# **Data Validation Summary Report**

For

# Shallow Supplemental Soil Sampling in Areas I and II

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# Acronyms and Abbreviations

Acronym	Meaning
%D	Percent Difference
%R	Percent Recovery
BRC	Basic Remediation Company
CEM	Certified Environmental Manager
CLP	Contract Laboratory Program
DOE	Department of Energy
DQI	Data Quality Indicator
DRO	Diesel Range Organics
DUP	Duplicate
EDD	Electronic Data Deliverable
EDXA	Energy Dispersive X-ray Analysis
EPA	U.S. Environmental Protection Agency
GC/MS	Gas Chromatograph/Mass Spectrometer
GRO	Gasoline Range Organics
НСВ	Hexachlorobenzene
ICP	Inductively Coupled Plasma
LCS	Laboratory Control Sample
MDL	Method Detection Limit
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NDEP	Nevada Division of Environmental Protection
OCP	Organochlorine Pesticide
ORO	Oil Range Organics
PARCCS	Precision, Accuracy, Representativeness, Comparability, Completeness,
	and Sensitivity
PCB	Polychlorinated Biphenyl
PQL	Practical Quantitation Limit
QAPP	Quality Assurance Project Plan
QC	Quality Control
R	Rejected
RPD	Relative Percent Difference
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SQL	Sample Quantitation Limit
SVOC	Semi-Volatile Organic Compound
TCDD	Tetrachlorodibenzodioxin
TCDF	Tetrachlorodibenzofuran
Tronox	Tronox LLC
VOC	Volatile Organic Compound

## **1. Introduction**

This data validation summary report (DVSR) was prepared to summarize the results of the data validation of analytical results associated with the Tronox Shallow Supplemental Soil Sampling in Areas I and II. These data were collected in December, 2009 and analyzed by Columbia Analytical Services (CAS) using their Kelso (CASK) and Rochester (CASR) laboratories as well as Test America Sacramento (TAS).

For this sampling and analysis event 129 soil samples, each for a variety of analytical suites, were collected and analyzed. The samples are identified in the *samples* table of the EDD. Also included with this project were equipment blanks and trip blanks identified in the appendices to this report.

## **1.1.Purpose and Objectives**

The purpose of this DVSR is to provide the results from the data validation process and to summarize the results of this process on the PARCCS parameters. Data validation is used in the assessment stage of the project cycle to evaluate the completeness, correctness, and conformance against the analytical methods and procedural requirements. The discussion of impacts on the PARCCS parameters in this report further extends the evaluation to begin the process of assessing data usability.

This sampling and analysis project was completed in accordance with the Tronox memorandums dated November 19, 2009: Scope for Additional Sampling- Phase B Investigation, Area 1 and December 11, 2009: Scope for Additional Sampling – Phase B Investigation, Area II.

Field samples and the associated field QC samples were logged into the laboratories in Sample Delivery Groups (SDGs). The following types of analyses were performed:

Analytical Laboratory	SDG	Area	Analytical Methods
CASK	R0907046	1	6010B, 6020
CASK	R0907070	П	314.0
CASK	R0907146	П	314.0, 6010B, 6020, 7471a
CASK	R0907171	П	314.0, 6010B, 6020, 7471a
CASR	R0907007	1	8270c
CASR	R0907024	1	8270c
CASR	R0907046	1	8081, 8260B, 8270c
CASR	R0907057	1	8270c
CASR	R0907070	П	8015b, 8081, 8270c
CASR	R0907146	П	7199, 8015b, 8081, 8082, 8270c
CASR	R0907171	П	7199, 8015b, 8270c
CASR	R0907257	П	8270c
TAS	G9L 100559	1	8290, ASTM 2216
TAS	G9L 110588	1	8290, ASTM 2216
TAS	G9L 120491	1	8290, ASTM 2216

#### Table 1-1. Project Summary

Analytical Laboratory	SDG	Area	Analytical Methods
TAS	G9L 160493	1	8290, ASTM 2216
TAS	G9L 170524	П	8290, ASTM 2216
TAS	G9L 170538	П	8290, ASTM 2216
TAS	G9L 180646	Ш	8290, ASTM 2216
TAS	G9L 240493	1, 11	8290, ASTM 2216

Table 1-2 lists all samples collected along with the analytical suite, and associated SDG.

