

**APPENDIX R – DATA VALIDATION AND DATA MANAGEMENT**

**FINAL REMEDIAL INVESTIGATION REPORT  
CASMALIA RESOURCES SUPERFUND SITE  
CASMALIA, CALIFORNIA**

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## **LIST OF ATTACHMENTS**

ATTACHMENT R-1 COMPACT DISC CONTAINING THE CASMALIA RI DATABASE WITH  
STRUCTURE NOTES

**LIST OF ACRONYMS**

CAS	Chemical Abstract Service
CB	Calibration Blank
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verifications
CSC	Casmalia Steering Committee
EDD	Electronic Data Deliverable
EPA	Environmental Protection Agency
IC	Initial Calibration
ICB	Initial Calibration Blank
ICS	Inference Check Samples
ICV	Initial Calibration Verifications
LCS	Laboratory Control Samples
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
RI/FS	Remedial Investigation/Feasibility Study
RPD	relative percent difference
RRF	Relative Response Factors
RSD	Relative Standard Deviation
SDG	Sample Delivery Group
TIC	Tentatively Identified Compound

## 1.0 INTRODUCTION

The Casmalia Steering Committee (CSC) has prepared the Casmalia Project Database to store the environmental data being collected during the Remedial Investigation (RI) in accordance with the June 2004 *RI/FS Work Plan* which was prepared by the CSC and submitted to the EPA (CSC, 2004). This appendix describes the database structure and the kinds of data that it contains. In addition, a summary of the data validation conducted for the data contained in the database is provided.

### 1.1 Data Management

The Casmalia Project database is designed to handle the large amount of data collected at and near the project site. The Microsoft Access® database format was chosen for its ease of use, availability to the greatest number of users, and because it is accepted as an industry wide standard tool. The current database structure can be found in the *Casmalia\_Database\_Casmalia\_Database\_Structure\_20071114.pdf* file which accompanies the database on compact disc (CD). The database houses: laboratory analytical results which include soil, sediment, soil vapor, surface water and groundwater data; sample locations; geology (hydrostratigraphic units); water levels; and threshold values collected for the RI and used to evaluate the data. The database was used to provide data management capabilities in support of the human health and ecological risk assessments and feasibility studies at the site.

In total, the CHEMICAL table is comprised of: 1117 soil samples (including 76 field duplicates); 90 sediment samples (including 8 field duplicates); 49 primary soil vapor samples (including 4 field duplicates); 31 surface water samples (including 3 field duplicates). This includes both data collected under the RI and historical/background data.

The RI portion of the database is comprised of: 929 soil samples (including 76 field duplicates); 90 sediment samples (including 8 field duplicates); 49 primary soil vapor samples (including 4 field duplicates); and 19 surface water samples (including 1 field duplicate). These samples were collected between July 19, 2004 and November 6, 2007 as part of the Remedial Investigation/ Feasibility Study (RI/FS) Component of Work for the Casmalia Resources Superfund Site.

An additional data table containing groundwater sampling results obtained in conjunction with RI activities has been added to the project database (CHEMICAL\_GW). The structure of the groundwater table is consistent with the RI chemical table. The groundwater table consists of 474 groundwater samples (including 39 duplicates) and 6 NAPL samples collected between October 2004 and June 2007 as a component of the RI/FS. Additionally, 24 surface water samples associated with seeps (taken prior to 2004) are included.

The project chemical database contains 553 location records and 269732 records of analytical results including tentatively identified compounds (TICs). Additionally, the groundwater chemical table contains 125 location records and 111668 records of analytical results including TICs.

The CHEMICAL table contains historic West Canyon data which, due to the nature of this historic data, has many fields that cannot be populated. This data consists of approximately 47 locations and 5920 results.

Analytical results were provided from the specified laboratories in the form of electronic data deliverables (EDDs) for ultimate importing into the project database, as well as a signed hardcopy of the results. QA/QC procedures in compliance with the QAPP were provided for both deliverable formats. The database consists of the target analytes and the top 30 tentatively identified compounds (TICs) as defined in the June 2004 *RI/FS Work Plan* QAPP as well as sample location data.

TICs are possibly (tentatively) identified compounds and therefore the appropriateness of assigning a CAS number from the CAS registry may be questionable due to the uncertainty in the TIC name assignment and may not be in keeping with standard practice. Therefore a project specific "CAS" number was assigned to each of these in order to be responsive to and comply with EPA's request. A field, TIC\_CAS, has been added to the database structure to track the CAS number for the TIC name. Additional fields have been added to the structure to track percent match and retention times of TICs which were either received at level IV validation or were EPA split samples.

Certain constituents, such as 1,2,4 Trichlorobenzene, were reported as TICs previously while they also appeared as target compounds. This is due to the laboratory reporting these compounds as TICs under laboratory methods for which they were not target compounds. Since the target analyte results are more reliable, the TIC reporting of these data have been removed.

Only 88 of the 96 EPA split samples report results in the database. The remaining eight split sample results from the laboratory show that no TICs were reported. Additionally, laboratories reported 0 percent match and 0.00 retention times on numerous TIC results. These results were added to the database as reported from the lab. Subsequently, the TICs that met the criteria during the level IV validation were removed from the database as directed by the data validator.

Based on a review of selected hard copy lab reports, two sample results were changed in the database and the following discrepancies were noted:

- The groundwater database which has been used since 1998 for the Casmalia groundwater project does not track the batch IDs. In order to populate the RI project data structure the sample delivery group (SDG) has been used for groundwater samples in the BatchID field. The groundwater database structure has been updated to allow future samples to track this item;
- Initial Sequoia lab results reported an elongated analyte list for the first sample delivery group. The CSC contacted the lab and they resubmitted the EDD with the originally requested analyte list. The database contains results for only those analytes which were tested under the RI Work Plan. Please note that extraneous analytes reported in these early hard copies, which are not found in the database, were non-detects;
- The SDG and Batch IDs were not included in the database for soil vapor samples. The database has been updated to reflect this information; and
- Database values for method detection limits (MDLs) sometimes vary from hard copy reports due to re-analyses and re-reporting by the laboratories not incorporated into the hard copy reports.

Additional updates to the database are documented in the "README.pdf" file found on the database CD.

## 2.0 DATA VALIDATION SUMMARY

The CSC reviewed and validated the site data in conformance with the Quality Assurance and Quality Control (QA/QC) Program outlined in the project QAPP (Appendix B of the June 2004 *RI/FS Work Plan*). Data validation was performed on all sample results received prior to submittal of this RI Report in accordance with validation procedures in the June 2004 *RI/FS Work Plan* QAPP.

Sequoia Analytical, in Petaluma, and Morgan Hill, California analyzed the soil samples for all analyses with the exception of EPA 8290, and amenable cyanide. BC Laboratories, Inc, in Bakersfield, California analyzed the water samples for all the analyses with the exception of the EPA 8290 analysis. EPA 8290 analyses were performed by Columbia Analytical Services, in Houston, Texas for the soil, and sediment samples, and Frontier Analytical Laboratory, in El Dorado Hills, California for water samples (the data validation for EPA 8290 is included in a separate memorandum). In addition, Sequoia Analytical and Columbia Analytical Services, in Kelso, Washington analyzed the sediment samples for all other analyses. Del Mar Analytical, in Irvine, California analyzed four soil samples for amenable cyanide. Air Toxics Ltd., in Folsom, California analyzed soil vapor samples for all primary analyses

The laboratory results were obtained as Level III and Level IV data packages, depending on the type of samples. The data were reviewed in accordance with URS Standard Operating Procedures and the principles presented in the *USEPA National Functional Guidelines for Laboratory Data Review, Organics* (USEPA, 1999), *USEPA National Functional Guidelines for Laboratory Data Review, Inorganics* (USEPA, 2004), *USEPA National Functional Guidelines for Low Concentration Organic Data Review* (USEPA, 2001), and *USEPA CLP National Functional Guidelines for Dioxin/Furan Data Validation* (USEPA, 2005).

