < 21 days<br>J(+)/R(-) if ext > 21 days (EcoChem PJ)<br><u>Solids/Wastes:</u><br>J(+)/UJ(-) if ext. > 14 and < 42 days<br>J(+)/R(-) if ext. > 42 days (EcoChem PJ)<br><br>J(+)/UJ(-) if analysis >40 days | 1           |
| Tuning                                                 | DFTPP<br>Beginning of each 12 hour period<br>Method acceptance criteria                             | R(+/-) all analytes in all samples associated with the tune                                                                                                                                                                                           | 5A          |
| Initial Calibration<br>(Minimum 5 stds.)               | RRF > 0.05                                                                                          | (EcoChem PJ, see TM-06)<br>If MDL= reporting limit:<br>J(+)/R(-) if RRF < 0.05<br><br>If reporting limit > MDL:<br>note in worksheet if RRF <0.05                                                                                                     | 5A          |
|                                                        | %RSD < 30%                                                                                          | (EcoChem PJ, see TM-06)<br>J(+) if %RSD > 30%                                                                                                                                                                                                         | 5A          |
| Continuing Calibration<br>(Prior to each 12 hr. shift) | RRF > 0.05                                                                                          | (EcoChem PJ, see TM-06)<br>If MDL= reporting limit:<br>J(+)/R(-) if RRF < 0.05<br><br>If reporting limit > MDL:<br>note in worksheet if RRF <0.05                                                                                                     | 5B          |
|                                                        | %D <25%                                                                                             | (EcoChem PJ, see TM-06)<br>If > +/-90%: J+/R-<br>If -90% to -26%: J+ (high bias)<br>If 26% to 90%: J+/UJ- (low bias)                                                                                                                                  | 5B          |
| Method Blank                                           | One per matrix per batch<br>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<br>Use method acceptance criteria                                                                                                                                            | Qualify parent only unless other QC indicates systematic problems:<br>J(+) if both %R > UCL<br>J(+)/UJ(-) if both %R < LCL<br>J(+)/R(-) if both %R < 10%<br>PJ if only one %R outlier | 8                  |
| MS/MSD (RPD)                   | One per matrix per batch<br>Use method acceptance criteria                                                                                                                                            | J(+) if RPD > CL                                                                                                                                                                      | 9                  |
| Lab Duplicate                  | <b>Results &gt;5X reporting limit (RL):</b><br>RPD criteria: use QAPP limits, all matrices<br><b>Results &lt;5X RL:</b><br>Solid: Absolute difference < 2X RL<br>Aqueous: Absolute difference < 1X RL | J(+)/UJ(-) if outside limits                                                                                                                                                          | 9                  |
| LCS low conc. H2O SVOA         | One per lab batch<br>Within method control limits                                                                                                                                                     | J(+) assoc. cmpd if > UCL<br>J(+)/R(-) assoc. cmpd if < LCL<br>J(+)/R(-) all cmpds if half are < LCL                                                                                  | 10                 |
| LCS regular SVOA (H2O & solid) | One per lab batch<br>Lab or method control limits                                                                                                                                                     | J(+) if %R > UCL J(+)/UJ(-) if %R < LCL<br>J(+)/R(-) if %R < 10% (EcoChem PJ)                                                                                                         | 10                 |
| LCS/LCSD (if required)         | One set per matrix and batch of 20 samples<br>RPD < 35%                                                                                                                                               | J(+) assoc. cmpd. in all samples                                                                                                                                                      | 9                  |
| Surrogates                     | Minimum of 3 acid and 3 base/neutral compounds<br>Use method acceptance criteria                                                                                                                      | Do not qualify if only 1 acid and/or 1 B/N surrogate is out unless <10%<br>J(+) if %R > UCL J(+)/UJ(-) if %R < LCL<br>J(+)/R(-) if %R < 10%                                           | 13                 |
| Internal Standards             | Added to all samples<br>Acceptable Range: IS area 50% to 200% of CCAL area<br>RT within 30 seconds of CC RT                                                                                           | J(+) if > 200%<br>J(+)/UJ(-) if < 50%<br>J(+)/R(-) if < 25%<br>R T>30 seconds, narrate and Notify PM                                                                                  | 19                 |
| Field Duplicates               | <b>Results &gt;5X reporting limit (RL):</b><br>RPD < 50% (all matrices, QAPP specified)<br><b>Results &lt;5X RL:</b><br>Solid: Absolute difference < 2X RL<br>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<br>R(+) target compounds from other fractions<br>See Technical Director for ID issues                                                             | 4                  |
| Quantitation/ Identification   | RRT within 0.06 of standard RRT<br>Ion relative intensity within 20% of standard<br>All ions in std. at > 10% intensity must be present in sample                                                     | See Technical Director if outliers                                                                                                                                                    | 14<br>21 (false +) |