The following data quality indicators for sensitivity are used in this document and further described in Section 2.0.

- Method Detection Limit. The term is consistent with the EPA requirements found in 40 CFR 136, Appendix B.
- Method Reporting Limit (MRL). This is the term utilized by the laboratories and is equivalent to a Practical Quantitation Limit in accordance with NDEP Guidance (NDEP 2008)

## **1.2.Validation Process**

A formal validation of the Shallow Supplemental Soil Sampling in Areas I and II data was performed to determine the suitability of the data for potential use in the conceptual site model, risk assessment, and other future on-site environmental assessments.

Consistent with the memorandums identified above, the Tronox Quality Assurance Project Plan (QAPP; AECOM/Northgate 2009), and NDEP Supplemental Guidance (NDEP 2009d), all of the Shallow Supplemental Soil Sampling in Areas I and II data were validated.

Approximately 90% of the analytical data were validated as Stage 2B and approximately 10% were validated by Stage 4 data validation procedures. EPA Stage 2B (EPA 2009) validation evaluates the following QC criteria:

- Completeness of deliverable;
- Technical holding times and sample preservation;
- Sample integrity and cooler/sample temperature at the time of laboratory receipt;
- Laboratory and field blank contamination;
- Surrogate spike recoveries;
- Internal Standards for Organics only (where applicable);
- MS/MSD recoveries and relative percent differences (RPDs);
- Laboratory duplicate RPDs;
- Laboratory control sample (LCS) recoveries; and

• Initial and continuing calibrations.

The comprehensive validation, consistent with EPA designation of Stage 4 (EPA 2009), involves in-depth review of compound identification and quantification, spot-checks of calculations, and verification of summary data against the raw data. SDG R0907046 for VOA analysis, SDG R0907046 for OC Pesticide analysis, SDGs R0907146 and R07171 for SVOC analysis and SDG R0907146 for PCB analysis all underwent data validation to Stage 4. This represents 5 of the 37 SDG/analytical suite combinations. Note, the percent moisture data did not undergo data validation. Table 1-2 is a cross-reference of laboratory SDGs, samples, analytical suites and associated validation reports.

Analytical data deliverables from the laboratories were provided as an electronic data deliverable (EDD). The electronic data packages were presented in PDF format.

Validation of the Area I and II soil data was performed by Neptune and Company, using the appropriate EPA guidelines (EPA 1999, 2004, 2008, 2009) or the BMI Plant Site-Specific Supplemental Guidance on Data Validation from NDEP (NDEP 2009b, 2009c, 2009d, 2009e) and the Basic Remediation Company (BRC) SOP 40, Data Review/Validation (BRC 2009). These federal EPA guidelines, prepared for CLP data, were adapted to reflect the analytical methods and measurement quality objectives established for the Shallow Supplemental Soil Investigation methods and the guidance provided by NDEP. Neptune validation reports for Area I and II soils are presented in Appendices A-J.

The analytical reports for all Shallow Supplemental Soil Sampling in Areas I and II data are presented in Appendix K (as electronic files in Adobe pdf format).

Qualifier	Definition
J	Data are estimated; the direction of potential bias is uncertain.
J+	Data are estimated, with a potential for being biased high.
J-	Data are estimated, with a potential for being biased low.
U	Indicates the analyte was analyzed for but not detected at or above the stated censoring limit.
UJ	Indicates the analyte was analyzed for but not detected. The censoring limit is an estimated value due to uncertainty in the analysis.
JK	The analytical result is an estimated maximum possible concentration (EMPC)
X	The result is not used for reporting. This is generally applied where a more accurate and/or precise result is reported in place of this datum.
R	The result is rejected due to serious deficiencies in meeting the quality control criteria.
Combinations of qualifiers: (J- , J+, J) + (J-, J+, J) = J	When more than one qualifier is applied, the resulting final qualifier has no bias direction.

#### Table 1-3: Data Qualifiers

Analytical data were qualified using the data validation qualifiers in Table 1-3 and project-specific reason codes shown in Table 1-4. The finalized NDEP EDD (NDEP 2009f) for the Area I and II soil is presented in Appendix L (as an electron file in Microsoft Access format).