Data validation ensures the validity of the laboratory analytical results. Valid data is defined as results that are generated within the instrument and quality controls parameters. Data validation procedures are designed to identify valid data and to assign qualifiers that indicate limited usability of other data. The data qualifiers are incorporated into the database along with the sample results.

This section summarizes the findings of the validation (10% full, 90% limited) of 929 soil samples (including 76 field duplicates); 90 sediment samples (including 8 field duplicates); 49 primary soil vapor samples (including 4 field duplicates); and 19 surface water samples (including 1 field duplicate). These samples were collected between July 19, 2004 and November 6, 2007 as part of the Remedial Investigation/ Feasibility Study (RI/FS) Component of Work for the Casmalia Resources Superfund Site.

The analytical data set generated as part of this RI/FS study, as qualified, with the exception of the rejected data (0.75%), is considered to be usable for meeting project objectives. As such, the analytical completeness, defined as the ratio of the number of valid analytical results (valid analytical results include values qualified as estimated) to the total number of analytical results requested on samples submitted for analysis, for the project is 99.25%. Additionally, because all samples in this data set were collected and analyzed under similar prescribed conditions, the data within this set are considered to be comparable. Qualifiers assigned to the data from the data validation procedures are given below.

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Data flagged as estimated (J) in the database are considered at their estimated values for spatial distribution analyses in this report. Data flagged as estimated non-detect (UJ) in the database are considered as non-detect values for spatial distribution analyses in this report. Data flagged as rejected (R) in the database are considered non data records. They are not considered in the spatial distribution analyses in this report.

Additional evaluation was conducted for four compounds that exhibited a significant amount of qualification. These compounds are hexachlorophene, 4,6-Dinitro-2-sec-butylphenol, 1-butanol, and 2-propanol. The result of the additional data evaluation is presented in Section 8 – Additional Data Validation. Attachment R-1 presents summary tables presenting the amount of qualified data by Study Area.

### 3.0 Data Review Narratives for Soil Samples

929 soil samples were collectively analyzed for volatile organic compounds (EPA method 8260B), organochlorine pesticides, and PCBs (EPA 8081A/8082), semivolatile organic compounds (EPA method 8270C), chlorinated herbicides (EPA method 8151A), polychlorinated dibenzodioxins (PCDDs) and polychlorinated dibenzofuransdioxins (PCDFs) (EPA 8290), organophosphorous pesticides (EPA method 8141A), metals (EPA methods 6010B, 6020, 7471A), PCB congeners (EPA 1668 and 1668 modified), polynuclear aromatic hydrocarbons (EPA 8270SIM), TPH-gasoline/BTEX (EPA methods 8015M/8021), TPH-diesel/TPH-motor oil (EPA 8015M), cyanide (EPA 9010B), amenable cyanide (EPA method 335.2), pH (EPA method 9045C), and TOC (Walkley Black method).

Seventy-six (76) soil field duplicate samples (8.2% of the primary soil samples) were collected in order to assess the overall sampling and analytical precision. This is slightly lower than the 10% requirement specified in the RI-FS work plan. However approximately 98.9% of the field duplicate results satisfied the applicable field duplicate evaluation criteria. Since the overwhelming majority of the duplicate results met duplicate evaluation criteria, the lower than desired duplicate collection was insignificant to the overall project objectives. As such, the overall precision (sampling and analytical precision) is acceptable and the data quality objectives of the project are met.

In addition a subgroup of samples were analyzed for a comprehensive list of analytes that include the RCRA Appendix IX compounds, and poor purging organic compounds presented in QAPP tables B3-C, B3-D, and B-16 (RI/FS Work Plan, Appendix B – CSC, 2004). The results for all soil samples were adjusted for dry weight. Full validation including recalculation was performed on approximately 10% of the laboratory data. The balance of the data underwent limited validation. The laboratory data were reviewed to evaluate compliance with this method, and the quality of the data reported. The following summarizes the results of this review.

Ten percent (10%) of the soil data were randomly selected to perform level IV data validation with preference toward SDGs with higher number of samples per analysis. In addition, data from all laboratories were included in the level IV data validation.

The areas of review are listed below. Those areas that were only reviewed as part of full validation & recalculation are designated below. A leading check mark (✓) indicates an area of review in which all data were acceptable. A preceding crossed circle (⊗) signifies areas where issues were raised during the course of the validation review and should be considered to determine any impact on data quality and usability.

- ✓ Overall Assessment
- ✓ Data Completeness
- ⊗ Holding Times and Preservation
- ⊗ Blanks
- ⊗ Calibration (Full Validation)
- ✓ Internal Standards (Full Validation)
- ✓ Instrument Performance Check Sample (Full Validation)
- ✓ Interference Check Samples (Full Validation)
- ⊗ System Monitoring Compounds (Surrogates)

- ⊗ Laboratory Control Samples
- ⊗ Matrix Spike/Matrix Spike Duplicate Samples
- ⊗ Column Agreement
- ⊗ Field Duplicates
- ✓ Labeled Standards (EPA 8290)
- ✓ Cleanup Standards (EPA 8290)
- ✓ Compound Identification

### **3.1 Overall Assessment**

The soil data reported in this package, as qualified (with the exception of the rejected data), are considered to be usable for meeting project objectives. All except 0.75% of the data are considered to be valid; the analytical completeness for the project is 99.25%. Additionally, because all samples in this data set were collected and analyzed under similar prescribed conditions, the data within this set are considered to be comparable.

### **3.2 Data Completeness**

All analyses were performed as requested on the chain-of-custody records.

### **3.3 Holding Times and Preservation**

Approximately one percent (1.46%) of the soil data were qualified due to holding time violations, of which less than 1.8% of the qualified data were rejected for EPA 8081A/8082, and amenable cyanide analyses. All other analyses were performed within each method-specified holding times.

In addition, all cooler temperatures upon receipt were within the recommended  $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$  acceptance range. Data qualification on the basis of sample receipt was not required.

### **3.4 Blanks**

#### **3.4.1 Preparation (Method) Blank**

Method blanks were analyzed at the proper frequency for each method for the number and types of samples analyzed. Several target analytes were detected in method blanks for EPA 6020B, 8015/8021B, 8151A, 8260B, 8270C, and 8270SIM. Consequently low level results for less than one percent of the data (0.43%) were qualified as non-detect (U) to reflect the potential high bias.

#### **3.4.2 Field Blanks**

Trip Blanks - No target analytes were detected in the trip blanks with the following exception. Less than one percent of the data (0.01%) were qualified as non-detect (U) to reflect the potential high bias. Thus, with the above exception, there is no evidence of cross-contamination occurring during sample storage and shipment.