DATA VALIDATION CRITERIA

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<br>Tissues <-10°C                                                                                                                                       | J(+)/UJ(-) if greater than 6 deg. C<br>(EcoChem PJ)                                                                                                        | 1           |
| Holding Time                       | Water: 7 days from collection<br>Soil: 14 days from collection<br>Analysis: 40 days from extraction                                                                                | J(+)/UJ(-) if ext/analyzed > HT<br>J(+)/R(-) if ext/analyzed > 3X HT (EcoChem PJ)                                                                          | 1           |
| Resolution Check                   | Beginning of ICAL Sequence<br>Within RTW Resolution >90%                                                                                                                           | Narrate (Use Professional Judgement to qualify)                                                                                                            | 14          |
| Instrument Performance (Breakdown) | DDT Breakdown: < 20%<br>Endrin Breakdown: <20%<br>Combined Breakdown: <30%<br>Compounds within RTW                                                                                 | J(+) DDT NJ(+) DDD and/or DDE<br>R(-) DDT - If (+) for either DDE or DDD<br><br>J(+) Endrin NJ(+) EK and/or EA<br>R(-) Endrin - If (+) for either EK or EA | 5A          |
| Retention Times                    | Surrogates:<br>TCX (+/- 0.05); DCB (+/- 0.10)<br>Target compounds:<br>elute before heptachlor epoxide (+/- 0.05)<br>elute after heptachlor epoxide (+/- 0.07)                      | NJ(+)/R(-) results for analytes with RT shifts<br>For full DV, use PJ based on examination of raw data                                                     | 5B          |
| Initial Calibration                | Pesticides: Low=QL, Mid=4X, High=16X<br>Multiresponse - one point Calibration<br>%RSD<20%<br>%RSD<30% for surr; two comp. may exceed if <30%<br>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)<br>%D < 25%<br><br>Resolution >90% in IND mixes;<br>100% for PEM                 | J(+)/UJ(-) J(+)/R(-) if %D > 90%<br><br>PJ for resolution                                                                                                  | 5B          |
| Method Blank                       | One per matrix per batch<br>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<br>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<br>Method Acceptance Criteria                                                                                                                                            | Qualify parent only unless other QC indicates systematic problems:<br>J(+) if both %R > UCL<br>J(+)/UJ(-) if both %R < LCL<br>J(+)/R(-) if both %R < 10%<br>PJ if only one %R outlier | 8           |
| MS/MSD (RPD)                 | One set per matrix per batch<br>Method Acceptance Criteria                                                                                                                                            | J(+) if RPD > CL                                                                                                                                                                      | 9           |
| Lab Duplicate                | <b>Results &gt;5X reporting limit (RL):</b><br>RPD criteria: use QAPP limits, all matrices<br><b>Results &lt;5X RL:</b><br>Solid: Absolute difference < 2X RL<br>Aqueous: Absolute difference < 1X RL | J(+)/UJ(-) if outside limits                                                                                                                                                          | 9           |
| LCS/LCSD (if required)       | One set per matrix and batch of 20 samples<br>RPD < 35%                                                                                                                                               | J(+) assoc. compd. in all samples                                                                                                                                                     | 9           |
| Surrogates                   | TCX and DCB added to every sample<br>%R = 30-150%                                                                                                                                                     | J(+)/UJ(-) if both %R = 10 - 60%<br>J(+) if both >150%<br>J(+)/R(-) if any %R <10%                                                                                                    | 13          |
| Quantitation/ Identification | Analyte within RTW on both columns<br>Quantitated using CCV or ICAL CF<br>Lowest value from either column reported<br>RPD between columns (25%)                                                       | J(+) if RPD = 25-60% (Pest/Aroclor);<br>40-60% (Herb/Phenol)<br>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<br>Florisil required for all samples<br>Sulfur is optional<br><br>Clean-up standard check %R within CLP limits                                                          | J(+)/UJ(-) if %R < LCL<br>J(+) if %R > UCL                                                                                                                                            | 14          |
| Field Duplicates             | <b>Results &gt;5X reporting limit (RL):</b><br>RPD < 50% (all matrices, QAPP specified)<br><b>Results &lt;5X RL:</b><br>Solid: Absolute difference < 2X RL<br>Aqueous: Absolute difference < 1X RL    | Narrate; do not qualify                                                                                                                                                               | na          |