Equipment Blanks – Several target analytes for EPA 6020, 8151, 8260, and 8270C were reported as present in the equipment blanks. Consequently low level results for less than one

percent of the data (0.06%) were qualified as non-detect (U) to reflect the potential high bias. With the above exceptions, for the vast majority of the samples there is no evidence of cross-contamination resulting from equipment decontamination by the sampling team.

### **3.4.3 Calibration Blanks (CB) – (Full Validation- Metals Analysis)**

Several target analytes were detected in the associated initial calibration blanks (ICBs), and continuing calibration blanks (CCBs). Data qualification was not considered necessary because the sample concentrations were greater than five times the blank concentrations or were “non-detect”.

## **3.5 Calibrations – (Full Validation)**

### **3.5.1 Initial Calibrations (IC)**

Appropriate initial calibrations were performed for each analyte for each method. The criteria for initial calibration standard Relative Response Factors (RRFs) of greater or equal to 0.01, and percent Relative Standard Deviation (%RSD) of less or equal to 50% were utilized for EPA 8260B, and 8270C analytes exhibiting poor response. In addition, for some analytes linear or quadratic calibration curves (not forced through zero) were necessary. For all these analytes the correlation coefficient “r” was equal or greater than 0.995. Less than one percent of the data (0.56%) were qualified, of which approximately 66% were rejected for the target analytes 1-butanol, and 1-propanol.

### **3.5.2 Initial, and Continuing Calibration Verifications (ICV, CCV)**

The continuing calibration evaluation criteria were satisfied for all initial calibration verification (ICVs), and continuing calibration verification (CCVs) for all parameters with the following exception. Less than one percent (0.05%) of the data for hexachlorophene were rejected (R), and less than one percent of the data for several EPA 8270C analytes were qualified as estimated (UJ).

## **3.6 Internal Standards (Full Validation – Organic Analyses)**

The sample internal standard retention times are within  $\pm 30$  seconds of the associated continuing calibration internal standard retention time. All internal standard area counts were within the acceptance criteria ( $\geq 50\%$  and  $\leq 200\%$ ) of the associated continuing calibration internal standard area counts with the exception of approximately 0.004% that were estimated.

## **3.7 Instrument Performance Check Sample (Full Validation – Organic Analyses)**

An instrument performance check sample was analyzed at the beginning of each 12-hour period during sample analysis for the EPA 8260B, and 8270C. The performance check samples met all ion abundance criteria. In addition, all samples were analyzed within the associated 12-hour periods, with the exception of two samples for the EPA 8260B analysis. The two samples were analyzed slightly after the 12-hour period, and for these two samples EPA 8260B data was qualified as estimated (J).

### **3.8 Interference Check Samples (ICS; Full Validation – Metals Analyses)**

The analyte recoveries in the ICS solutions were within the acceptance range and data qualification was not necessary.

### **3.9 System Monitoring Compounds (Surrogates)**

An appropriate number of surrogate compounds were spiked into each sample for the EPA 8260B, 8270SIM, 8081A, 8082, 8151A, 8141A, and 8270C analyses. The recoveries of several surrogate compounds were outside of the laboratory's statistically determined acceptance ranges. Consequently, the results for approximately one percent (0.78%) of the data were qualified, of which 6.3% of the qualified data for EPA 8151A, 8270SIM, and 8015BM were rejected (R) due to low surrogate recoveries. All of the rejected results were reported as "non-detect".

With the above exceptions, all surrogate compounds recoveries were within the laboratory's statistically determined acceptance ranges.

### **3.10 Laboratory Control Samples (LCS)**

LCSs were prepared and analyzed for the applicable analyses at the proper frequency. The recoveries of all spiked analytes, and the relative percent differences (RPDs) between the LCS/LCSD results were within the laboratory's acceptance ranges with the following exceptions. Approximately one percent (1.0%) of the data were qualified, of which 17% of the qualified data were rejected (R) due to low LCS recoveries. All of the rejected results were reported as "non-detect" and were associated with methods 8081A, 8082, 8141A, 8151A, 8270, and 8270SIM.

With the exceptions above, the recoveries of all spike analytes were within the laboratory's statistically determined acceptance ranges. These LCS results indicate that the level of accuracy demonstrated by each analytical method with respect to clean sample matrices is acceptable.

### **3.11 Matrix Spike/Matrix Spike Duplicate (MS/MSD)**

Appropriate numbers of project samples were utilized for the MS/MSD analyses for each method. The recoveries of all spiked analytes, and the RPDs between the MS/MSD results were within the laboratory's statistically determined acceptance ranges with the following exceptions. Approximately three percent (3.8%) of the data were qualified, of which 5.5% of the qualified data were rejected (R). All of the rejected results were reported as "non-detect" and were associated with methods 8015BM, 8081A, 8082, 8141A, 8151A, 8270, antimony and 8270SIM.

With the above exceptions, the MS/MSD results for all analyses indicate that acceptable levels of accuracy and precision were attained with respect to the site-specific soil matrix.

### **3.12 Column Agreement (Full Validation – Organic Analyses)**

Percent differences between the two columns for all applicable analyses indicated acceptable precision for all analytes with the following exception. Less than one percent (0.001%) of the data were qualified as estimated (UJ/J) due to poor column agreement.

### **3.13 Field Duplicates**

Seventy-five blind field duplicate samples were submitted to the laboratories. Results for field duplicate sample analyses were compared to the following concentration-dependent evaluation criteria. For analytes in which both the sample and the field duplicate results are greater than the reporting limit (RL), the RPD between the results was compared to a criterion of <100%. For analytes in which either the sample or field duplicate concentration is <RL, the RL is used for calculation purposes. Approximately 0.17% of the data were qualified as estimated (J/UJ) due to duplicate imprecision. Of the positive target analyte results reported from either the duplicate sample or the primary sample, 99.8% of the results meet the ≤100 percent RPD criterion.

In general, the field duplicate results indicate that the overall precision (sampling and analytical precision) is acceptable. The field duplicate results also suggest that the samples collected can be considered representative of the medium sampled.

### **3.14 Labeled Standards**

The labeled standards employed by the laboratory (EPA 8290) are appropriate to the method. All labeled standard recoveries met the laboratory's acceptance criteria.

### **3.15 Cleanup Standards**

The cleanup standard 37C1-2, 3, 7, 8-TCDD is appropriate to the method (EPA 8290). All cleanup standard recoveries met the laboratory's acceptance criteria.

### **3.16 Compound Identification**

All detection limits comply with project specifications as provided in the QAPP.

In addition, all dilutions performed were appropriate, and the data met QC criteria established in the QAPP.

Approximately 10% of the data review process included result recalculation and transcription error checking from the raw data. All results reported by the laboratory were confirmed or corrected.

## 4.0 DATA REVIEW NARRATIVES FOR SEDIMENT SAMPLES

90 sediment samples were collectively analyzed for volatile organic compounds (EPA method 8260B), organochlorine pesticides, and PCBs (EPA 8081A/8082), semivolatile organic, compounds (EPA method 8270C), polychlorinated dibenzodioxins (PCDDs) and polychlorinated dibenzofuransdioxins (PCDFs) (EPA 8290), chlorinated herbicides (EPA method 8151A), PCB congeners (EPA 1668 and 1668 modified), organophosphorous pesticides (EPA method 8141A), metals (EPA methods 6010B, 6020, 7471A), Acid Volatile Sulfides/SEM (EPA Method 821/R-91-100), polynuclear aromatic hydrocarbons (EPA 8270SIM), cyanide (EPA 9010B), amenable cyanide (EPA method 335.2), pH (EPA method 9045), and TOC (Walkley Black method). In addition a subgroup of samples were analyzed for a comprehensive list of analytes that include the RCRA Appendix IX compounds, and poor purging organic compounds presented in QAPP tables B3-C, B3-D, and B-16 (RI/FS Work Plan, Appendix B – CSC, 2004). The results for all sediment samples were adjusted for dry weight. Full validation including recalculation was performed on approximately 10% of the laboratory data. The balance of the data underwent limited validation. The laboratory data were reviewed to evaluate compliance with this method, and the quality of the data reported. The following summarizes the results of this review.

10% of the sediment data were randomly selected to perform level IV data validation with preference toward SDGs with higher number of samples per analysis. In addition, data from all laboratories were included in the level IV data validation.

The areas of review are listed below. Those areas that were only reviewed as part of full validation & recalculation are designated below. A leading check mark (✓) indicates an area of review in which all data were acceptable. A preceding crossed circle (⊗) signifies areas where issues were raised during the course of the validation review and should be considered to determine any impact on data quality and usability.

- ✓ Overall Assessment
- ✓ Data Completeness
- ⊗ Holding Times and Preservation
- ⊗ Blanks
- ⊗ Calibration (Full Validation)
- ✓ Internal Standards (Full Validation)
- ✓ Instrument Performance Check Sample (Full Validation)
- ✓ Interference Check Samples (Full Validation)
- ⊗ System Monitoring Compounds (Surrogates)
- ⊗ Laboratory Control Samples
- ⊗ Matrix Spike/Matrix Spike Duplicate Samples
- ✓ Column Agreement (Full Validation)
- ⊗ Field Duplicates
- ✓ Labeled Standards (EPA 8290)
- ✓ Cleanup Standards (EPA 8290)
- ✓ Compound Identification

## **4.1 Overall Assessment**

The data reported in this package, as qualified (with the exception of the rejected data), are considered to be usable for meeting project objectives. All except 0.43% of the data are considered to be valid; the analytical completeness for the project is 99.6%. Additionally, because all samples in this data set were collected and analyzed under similar prescribed conditions, the data within this set are considered to be comparable.

## **4.2 Data Completeness**

All analyses were performed as requested on the chain-of-custody records.

## **4.3 Holding Times and Preservation**

Approximately twelve percent (11.6%) of the sediment data were qualified as estimated due to holding time violations for target analytes associated with EPA 8151A, 8270C, 9010B, and pH. Holding times were grossly exceeded for fourteen sediment samples for the EPA 8151A analyses performed by Columbia Analytical. However data was accepted as estimated (UJ) since prior analyses by Sequoia confirmed the results.

In addition, all cooler temperatures upon receipt were within the recommended  $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$  acceptance range with the following exception. The cooler temperature for fourteen sediment samples was outside of the recommended acceptance range upon arrival at Columbia Analytical in Kelso, Washington. Consequently, the data for all semivolatile analyses performed by Columbia was appropriately qualified as estimated (J/UJ).

## **4.4 Blanks**

### **4.4.1 Preparation (Method) Blank**

Method blanks were analyzed at the proper frequency for each method for the number and types of samples analyzed. Several target analytes were detected in method blanks for EPA 6020B, 8260B, and 8270C. Consequently low-level results for approximately 0.45% of the data were qualified as non-detect (U) to reflect the potential high bias.

### **4.4.2 Field Blanks**

Trip Blanks - No target analytes were detected in the vast majority of the trip blanks. Only six data records (less than 0.03%) for EPA 8260B were qualified as non-detect (U) to reflect the potential high bias. Thus, with the above exception, there is no evidence of cross-contamination occurring during sample storage and shipment. Data qualification on the basis of the trip blank results was not necessary.

Equipment Blanks – No target analytes were detected in the vast majority of the equipment blanks. Only 0.22% of the data for EPA 8260B, and 6020 were qualified as non-detect (U) to reflect the potential high bias. With the above exceptions, there is no evidence of cross-contamination resulting from equipment decontamination by the sampling team.

#### **4.4.3 Calibration Blanks (CCB) – (Full Validation- Metals analyses)**

Several target analytes were detected in the associated ICBs, and CCBs. Data qualification was not considered necessary because the sample concentrations were greater than five times the blank concentrations, or were “non-detect”.

#### **4.5 Calibrations (Full Validation)**

##### **4.5.1 Initial Calibrations (IC)**

Appropriate initial calibrations were performed for each analyte for each method. The criteria for initial calibration standard RRFs of greater or equal to 0.01, and %RSD of less or equal to 50% were utilized for EPA 8260B, and 8270C analytes exhibiting poor response. Less than one percent (0.06%) of the semivolatile organics data were qualified as estimated.

##### **4.5.2 Initial, and Continuing Calibration Verifications (ICV, CCV)**

The continuing calibration evaluation criteria were satisfied for all ICVs, and CCVs for all parameters with the following exception. Less than one percent (0.04%) of the data for hexachlorophene were rejected (R), and less than one percent of the data for several analytes were qualified as estimated (UJ).

#### **4.6 Internal Standards (Full Validation – Organic Analyses)**

The sample internal standard retention times are within  $\pm 30$  seconds of the associated continuing calibration internal standard retention time. All internal standard area counts were within the acceptance criteria ( $\geq 50\%$  and  $\leq 200\%$ ) of the associated continuing calibration internal standard area counts.

#### **4.7 Instrument Performance Check Sample (Full Validation – Organic Analyses)**

An instrument performance check sample was analyzed at the beginning of each 12-hour period during sample analysis for the EPA 8260B and 8270C. The performance check samples met all ion abundance criteria. In addition all samples were analyzed within the associated 12-hour periods.

#### **4.8 Interference Check Samples (ICS; Full Validation – Metals Analyses)**

The analyte recoveries in the ICS solutions were within the acceptance range and data qualification was not necessary.

#### **4.9 System Monitoring Compounds (Surrogates)**

An appropriate number of surrogate compounds were spiked into each sample for the EPA 8260B, 8270SIM, 8081A, 8082, 8151A, 8141A, and 8270C analyses. The recoveries of all surrogate compounds were within the laboratories statistically determined acceptance ranges with the following exceptions. The results for less than one percent (0.21%) of the data were qualified as estimated (J/UJ) for EPA 8151A due to low surrogate recoveries.

With the above exception, all surrogate compounds recoveries were within the laboratory's statistically determined acceptance ranges.

#### **4.10 Laboratory Control Samples (LCS)**

LCSs were prepared and analyzed for the applicable analyses at the proper frequency. The recoveries of all spiked analytes, and the RPDs between the LCS/ LCSD results were within the laboratory's acceptance ranges with the following exceptions. Approximately less than one percent (0.6%) of the data were qualified, of which 1.7% of the qualified data were rejected (R) due to low LCS recoveries. All of the rejected results were reported as "non- detect" and were associated with methods 8141A, and 8151A.