# 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<br>Tissues <-10°C<br>Water Only: Nitric Acid to pH < 2<br>For Dissolved metals, 0.45 um filter preserve after filtration                          | EcoChem PJ<br>J(+)/UJ(-) if preservation requirements are not met                                                                                                                                                    | 1           |
| Holding Time                                | 180 days                                                                                                                                                                     | EcoChem PJ<br>J(+)/UJ(-) if holding time exceeded<br>J(+)/R(-) if HT exceeded by 3x                                                                                                                                  | 1           |
| Tune                                        | Prior to ICAL<br>Analyzed 5 times with Std Dev. ≤ 5%<br>mass calibration <0.1 amu from True Value<br>Resolution < 0.9 AMU @ 10% peak height or<br><0.75 amu @ 5% peak height | EcoChem PJ<br>No Tune - R all results<br>criteria not met - J(+)/UJ(-)                                                                                                                                               | 5A          |
| Initial Calibration                         | Minimum Blank+1 Standard every 24 hours                                                                                                                                      | EcoChem PJ<br>J(+)/UJ(-) >24 hours<br>J(+)/UJ(-) if r<0.995 (for multi point cal)                                                                                                                                    | 5A          |
| Initial Calibration Verification (ICV)      | Independent source;<br>analyzed post ICAL and prior to samples<br>+/-10% of the True value                                                                                   | EcoChem PJ<br>J(+)/UJ(-) if %R 75%-89%<br>J(+) if %R = 111-125%<br>R(+) if %R > 125%<br>R(+/-) if %R < 75%                                                                                                           | 5A          |
| Continuing Cal Verification (CCV)           | Every 10 samples, post ICV/ICB and end of run<br>+/- 10% of True value                                                                                                       | EcoChem PJ<br>J(+)/UJ(-) if %R 75%-89%<br>J(+) if %R = 111-125%<br>R(+) if %R > 125%<br>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)<br>Not required for Al, Ba, Ca, Fe, Mg, Na, K<br>%R = 70%-130% (50%-150% Co, Mn, Zn)         | EcoChem PJ<br>R(-),(+) < 2XRL if %R < 50% (< 30% Co, Mn, Zn)<br>J(+)<2XRL, UJ(-) if %R 50-69% (30%-49% Co, Mn, Zn)<br>J(+)<2X RL if %R 130%-180% (150%-200% Co, Mn, Zn)<br>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.<br>For (+) blk value, U(+) values < AL<br>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.<br>For (+) blk value, U(+) values < AL<br>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<br>ICSA/ICSAB | ICSAB +/- 20% of true value<br>ICSA < +/- IDL                                                                                                   | Where Al,Ca,Fe,Mg = ICS levels<br>J(+) if %R >120%<br>J(+)/UJ(-) if %R = 50% to 79%<br>R(+/-) if %R<50%<br>EcoChem PJ for ICSA > +/- IDL | 17          |
| Post Digestion Spike                     | If ICP Matrix Spike is outside 75-125%<br>Spike parent sample at 2X the sample conc.                                                            | EcoChem PJ - usually no action                                                                                                           | 14          |
| Matrix Spike                             | One per matrix, batch and SDG<br>75-125% for samples where results<br>do not exceed 4x spike level                                              | J (+) if %R > 125%<br>J(+)/UJ(-) if %R < 75%<br>J(+)/R(-) if %R < 30%<br>UJ(-) if %R = 30-74%                                            | 8           |
| Laboratory Duplicate                     | One per matrix per batch<br>RPD <20% for samples > 5x RL<br>Diff<RL for samples >RL and <5 x RL<br>(may use RPD < 35%, Diff < 2X RL for solids) | J(+)/UJ(-) associated samples<br>if RPD > 20% or diff > RL                                                                               | 9           |
| Laboratory Control Sample                | Waters:<br>One per matrix per batch<br>%R (80-120%)                                                                                             | R(+/-) if %R < 50%<br>J(+)/UJ(-) if %R = 50-79%<br>J(+) if %R >120%                                                                      | 10          |
|                                          | Soils:<br>One per matrix per batch<br>result within manufacturer's certified acceptance range                                                   | J(+)/UJ(-) if < LCL,<br>J(+) if > UCL                                                                                                    | 10          |
| Serial Dilution                          | 5x dilution one per matrix (or SDG)<br>%D <10% of the undiluted value<br>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<br>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<br>samples diluted to fall within range                                                                                       | J(+) values over range                                                                                                                   | 20          |



**EcoChem, INC.**  
Environmental Data Quality

# **APPENDIX B**

# **DATA VALIDATION REPORTS**

**DATA VALIDATION REPORT**  
**Portland Harbor RI/FS**  
**Lamprey Ammocoete Toxicity Testing**  
**Naphthalene – Method SW8260B**

This report documents the review of analytical data from the analyses of water 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 |
|----------|-------------------------|------------------|
| K0710392 | 31 Water                | Summary          |
| K0710943 | 32 Water & 1 Trip Blank | Summary          |

**I. DATA PACKAGE COMPLETENESS**

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

**II. EDD TO HARDCOPY VERIFICATION**

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

**III. TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below.

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

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

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

**Holding Times and Sample Receipt**

As stated in validation guidance documents, sample shipping coolers should be within the advisory temperature range of 2° to 6°C upon receipt at the laboratory. The laboratory received several of the sample coolers with temperatures outside of the advisory control limits. These outliers did not impact data quality and no qualifiers were required.

## Field and Trip Blanks

Laboratory (method) blanks are used to evaluate all associated field and trip blanks. Any remaining positive results in the field and trip blanks are used to evaluate all associated samples.

**SDG K0710943:** One trip blank, Sample Trip Blank, was included. Naphthalene was not detected in this blank.

## 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).

**SDG K0710392:** The %R values for dibromofluoromethane were less than lower control limits in Samples NAS#1387G Day0-0.03mg/L Naphthalene NEW and NAS#1416G Day4-0.3mg/L Naphthalene NEW. Since naphthalene is not directly associated with this surrogate and as the %R values for both other surrogates were within limits; no qualifiers were applied.

## Field Replicates

Replicate 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 reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and duplicate must be less than two times the RL. No data were qualified based on duplicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

**SDG K0710392:** One duplicate set was included. For Samples NAS#1412G Day2-30mg/L Naphthalene NEW and NAS#1413G Day2-30mg/L Naphthalene NEW-Dupl, the RPD values were within control limits.

**SDG K0710943:** Two duplicate sets were included. For Samples NAS#1446G Day 2- 1.25 mg/L Naphthalene & NAS#1451G Day 2 – 1.25 mg/L Naphthalene Duplicate and Samples NAS#1443G Day 1 – 2.02 mg/L Naphthalene & NAS#1444G Day 1 – 20.0 mg/L Naphthalene Duplicate, the RPD values were within control limits.

## Reporting Limits

Some of the sample reporting limits were elevated due to sample dilutions required to bring the naphthalene result into the instrument linear range. Several elevated reporting limits exceeded the project target reporting limits as listed in the QAPP. No action was taken other than to note the discrepancies.

#### **IV. OVERALL ASSESSMENT**

As was determined by this evaluation, the laboratory followed the appropriate analytical method. Accuracy was acceptable, as demonstrated by the surrogate, matrix spike/matrix spike duplicate (MS/MSD), and laboratory control sample (LCS) %R values. Precision was acceptable as demonstrated by the RPD values for the MS/MSD and duplicate analyses.

No data were qualified for any reason.

All data, as reported, are acceptable for use.

**DATA VALIDATION REPORT**  
**Portland Harbor RI/FS**  
**Lamprey Ammocoete Toxicity Testing**  
**Aniline – Method SW8270C**

This report documents the review of analytical data from the analyses of water 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 |
|----------|-------------------|------------------|
| K0709054 | 12 Water          | Summary          |
| K0709170 | 12 Water          | Summary          |
| K0709308 | 7 Water           | 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 | Holding Times and Sample Receipt | Laboratory Control Samples (LCS/LCSD)             |
|   | Initial Calibration (ICAL)       | 1 Field Replicates                                |
|   | Continuing Calibration (CCAL)    | 1 Reporting Limits                                |
|   | Laboratory Blanks                | Compound Identification                           |
|   | Surrogate Compounds              | 1 Calculation Verification (full validation only) |
| 1 | Matrix Spikes                    |                                                   |

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

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

**Holding Times and Sample Receipt**

The laboratory received many of the sample coolers with temperatures outside the advisory control limits of 2° to 6°C. The temperature outliers ranged from -0.6° to 9.8°C. These temperature outliers did not impact data quality and no qualifiers were required.

## Matrix Spikes

*SDGs K0709170 & K0709308:* No matrix spike (MS) analysis was performed. Accuracy was assessed by evaluating the recovery values for the surrogates and the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) spiked compounds; precision was measured using LCS/LCSD analyses.

## Field Replicates

Replicate 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 reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and duplicate must be less than two times the RL. No data were qualified based on duplicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

*SDG K0709308:* Samples NAS# 1333G Day 4 – 300 mg/L Aniline and NAS# 1337G Day 4 – 300 mg/L Aniline Dupl were submitted as replicates. The RPD value was within the acceptance criteria.

## Calculation Verification

*SDG K0709308:* 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 appropriate analytical method. Accuracy was acceptable, as demonstrated by the surrogate, MS, and LCS/LCSD percent recovery values. Precision was acceptable as demonstrated by the RPD values for the LCS/LCSD and replicate analyses.