With the exceptions above, the recoveries of all spike analytes were within the laboratory's statistically determined acceptance ranges. These LCS results indicate that the level of accuracy demonstrated by each analytical method with respect to clean sample matrices is acceptable.

#### **4.11 Matrix Spike/Matrix Spike Duplicate (MS/MSD)**

Appropriate numbers of project samples were utilized for the MS/MSD analyses for each method. The recoveries of all spiked analytes, and the RPDs between the MS/ MSD results were within the laboratory's statistically determined acceptance ranges with the following exceptions. Approximately three percent (3.4%) of the data were qualified, of which 1.0% of the qualified data were rejected (R). All of the rejected results were reported as "non- detect" and were associated with methods 8081A, 8151A, and 8141A.

With the above exceptions, the MS/MSD results for all analyses indicate that acceptable levels of accuracy and precision were attained with respect to the site-specific soil matrix.

#### **4.12 Column Agreement (Full Validation – Organic Analyses)**

Percent differences between the two columns for all applicable analyses indicated acceptable precision for all analytes.

#### **4.13 Field Duplicates**

Eight blind field duplicate samples were submitted to the laboratories for analyses. The duplicate samples were evaluated by calculating the RPD between the primary sample and its duplicate. The RPD was calculated only for those constituents that were detected in either sample at levels above or near the reporting limits. Precision acceptance criterion is a RPD of  $\leq 100$  percent. Results for all analytes satisfied the applicable evaluation criteria with the exception of 0.02% of the data.

In general, the field duplicate results indicate that the overall precision (sampling and analytical precision) is acceptable. The field duplicate results also suggest that the samples collected can be considered representative of the medium sampled.

**4.14 Labeled Standards**

The labeled standards employed by the laboratory (EPA 8290) are appropriate to the method. All labeled standard recoveries met the laboratory's acceptance criteria.

**4.15 Cleanup Standards**

The cleanup standard 37C1-2, 3, 7, 8-TCDD is appropriate to the method (EPA 8290). All cleanup standard recoveries met the laboratory's acceptance criteria.

**4.16 Compound Identification**

All detection limits comply with project specifications as provided in the QAPP.

In addition, all dilutions performed were appropriate, and the data met QC criteria established in the QAPP.

Approximately 10% of the data review process included result recalculation and transcription error checking from the raw data. All results reported by the laboratory were confirmed or corrected.

## 5.0 DATA REVIEW NARRATIVES FOR SURFACE WATER SAMPLES

Nineteen surface water samples were collectively analyzed for a comprehensive list of analytes that include the RCRA Appendix IX compounds, and poor purging organic compounds presented in QAPP table B-16 by EPA methods 8290, 8270C, 8270SIM, 8260B, 8081A, 8082, 6010B, 7470A, 200.8, 504.1, 8151A, 8141A, 8015M, and 9012. Full validation including recalculation was performed on approximately 10% of the laboratory data. The balance of the data underwent limited validation. The laboratory data were reviewed to evaluate compliance with these methods, and the quality of the data reported. The following summarizes the results of this review.

10% of the water data were randomly selected to perform level IV data validation with preference toward SDGs with higher number of samples per analysis. In addition, data from all laboratories were included in the level IV data validation.

The areas of review are listed below. Those areas that were only reviewed as part of full validation & recalculation are designated below. A leading check mark (✓) indicates an area of review in which all data were acceptable. A preceding crossed circle (⊗) signifies areas where issues were raised during the course of the validation review and should be considered to determine any impact on data quality and usability.

- ✓ Overall Assessment
- ⊗ Data Completeness
- ⊗ Holding Times and Preservation
- ⊗ Blanks
- ⊗ Calibration (Full Validation)
- ✓ Internal Standards (Full Validation)
- ✓ Instrument Performance Check Samples (Full Validation)
- ✓ Interference Check Samples (Full Validation)
- ⊗ System Monitoring Compounds (Surrogates)
- ✓ Laboratory Control Samples
- ⊗ Matrix Spike/Matrix Spike Duplicate Samples
- ✓ Column Agreement (Full Validation)
- ⊗ Field Duplicates
- ✓ Labeled Standards (EPA 8290)
- ✓ Cleanup Standards (EPA 8290)
- ✓ Compound Identification

### 5.1 Overall Assessment

The data reported in this package, as qualified (with the exception of the rejected data), are considered to be usable for meeting project objectives. All except 0.23% of the data are considered to be valid; the analytical completeness for the project is approximately 99.8%. Additionally, because all samples in this data set were collected and analyzed under similar prescribed conditions, the data within this set are considered to be comparable.

## **5.2 Data Completeness**

All analyses were performed as requested on the COCs with the exception of EPA 8270C analysis for n-nitrosodimethylamine (NDMA). The laboratory failed to analyze NDMA for project samples SWS00001, SWS00010, SWS00012, SWS00013, and SWS00014.

## **5.3 Holding Times and Preservation**

Less than one percent (0.5%) of the surface water data were qualified as estimated (UJ) due to holding time violations for NDMA and target analytes associated with EPA 8141A. No data were rejected due to holding time violations. All other analyses were performed within each method-specified holding times.

In addition, all cooler temperatures upon receipt were within the recommended  $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$  acceptance range. Data qualification on the basis of sample receipt was not required.

## **5.4 Blanks**

### **5.4.1 Preparation (Method) Blank**

Method blanks were analyzed at the proper frequency for each method for the number and types of samples analyzed. Low-level results for approximately 0.31% of the data were qualified as non-detect (U) to reflect the potential high bias. The qualified data were associated with EPA method 6010B.

### **5.4.2 Field Blanks**

Trip Blanks - No target analytes were detected in the vast majority of the trip blanks. Low-level results for two EPA 8015M analytes (Less than 0.01% of the data) were qualified as non-detect (U) to reflect the potential high bias. Thus, with the above exception, there is no evidence of cross-contamination occurring during sample storage and shipment.

Equipment Blanks – No target analytes were detected in the vast majority of the equipment blanks. Only thirty-eight results for EPA 6010B, 7470A, 8270SIM (less than 1.2% of the data) were qualified as non-detect (U) to reflect the potential high bias. With the above exceptions, there is no evidence of cross-contamination resulting from equipment decontamination by the sampling team.

### **5.4.3 Calibration Blanks (ICB, CCB) – (Full Validation - Metals Analyses)**

Several target analytes were detected in the associated ICBs, and CCBs. Data qualification was not considered necessary because the sample concentrations were greater than five times the blank concentrations or non-detect.

## **5.5 Calibrations (Full Validation)**

### **5.5.1 Initial Calibrations (IC)**

Appropriate initial calibrations were performed for each analyte for each method. The criteria for initial calibration standard RRFs of greater or equal to 0.01, and %RSD of less or equal to 50% were utilized for EPA 8260B, and 8270C analytes exhibiting poor response. Less than one percent (0.83%) of the data were qualified as estimated (UJ). The qualified data were associated with EPA method 8270C.

### **5.5.2 Initial, and Continuing Calibration Verification (ICV, CCV)**

The ICV, and CCV calibration evaluation criteria were satisfied for all parameters with the following exceptions. Approximately one percent (0.97%) of the data were qualified as estimated (UJ). The qualified data were associated with EPA methods 8081A, 8260B, and 8270C.