All data are acceptable for use.

**DATA VALIDATION REPORT**  
**Portland Harbor RI/FS**  
**Lamprey Ammocoete Toxicity Testing**  
**Chlorophenols – Method SW8151 (Modified)**

This report documents the review of analytical data from the analyses of water 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 |
|----------|-------------------|------------------|
| K0712117 | 32 Water          | Summary          |

### **I. DATA PACKAGE COMPLETENESS**

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

### **II. EDD TO HARDCOPY VERIFICATION**

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

### **III. TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below.

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

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

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

#### **Holding Times and Sample Receipt**

The laboratory received many of the sample coolers with temperatures outside the advisory control limits of 2° to 6°C. The temperature outliers ranged from -0.6° to 9.8°C. These temperature outliers did not impact data quality and no qualifiers were required.

## Matrix Spike/Matrix Spike Duplicates

For the matrix spike/matrix spike duplicate (MS/MSD) analyses performed using Sample NAS#1541G Day0-0.2mg/l Pentachlorophenol and a batch QC sample, the sample concentrations greatly exceeded the spiked amounts; no qualifiers were applied for percent recovery (%R) outliers.

For the MS/MSD analyses performed using Sample NAS#1545G Day1-0mg/l Pentachlorophenol, the relative percent difference (RPD) between results for pentachlorophenol exceeded the control limit; this analyte was not detected in the parent sample so no qualifier was required.

## Field Replicates

Replicate sample pairs are listed below. The following acceptance criteria were applied: 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 duplicate must be less than two times the RL. No data were qualified based on duplicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

*SDG K0712117:* Samples NAS# 1566G Day 4 – 0.0125 mg/L Pentachlorophenol and NAS# 1571G Day 4 – 0.0125 mg/L Pentachlorophenol Dupl were submitted as duplicates. The RPD value was within the acceptance criteria.

## IV. OVERALL ASSESSMENT

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

All data are acceptable for use.

**DATA VALIDATION REPORT**  
**Portland Harbor RI/FS**  
**Lamprey Ammocoete Toxicity Testing**  
**Gamma-BHC (Lindane) – Method SW8081A**

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

| SDG      | Number of Samples | Validation Level |
|----------|-------------------|------------------|
| K0709896 | 35 Water          | Summary          |

**I. DATA PACKAGE COMPLETENESS**

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

**II. EDD TO HARDCOPY VERIFICATION**

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

**III. TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below.

- |   |                                  |   |                                                 |
|---|----------------------------------|---|-------------------------------------------------|
| 1 | Holding Times and Sample Receipt | 2 | Laboratory Control Samples (LCS)                |
|   | Initial Calibration (ICAL)       | 1 | Field Replicates                                |
|   | Continuing Calibration (CCAL)    |   | Compound Identification                         |
| 1 | Laboratory Blanks                | 1 | Reporting Limits                                |
| 1 | Surrogate Compounds              |   | Calculation Verification (full validation only) |
|   | Matrix Spikes (MS)               |   |                                                 |

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

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

**Holding Times and Sample Receipt**

The laboratory received the majority of the sample coolers with temperatures outside the advisory control limits of 2° to 6°C, ranging from -1.2° to 7.6°C. 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. A summary of contaminant levels, associated samples, and action levels is provided in the data validation worksheets. Lindane was detected in the method blanks. The lindane results in all samples were greater than the action levels; no qualifiers were required.

## Surrogate Compounds

Due to the levels of lindane spiked, most of the sample extracts were diluted prior to analysis. Many surrogate percent recovery (%R) values exceeded control limits or were not recovered. No qualifiers were applied when surrogates were diluted out.

## Laboratory Control Samples

All %R values were within the control limits and all relative percent difference (RPD) values were less than the control limit in the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses, with the exceptions noted below. If the outliers indicated a potential high bias, the associated positive results were estimated (J-10). If the outliers indicated a potential low bias, positive results and reporting limits were estimated (J/UJ-10).

The RPD value for lindane exceeded the control limit of 30%, at 43% in the LCS/LCSD set extracted 10/29/07. The lindane results were qualified as estimated (J-9) for precision in the associated samples.

## Field Replicates

Replicate sample pairs are listed below. The following acceptance criteria were applied: 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 duplicate must be less than two times the RL. No data were qualified based on duplicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

**SDG K0709896:** Two pairs of replicate samples were analyzed. For Samples NAS# 1354G Day 1 – 1.0 mg/L Lindane and NAS#1358G Day 1 – 1.0 mg/L Lindane Dupl, results met the acceptance criterion. For Samples NAS# 1369G Day 3 – 4.0 mg/L Lindane and NAS#1371G Day 3 – 4.0 mg/L Lindane Dupl, the RPD value exceeded the acceptance criterion, at 81.4%.