## **5.6 Internal Standards (Full validation – Organic Analyses)**

The sample internal standard retention times are within  $\pm 30$  seconds of the associated continuing calibration internal standard retention time. All internal standard area counts were within the acceptance criteria ( $\geq 50\%$  and  $\leq 200\%$ ) of the associated continuing calibration internal standard area counts.

## **5.7 Instrument Performance Check Samples (Full Validation – Organic Analyses)**

An instrument performance check sample was analyzed at the beginning of each 12-hour period during sample analysis for the EPA 8260B, 8270C, and 8270SIM. The performance check samples met all ion abundance criteria. In addition, all samples were analyzed within the associated 12-hour periods.

## **5.8 Interference Check Samples (ICS; Full Validation – Metals Analyses)**

The analyte recoveries in the ICS solutions were within the acceptance range and data qualification was not necessary.

## **5.9 System Monitoring Compounds (Surrogates)**

An appropriate number of surrogate compounds were spiked into each sample for the EPA 8260B, 8270SIM, 8081A, 8082, 8151A, 8141A, and 8270C analyses. The recoveries of all surrogate compounds were within the laboratories statistically determined acceptance ranges with the following exceptions. The results for approximately 5.8% of the data were qualified as estimated (J/UJ) for EPA 8081A, 8082 due to low surrogate recoveries.

With the above exception, all surrogate compounds recoveries were within the laboratory's statistically determined acceptance ranges.

### **5.10 Laboratory Control Samples (LCS)**

LCSs were prepared and analyzed for the applicable analyses at the proper frequency. The recoveries of all spiked analytes, and the RPDs between the LCS/ LCSD results were within the laboratory's acceptance. These LCS results indicate that the level of accuracy demonstrated by each analytical method with respect to clean sample matrices is acceptable.

### **5.11 Matrix Spike/Matrix Spike Duplicate (MS/MSD)**

Appropriate numbers of project samples were utilized for the MS/MSD analyses for each method. The recoveries of all spiked analytes, and the RPDs between the MS/ MSD results were within the laboratory's statistically determined acceptance ranges with the following exceptions. Approximately one percent (1.1%) of the data were qualified, of which 3.8% of the qualified data were rejected (R). All of the rejected results were reported as "non-detect" and were associated with method 8015M.

With the above exceptions, the MS/MSD results for all analyses indicate that acceptable levels of accuracy and precision were attained with respect to the site-specific aqueous matrix.

### **5.12 Column Agreement (Full Validation – Organic Analyses)**

Percent differences between the two columns for all applicable analyses indicated acceptable precision for all analytes.

### **5.13 Field Duplicates**

One blind field duplicate sample was submitted to the laboratories for analyses. Results for field duplicate sample analyses were compared to the following concentration-dependent evaluation criteria. For analytes in which both the sample and the field duplicate results are greater than the reporting limit (RL), the RPD between the results was compared to a criterion of <50% for water samples. For analytes in which either the sample or field duplicate concentration is <RL, the RL is used for calculation purposes. In general, the field duplicate results indicate that the overall precision (sampling and analytical precision) is acceptable. The field duplicate results also suggest that the samples collected can be considered representative of the medium sampled.

### **5.14 Labeled Standards**

The labeled standards employed by the laboratory (EPA 8290) are appropriate to the method. All labeled standard recoveries met the laboratory's acceptance criteria.

### **5.15 Cleanup Standards**

The cleanup standard 37C1-2, 3, 7, 8-TCDD is appropriate to the method (EPA 8290). All cleanup standard recoveries met the laboratory's acceptance criteria.

**5.16 Compound Identification**

All detection limits comply with project specifications as provided in the QAPP.

In addition, all dilutions performed were appropriate, and the data met QC criteria established in the QAPP.

Approximately 10% of the data review process included result recalculation and transcription error checking from the raw data. All results reported by the laboratory were confirmed, and/or corrected.

## 6.0 DATA REVIEW NARRATIVES FOR SOIL VAPOR SAMPLES

A Level III QC validation was performed on 100% of the soil vapor data by Geosyntec Consultants. After the Level III QC validation was completed, a Level IV validation was done on 10% of the data. Forty-nine primary soil vapor samples were collectively analyzed for volatile organic compounds (EPA method TO-15) and isobutane (ASTM method D-1495). Air Toxics Ltd., in Folsom, CA analyzed the soil vapor samples for all analyses.

The areas of review are listed below. Those areas that were only reviewed as part of full validation and recalculation are designated below. A leading check mark (✓) indicates an area of review in which all data were acceptable. A preceding crossed circle (⊗) signifies areas where issues were raised during the course of the validation review and should be considered to determine any impact on data quality and usability.

- ✓ Data Completeness
- ⊗ Holding Times
- ✓ Blanks
- ⊗ Calibration (full Validation)
- ✓ Internal Standards
- ✓ Performance Check Sample
- ✓ System Monitoring Compounds (Surrogates)
- ⊗ Laboratory Control Samples
- ✓ Matrix Spike/Matrix Spike Duplicate Samples
- ⊗ Field Duplicates
- ⊗ Compound Identification

### 6.1 Data Completeness

All analyses were performed as requested on the chain-of-custody records.

### 6.2 Holding Times

One sample was qualified due to a missed holding time. The holding time was missed by one day resulting in all positive results for the sample being J qualified as estimated and the non-detect results being UJ qualified as estimated below the reporting limit. One other sample was reanalyzed at a dilution one day past the holding time leading to the J qualification of 17 compounds as estimated and the non-detect compounds as UJ qualified estimated less than the reporting limit. Qualifications based on missed holding times were less than 5% (4.6%), no other data were qualified and none of the data were rejected due to holding time violations.

### 6.3 Blanks

#### 6.3.1 Preparation (Method) Blank

Method blanks were analyzed at the proper frequency for each method for the number and types of samples analyzed. Data qualification on the basis of the method blank results was not necessary.

### **6.3.2 Field Blank**

Trip Blank - No target analytes were detected in the trip blank. Thus, there is no evidence of cross-contamination occurring during sample storage and shipment. Data qualification on the basis of the trip blank results was not necessary.

### **6.3.3 Calibration Blanks (CB) - Full Validation**

Calibration blanks were analyzed for the method as required. Data qualification on the basis of the calibration blank results was not necessary.

## **6.4 Calibrations (Full Validation)**

### **6.4.1 Initial Calibrations (IC)**

Appropriate initial calibrations were performed for each analyte for each method. For all target analytes, the average relative response factors (RRFs) were greater than 0.05 and the percent relative standard deviation (%RSD) was within the acceptance criteria of (+/- 30% with a method allowance of 2 compounds at +/- 40%) with the following exceptions: less than 0.5% of the data were qualified as estimated less than the reporting limit due to 4 data records affected by calibration results outside of the initial calibration criteria.

### **6.4.2 Continuing Calibration Verification (CCV)**

The continuing calibration evaluation criteria were satisfied for all parameters and data qualification were not considered necessary.

## **6.5 Internal Standards (Full validation – Organic Analyses)**

The sample internal standard retention times are within  $\pm 30$  seconds of the associated continuing calibration internal standard retention time. All internal standard area counts were within the acceptance criteria ( $\geq 50\%$  and  $\leq 200\%$ ) of the associated continuing calibration internal standard area counts.

## **6.6 Instrument Performance Check (Full Validation – Organic Analyses)**

An instrument performance check sample was analyzed at the beginning of each 24-hour period during sample analysis. The performance check samples met all ion abundance criteria. In addition all samples were analyzed within the associated 24-hour periods.