## Reporting Limits

Many of the sample reporting limits were elevated due to sample dilutions required to bring the lindane result into the instrument linear range. Several elevated reporting limits exceeded the project target reporting limits as listed in the QAPP. No action was taken other than to note the discrepancies.

## 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 matrix spike %R values, with the exceptions noted above. Precision was acceptable where assessed, as demonstrated by the RPD values for the LCS/LCSD and replicate analyses, with the exceptions noted above.

Data were estimated due to a LCS/LCSD RPD outlier.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT**  
**Portland Harbor RI/FS**  
**Lamprey Ammocoete Toxicity Testing**  
**Diazinon by Method SW8141A**

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

| SDG      | Number of Samples | Validation Level |
|----------|-------------------|------------------|
| K0800585 | 32 Water          | Summary          |

### **I. DATA PACKAGE COMPLETENESS**

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

### **II. EDD TO HARDCOPY VERIFICATION**

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

### **III. TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below.

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

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

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

### **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:

A positive result for diazinon was detected in the method blank. This analyte was detected in all the associated samples at levels greater than the action level; no qualifiers were required.

### **Matrix Spikes/Matrix Spike Duplicates**

**SDG K0800585:** No matrix spike/matrix spike duplicate (MS/MSD) set was submitted. Precision was assessed from the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) and duplicate sample analyses.

### **Field Replicates**

Replicate 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 reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and duplicate must be less than two times the RL. No data were qualified based on duplicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

Replicate analyses were performed on Samples NAS# 1596G Day 1-10.0 mg/L Diazinon & NAS# 1599G Day 1-10.0 mg/L DiazinonDUPLICATE and NAS# 1605G Day 2-2.5 mg/L Diazinon and NAS# 1610G Day 2-2.5 mg/L DiazinonDUPLICATE. All RPD values were within the acceptance criteria.

### **Compound Identification**

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

The Diazinon result for Sample NAS#1588G Day 0-20.0 mg/L Diazinon was estimated (J-3).

### **Reporting Limits**

Most samples were analyzed at dilution and reporting limits were elevated accordingly.

#### **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 LCS/LCSD %R values. Precision was acceptable where assessed, as demonstrated by the RPD values for the LCS/LCSD and replicate analyses.

Data were estimated because the confirmation criteria were not met.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT**  
**Portland Harbor RI/FS**  
**Lamprey Ammocoete Toxicity Testing**  
**Chlorinated Herbicides – Method SW8151A**

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

| SDG      | Number of Samples | Validation Level |
|----------|-------------------|------------------|
| K0706805 | 1 Soil            | Summary          |

### **I. DATA PACKAGE COMPLETENESS**

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

### **II. EDD TO HARDCOPY VERIFICATION**

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

### **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)  |
|   | Initial Calibration (ICAL)       | 2 | Laboratory Control Samples (LCS)                |
|   | Continuing Calibration (CCAL)    |   | Compound Identification                         |
|   | Laboratory Blanks                | 1 | Reporting Limits                                |
|   | Surrogate Compounds              |   | Calculation Verification (full validation only) |

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

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

#### **Holding Times and Sample Receipt**

The cooler temperatures were not indicated on the cooler receipt form by the laboratory.

#### **Matrix Spikes/Matrix Spike Duplicates**

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed at an acceptable frequency. All percent recovery (%R) values were within the control limits, with the exception noted below.

When outliers were present, the associated compounds were qualified in the parent sample only. If the outliers indicated a potential high bias, only the associated positive results were qualified as estimated (J-8). If the outliers indicated a potential low bias, results and reporting limits were estimated (J/UJ-8). When the %R was less than 10% results were estimated and reporting limits were rejected (J/R-8).

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.

RPD and %R value outliers (with bias, when appropriate) are listed below. Data were qualified as discussed above.

- NAS#1135G Waupaca Materials – dinoseb – MSD only (low bias <10% and high RPD)

### **Laboratory Control Samples**

All %R values were within the control limits and all RPD values were less than the control limit in the laboratory control sample (LCS) analyses, with the exception noted below. If the outliers indicated a potential high bias, the associated results were estimated (J-10). If the outliers indicated a potential low bias, results and reporting limits were estimated (J/UJ-10). When the %R was less than 10%, results were estimated and reporting limits were rejected (J/R-10).

- NAS#1135G Waupaca Materials – dinoseb – LCS only (low bias <10%)

### **Reporting Limits**

The laboratory elevated the detection limits for some analytes due to background interferences.