## **6.7 System Monitoring Compounds (Surrogates)**

The recoveries of all surrogate compounds were within the laboratories statistically determined acceptance ranges.

## **6.8 Laboratory Control Samples (LCS)**

LCSs were prepared and analyzed for the applicable analyses at the proper frequency. The recoveries of all spiked analytes, and the relative percent differences (RPDs) between the LCS/

LCSD results were within the laboratory's acceptance ranges with the following exceptions; less than 1 percent (0.4%) of the data were qualified because sixteen data records were qualified (UJ or J) due to low and high LCS recoveries.

With the exceptions above, the recoveries of all spike analytes were within the laboratory's statistically determined acceptance ranges. These LCS results indicate that the level of accuracy demonstrated by each analytical method is acceptable.

### **6.9 Field Duplicates**

Four field duplicate samples were submitted to the laboratories for analyses. The duplicate samples were evaluated by calculating the RPD between the primary sample and its duplicate. The RPD was calculated only for those constituents that were detected in either sample at levels above or near the reporting limits. Precision acceptance criterion is a RPD of  $\leq 100$  percent. Results for all analytes satisfied the applicable evaluation criteria with the following exception. Six data records (Less than 0.4%) were listed as estimates (J) due to the RPD values outside of acceptable criteria.

With the exceptions above, the field duplicate results indicate that the overall precision (sampling and analytical precision) is acceptable.

### **6.10 Compound Identification**

All detection limits comply with project specifications as provided in the QAPP.

In addition, all dilutions performed were appropriate with the following exceptions; five data records, less than 1% (0.2%) were J qualified as estimated because they were reported as estimated concentrations greater than the high end of the calibration range.

Approximately 10% of the data review process included result recalculation and transcription error checking from the raw data. All results reported by the laboratory were confirmed.

## 7.0 OVERALL ASSESSMENT

The sections below detail the overall assessment of the data with respect to the quantitative and qualitative data quality indicators.

### 7.1 Sensitivity

Sensitivity refers to the ability of the analytical system to attain the detection limits necessary for meeting project objectives. For samples not requiring dilutions the method detection limits obtained satisfied the requirements of the QAPP with the exception of samples requiring dilutions greater than 10x. However, for samples analyzed at a dilution of 10x or greater, one or more target analytes was detected. Thus, there are not expected to be any limitations on the use of the data with respect to the detection limits obtained.

### 7.2 Accuracy

Accuracy is defined as the degree of agreement of a measurement to an accepted reference or true value. Accuracy was measured as the percent recovery (%R) of an analyte in a reference standard or spiked sample.

#### 7.2.1 LCS Summary

As noted in the data review narratives, the vast majority of percent recoveries for laboratory control samples, approximately 99.7%, were within their respective acceptance criteria indicating that acceptable levels of accuracy were attained on clean sample matrices. Sample results associated with recoveries outside acceptance criteria were qualified as necessary. Overall, the LCS results indicated that acceptable accuracy was obtained by the method on a control sample matrix.

#### 7.2.2 Surrogate

An appropriate number of surrogate compounds was spiked into each sample for the EPA 8260B, 8270SIM, 8081A, 8082, 8151A, 8141A, and 8270C analyses. The vast majority of the surrogate recoveries, approximately 99.8% were within acceptance limits. Therefore, the surrogate compound recoveries are considered to indicate that the overall level of accuracy demonstrated on the individual sample matrices is acceptable.

#### 7.2.3 MS/MSD Summary

The vast majority of the matrix spike and matrix spike duplicate recoveries for both site-specific samples and non-site samples (approximately 99.7%) were with the laboratory determined acceptance ranges. In general, the overall level of accuracy demonstrated by the analyses is considered to be acceptable.

### **7.3 Precision**

Precision is defined as the agreement between a set of replicate measurements without assumption and knowledge of the true value. Precision of laboratory measurements was evaluated by the comparison of spike sample/spike sample duplicate results.

The vast majority of the field duplicate pair results satisfied the applicable evaluation criteria. As such, the overall level of analytical precision demonstrated is considered acceptable.

In addition, sixty-six soil duplicate samples, eight sediment duplicate samples, and one surface water duplicate sample were collected in order to assess the overall sampling and analytical precision. The vast majority (approximately 99%) of the results satisfied the applicable evaluation criteria. As such, the overall sampling and analysis precision demonstrated is considered to be acceptable.

### **7.4 Completeness**

With the exception of the rejected data (approximately 0.7%), all data was usable as qualified. As such, the analytical completeness, defined as the ratio of the number of valid analytical results (valid analytical results include values qualified as estimated) to the total number of analytical results requested on samples submitted for analysis, for the project is 99.3%. All valid results are considered usable for meeting project objectives.

### **7.5 Comparability**

Comparability expresses the confidence with which one data set can be compared to another. Comparability can be related to accuracy and precision because these quantities are measures of data reliability. Data are comparable if collection techniques, measurement procedures, method and reporting are equivalent for the samples within a sample set. As the samples in this set were analyzed in accordance with the quality assurance and quality control measures prescribed in the QAPP; and acceptable levels of overall accuracy and precision were attained, the data within these sets are considered to be comparable to each other.

### **7.6 Representativeness**

Representativeness is a qualitative parameter that expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness was evaluated by comparing the results obtained for field duplicate sample pairs. The vast majority of the results satisfied the field duplicate evaluation criteria. Thus, the samples are considered to be representative of the medium sampled.

### **7.7 Data Qualifier Definitions for Organic Analyses**

- 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 its 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.

### 7.8 Data Qualifier Definitions for Inorganic Analyses

- U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
- J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
- UJ The analyte was analyzed for, but was not detected. The reported sample quantitation limit is approximate and may be inaccurate or imprecise.
- R The data are unusable. The sample results are rejected due to serious deficiencies in meeting quality control (QC) criteria. The analyte may or may not be present in the sample.

### 7.9 Data Qualifier Definitions — Reason Code Definitions

- a Analytical sequence deficiency or omission.
- b Gross compound breakdown (4,4'-DDT/Endrin).
- c Calibration failure; poor or unstable response.
- d Laboratory duplicate imprecision.
- e Laboratory duplicate control sample imprecision.
- f Field duplicate imprecision.
- g Poor chromatography.
- h Holding time violation or temperature outside criterion.
- i Internal standard failure.
- j Poor mass spectrographic performance.
- k Serial dilution imprecision.
- l Laboratory control sample recovery failure.
- m Matrix spike/matrix spike duplicate recovery failure.
- n Interference check sample recovery failure.
- o Calibration blank contamination (metals/inorganics only).
- p Preparation blank contamination (metals/inorganics only).
- q Quantitation outside linear range.
- r Linearity failure in initial calibration.
- s Surrogate spike recovery failure (GC organics and GC/MS organics only).

#### INTERPRETATION KEY

The following example shows how an analytical result which includes qualifiers assigned by both the URS data review team and the analytical laboratory could be displayed in the data tables:

**<5.20 Uz | JB**

The qualifier assigned by the URS data review team precedes the “|”; the qualifier assigned by the laboratory follows it. In this example, the result is qualified as a non-detection data to the bias introduced by contamination of the associated method blank. Presence of the analyte in the method blank is indicated by the laboratory qualifier (B). The qualifier assigned by the URS data review team (Uz) indicates that the analyte concentration is considered to be below the adjusted detection limit (quantitation limit) based on the level of contamination in the method blank.