## **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 acceptable, as demonstrated by the RPD values for the MS/MSD analyses, with the exception noted above.

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

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT**  
**Portland Harbor RI/FS**  
**Lamprey Ammocoete Sampling**  
**Chlorinated Pesticides – Method SW8081A**

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

| SDG      | Number of Samples | Validation Level |
|----------|-------------------|------------------|
| K0706805 | 1 Soil            | Summary          |

**I. DATA PACKAGE COMPLETENESS**

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

**II. EDD TO HARDCOPY VERIFICATION**

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

**III. TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below.

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

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

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

**Holding Times and Sample Receipt**

The cooler temperatures were not indicated on the receipt form by the laboratory.

## Continuing Calibration

The percent difference (%D) value for methoxychlor was greater than the control limit of  $\pm 25\%$  on the DB-XLB column from the 9/16/07 continuing calibration (CCAL). The %D values for these analytes were within control limits on the DB-35ms column and no qualifiers were required.

## Laboratory Control Samples

All reported percent recovery (%R) values were within the control limits and all relative percent difference (RPD) values were less than the control limit in the laboratory control sample (LCS) analysis.

Due to laboratory error the LCS was not spiked for hexachlorobutadiene, oxychlordan, cis-nonachlor, or trans-nonachlor. This discrepancy was noted in the case narrative. No qualifiers were applied.

## 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 matrix spike/matrix spike duplicate (MS/MSD) %R values. Precision was acceptable where assessed, as demonstrated by the RPD values for the MS/MSD analyses.

All data, as reported, are acceptable for use.

**DATA VALIDATION REPORT**  
**Portland Harbor RI/FS**  
**Lamprey Ammocoete Toxicity Testing**  
**Total & Dissolved Copper – EPA Method 200.8**

This report documents the review of analytical data from the analyses of water 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 |
|----------|-------------------|------------------|
| K0708429 | 14 Water          | Full             |
| K0708432 | 24 Water          | Summary          |
| K0708470 | 26 Water          | Summary          |

**I. DATA PACKAGE COMPLETENESS**

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

**II. EDD TO HARDCOPY VERIFICATION**

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

**III. TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below.

- |                                                                                                                                                                                  |                                                                                                                                                                                                                                                                                |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <ul style="list-style-type: none"> <li>1 Holding Times and Sample Preservation</li> <li>Initial Calibration</li> <li>Calibration Verification</li> <li>CRDL Standards</li> </ul> | <ul style="list-style-type: none"> <li>Laboratory Duplicates</li> </ul>                                                                                                                                                                                                        |
| <ul style="list-style-type: none"> <li>2 Laboratory Blanks</li> <li>Laboratory Control Samples</li> <li>Matrix Spike Samples</li> </ul>                                          | <ul style="list-style-type: none"> <li>1 Field Replicates</li> <li>ICPMS Internal Standards</li> <li>Serial Dilutions</li> <li>ICP Interference Standards</li> <li>Reporting Limits and Reported Results</li> <li>1 Calculation Verification (Full validation only)</li> </ul> |

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *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  $4^{\circ} \pm 2^{\circ}\text{C}$  (the range of recorded temperature was  $-0.1^{\circ}$  to  $7.3^{\circ}\text{C}$ ). The temperature outliers did not impact data quality and no data were qualified.

## Laboratory Blanks

Various analytes were detected in the method and instrument blanks at levels greater than the method detection limits (MDL). To evaluate the effect on the sample data, action levels of five times (5x) the blank concentrations were established. Positive results less than the action levels in the associated samples were qualified as not detected (U) at the reported concentration. No action was taken for non-detects.

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

**SDG K0708429:** The copper results for the instrument blanks were less than the negative MDL. Associated results were greater than the action levels and no qualification of data was necessary.

**SDG K0708432:** The copper results for two instrument blanks were less than the negative MDL. Associated results were greater than the action levels and no qualification of data was necessary.

**SDG K0708470:** Copper was detected in both method blanks at levels greater than the MDL. The result for Sample NAS# 1252G Day 3—0.0 mg/L was less than the action level and was qualified as not detected (U-7). All other sample results were greater than the action levels

The copper results for the instrument blanks were less than the negative MDL. Associated results were positive and greater than the action levels and no qualification of data was necessary.

## Field Replicates

Replicate 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 reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and duplicate must be less than two times the RL. No data were qualified based on replicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

**SDG K0708429:** Replicate analyses were performed on Samples NAS# 1208G Day 0-0.05 mg/L Copper & NAS# 1217G Day 0-0.05 mg/L Copper. All RPD values were within the acceptance criteria.