- 
- t Instrument tuning failure.
  - u No valid confirmation column (GC Organics only).
  - v Value is estimated below the MDA (Rads only).
  - w Retention time (RT) outside of RT window.
  - x Field blank contamination.
  - y Trip blank contamination.
  - z Method blank contamination.
  - a1 Poor agreement between columns (GC Organics only).
  - m1 Post digestion spike recovery failure (metals).
  - ii Relative intensity of major ions less than 10% of Internal Standard (TIC Data review only).

## 8.0 ADDITIONAL DATA VALIDATION

### 8.1 Introduction

This report summarizes the findings of the additional evaluation of the data associated with the Remedial Investigation/ Feasibility Study (RI/FS) Component of Work for the Casmalia Resources Superfund Site in Santa Barbara County, California. Additional evaluation was conducted for four compounds that exhibited a significant amount of qualification. Approximately an additional 20% of the data generated by Sequoia Analytical were reviewed for hexachlorophene, 4,6-Dinitro-2-sec-butylphenol, 1-butanol, and 2-propanol. The data were reviewed in accordance with URS standard operating procedures and the principles presented of *USEPA National Functional Guidelines for Laboratory Data Review, Organics* (USEPA, 1999). In addition, this section presents a summary of the Tentatively Identified Compounds (TICs).

### 8.2 Data Review Narratives

Per EPA's recommendation an additional 20% of the data for 4,6-Dinitro-2-sec-butylphenol (EPA 8151A), hexachlorophene (EPA 8270C), and 1-butanol, and 2-butanol (EPA 8260B) were reviewed to evaluate compliance with these methods, and the quality of the data reported. The following Level IV data validation procedures were applied:

- Evaluation of sample holding times;
- Instrument performance criteria;
- Standard calibrations;
- Blanks;
- Surrogates;
- QC samples, including matrix duplicate, matrix spike/matrix spike duplicate (MS/MSD), and laboratory control sample (LCS) analyses;
- Internal standard areas, where applicable;
- Target compound identification; and
- An overall assessment of the data.

Guidelines for minimum relative response factors (RRFs) were applied to volatile and semivolatile organic Appendix IX compounds. The minimum RRF QC criterion of 0.05 was lowered to 0.01 to compensate for the lower RRFs observed for some of the Appendix IX compounds in accordance with National Functional Guidelines. The data validation was performed according to the Casmalia Quality Assurance Project Plan, individual relevant SW-846 methodology, and USEPA Contract Laboratory Program (CLP) National Functional Guidelines for Organic Data Review (USEPA, 1999).

#### 8.2.1 Hexachlorophene (EPA 8270C)

Approximately 30% of the data for this compound were rejected due to low response factors in the continuing calibration verification standards (CCVs). All rejected results were reported as "non-detect". Results were rejected because there is a significant probability that, if the compound was present at or above the reporting limit, the analysis would not have detected the compound. It appears that low response factors in CCVs are due to method related problems

(i.e., the compound is very unstable and is a “poor performer” under this method). In a continuous effort to remedy this problem the laboratory periodically changed columns. It is for this reason that we recommend to review 100% of the raw data for this compound.

### **8.2.2 4,6-Dinitro-2-Sec-Butylphenol (EPA 8151A)**

Approximately 80% of the data reported as “non-detect” for this compound were rejected in response to MS/MSD and/or LCS recoveries not meeting the technical acceptance criteria. This compound was also calibrated and analyzed for as part of the EPA 8270C method. However it was not reported as part of the EPA 8270C list. The practical quantitation limit (PQL) as provided in the QAPP for EPA 8151A was compared with the PQL for EPA 8270C as provided by the laboratory. The method-specific comparisons are listed below:

EPA 8151A QAPP MDL/RL (mg/kg)	EPA 8270C Lab MDL /RL (mg/kg)
0.0038 / 0.016	0.215 / 0.330

Since the residential PRG for this compound is 61 mg/kg, the EPA 8270C results could be reported for this compound and be sufficient for use in risk assessment. It is therefore recommended to report this compound under the EPA 8270C compound list.

### **8.2.3 1-Butanol, and 2-Propanol**

No additional data were rejected. Although these compounds exhibit poor purging efficiency, none were rejected as the result of additional validation. With the exception of the previously rejected data, the data for these two compounds are considered usable in meeting project objectives.

## **8.3 Overall Assessment**

The data reported as part of this RI/FS (with the exception of the rejected data) are considered to be valid; the analytical completeness defined as the ratio of the number of valid analytical results (valid analytical results include values qualified as estimated) to the total number of analytical results requested on samples submitted for analysis, for the project is greater than 99% exceeding the project quality objectives for completeness.

The laboratory has continued to show that they are capable of analyzing these samples with a high level of confidence. The majority of the compounds were rejected due to method-related or matrix-related problems.

## **8.4 Tentatively Identified Compounds (TICs)**

As part of the Casmalia RI/FS program, for EPA 8270C and 8260B analyses, for each sample, laboratories conducted a mass spectral search of the NIST library and reported the possible identity of the unknown compounds. The identification of these compounds was not assessed by the laboratories. In general, any compound that had area or height greater than 10% of the area or height of the nearest internal standard was reported as TIC.

Upon EPA's request, TIC raw data for soil, sediment and surface water samples were reviewed by URS data validation team in accordance with the guidelines provided in National Functional

Guidelines for Laboratory Data Review, Organics (EPA, 1999). TIC data was then qualified by URS and reported as follows:

- Percent match and retention times were provided for all TICs reported.
- Target compounds that were inadvertently reported as TICs were removed.
- Common laboratory contaminants, that were also present in blanks, such as '2-Pentanone, 4-hydroxy-4-methyl', and '3-Penten-2-one, 4-methyl' were qualified as rejected (R).
- All similar isomers were reported as a total.
- Compounds with percent match of less than 85% if not rejected were reported as: unknown, unknown siloxanes, or unknown Alkanes.

Table R-1 presents the top 30 TICs reported in each matrix (soil, sediment, surface water and groundwater from the groundwater database). Since the concentrations of TICs reported are semi-quantitative and cannot be confirmed, the top 30 TICs were determined as the number of times each compound was detected in a matrix analyzed by the particular analytical method. For the most part, the TICs were detected relatively infrequently. In general, hydrocarbons, natural plant chemicals, and analytical artifacts (not present in blanks) including solvent preservatives and acetone byproducts, account for majority of the TICs.

Based on the nature of the TICs reported, it is CSC's opinion that none of these compounds should be added as target analytes or included in risk evaluation.

## 9.0 REFERENCES

Casmalia Steering Committee (CSC), June 2004 *RI/FS Work Plan*, June 2004.

USEPA, 2005. USEPA CLP National Functional Guidelines for Dioxin/Furan Data Validation. September.

USEPA, 2004. USEPA National Functional Guidelines for Laboratory Data Review, Inorganics. October.

USEPA, 2001. USEPA CLP National Functional Guidelines for Low Concentration Organic Data Review. June.

USEPA, 1999. USEPA National Functional Guidelines for Laboratory Data Review, Organics. October.