**SDG K0708470:** Replicate analyses were performed on Samples NAS# 1250G Day 3-0.1 mg/L Copper & NAS# 1258G Day 3-0.1 mg/L Copper. All RPD values were within the acceptance criteria.

### **Calculation Verification**

**SDG K0708429:** 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 matrix spike recoveries. Precision was also acceptable as demonstrated by the RPD values for the laboratory and field replicate analyses.

The copper result for one sample was qualified as not detected based on laboratory blank contamination.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT**  
**Portland Harbor RI/FS**  
**Lamprey Ammocoete Toxicity Testing**  
**General Chemistry Parameters**

This report documents the review of analytical data from the analyses of water 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 |
|----------|-------------------|------------------|
| K0708429 | 1 Water           | Summary          |
| K0708470 | 2 Water           | Summary          |
| K0709054 | 1 Water           | Summary          |
| K0709308 | 1 Water           | Summary          |
| K0709896 | 3 Water           | Summary          |
| K0710943 | 3 Water           | Summary          |
| K0712117 | 3 Water           | Summary          |
| K0800585 | 5 Water           | Summary          |

The analytical tests that were performed are summarized below:

| Parameter                    | Method        |
|------------------------------|---------------|
| Total Suspended Solids (TSS) | SM 2540D      |
| Total Organic Carbon (TOC)   | 415.1         |
| Ammonia as Nitrogen          | SM 4500-NH3 E |

## **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                   | Laboratory Duplicates                             |
|   | Calibration Verification              | 1 Field Replicates                                |
| 2 | Laboratory Blanks                     | Reporting Limits (MDL and MRL)                    |
|   | Laboratory Control Samples            | 1 Calculation Verification (Full validation only) |

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified*

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

#### Holding Times and Sample Preservation

The laboratory received many of the sample coolers with temperatures outside the advisory control limits of 2° to 6°C. The temperature outliers ranged from -0.1° to 9.8°C. These temperature outliers did not impact data quality and no qualifiers were required.

**SDG K0709896:** Total suspended solids (TSS) were initially analyzed on 10/25/07. The analyst disposed of the method blank filter before it was recorded. Reanalysis of the sample was performed outside of holding time. Because TSS were not detected in either analysis, the result from the original analysis should be used. The result of the reanalysis was qualified as do-not-report (DNR-11).

#### Laboratory Blanks

**SDG K0709054:** Ammonia was detected in the method and instrument blanks at levels greater than the method detection limit (MDL). To evaluate the effect on the sample data, an action level of five times (5x) the blank concentrations were established. The associated result was greater than the action level, therefore no qualification of data was necessary.

**SDG K0709308:** Ammonia was detected in the method and instrument blanks at levels greater than MDL. The associated positive result was less than the action level and was qualified as not detected (U-7) at the reported concentration.

**SDG K0709896:** Total organic carbon (TOC) was detected in the instrument blanks at levels greater than MDL. The associated positive results were less than the action level and were qualified as not detected (U-7) at the reported concentrations.

**SDG K0710943:** TOC was detected in the instrument blanks at levels greater than MDL. The associated results were greater than the action levels, therefore no qualification of data was necessary.

**SDG K0712117:** TOC was detected in the instrument blanks at levels greater than MDL. The associated results were greater than the action levels, therefore no qualification of data was necessary.

**SDG K0800585:** Ammonia was detected in the method blank and instrument blanks at levels greater than MDL. The associated sample results were less than the action level and were qualified as not detected (U-7) at the reported concentrations.

### **Field Replicates**

Replicate 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 reporting limit (RL). For results less than five times the RL, the absolute difference between the sample and duplicate must be less than two times the RL. No data were qualified based on duplicate precision outliers. Users of the data should consider the impact of field precision outliers on the reported results.

**SDG K0709896:** Samples NAS# 1378G Day 4 – Ammonia and NAS# 1379G Day 4 – Ammonia Dupl were submitted as replicates. The RPD value was within the acceptance criteria.

### **Calculation Verification**

**SDG K0709308:** Several results were verified by recalculation from the raw data. No calculation or transcription errors were found.

## **IV. OVERALL ASSESSMENT**

As determined by this evaluation, the laboratory followed the specified analytical methods. The laboratory duplicate RPD values indicated acceptable precision. Accuracy was also acceptable, as demonstrated by the matrix spike and laboratory control sample recoveries.

Results for ammonia and TOC were qualified as not-detected based on blank contamination. One TSS result was qualified as do-not-report (DNR) to indicate which result should be used from multiple results for the same sample.

Data that have been labeled as DNR should not be used for any reason. All other data, as qualified, are acceptable for use.