Analytical Suite	Analytical Method	Holding Time		
		Water	Soil	
Metals/Elements	SW-6010B SW-6020	180 days	180 days	
Mercury	SW-7470 SW-7471A	28 days	28 days	
Hexavalent Chromium	SW-7199	24 hours	30 days to extract, 4 days for analysis	
Perchlorate	314.0			
Dioxin/Furans	SW-8290	30 days to extract, 45 days for analysis	30 days to extract, 45 days for analysis	
Volatile Organic Compounds	SW-8260	14 days	TerraCore sampler with preservative: 14 days to extract, 40 days for analysis	
Semi-Volatile Organic Compounds	SW-8270	7 days to extract, 40 days for analysis	14 days to extract, 40 days for analysis	
Organochlorine Pesticides	SW-8081	7 days to extract, 40 days for analysis	14 days to extract, 40 days for analysis	
PCBs	SW-8082	7 days to extract, 40 days for analysis	14 days to extract, 40 days for analysis	
Total Petroleum Hydrocarbons	SW-8015B	GRO: 14 days DRO/ORO: 7 days to extract, 40 days for analysis	GRO: 14 days DRO/ORO: 14 days to extract, 40 days for analysis	

#### **Table 1-5: Holding Time Requirements**

## 2. Data Validation

The data validation qualifiers and reason codes were used to indicate all the data in the database where results were qualified as a result of validation. This following QC review elements are reviewed in this section:

- Holding times and sample preservation;
- Initial and continuing calibrations;

- Serial dilution;
- Laboratory blanks/equipment blanks/field blanks;
- LCS/Laboratory Control Sample Duplicate (LCSD) results;
- MS/MSD results;
- Surrogate recoveries;
- Internal standard performance;
- Laboratory duplicate results;
- Field duplicate results; and
- Quantitation problems.

Quantitation limits are critical to the proper evaluation of method sensitivity and non-detect data. Three types of quantitation limits were evaluated for stable chemistries as follows:

- Method detection limit (MDL) This limit was established by Paragon according to the requirement in 40 CFR 136, Appendix B, and represents the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. MDLs are established using matrices with little or no interfering species using reagent matrices and are considered the lowest possible reporting limit. Often, the MDL is represented as the instrument detection limit. Because these limits do not reflect sample-specific characteristics and preparation volumes/masses, MDLs were not reported in the hardcopy or EDDs for individual samples. However, MDLs can be indirectly obtained from the limits reported for method blanks, as method blanks were reported to the MDL.
- Sample quantitation limit (SQL) The SQL is defined as the MDL adjusted to reflect samplespecific actions, such as dilution or use of smaller aliquot sizes, and takes into account sample characteristics, sample preparation, and analytical adjustments. The SQL represents the sample-specific detection limit and all non-detected results are reported to this level.
- Practical quantitation limit (PQL) This limit is defined as the lowest level at which the entire analytical system gives a recognizable signal and acceptable calibration point for the analyte, and includes the predicted effect of sample matrices with typical interfering species. The laboratories reported data using the term MRL, this is equivalent to the PQL. The PQL is the lowest concentration of an analyte that can be reliably measured within specified limits of precision and accuracy during routine laboratory operating conditions. PQLs are used to estimate or evaluate the minimum concentration at which the laboratory can be expected to reliably measure a specific chemical contaminant during day-to-day analyses of different sample matrices. Detected results greater than the SQL, but less than the PQL were qualified by the laboratory as estimated.

In the EDD provided with this DVSR, the units for the SQL values are identical to the units associated with the results field (*result-units*). MDL, and PQL units are explicitly provided in the EDD - see the *mdl\_pql\_units* field.

Table 2-1 contains all results that were qualified where the reported value is greater than the SQL but less than the PQL.

Note that <u>no qualifiers</u> were applied to samples analyzed for mercury analysis (Method 7471A), perchlorate analysis (Method 314.0), hexavalent chromium analysis (Method 7199), total petroleum hydrocarbon analysis (Method 8015B), or VOC analysis (Method 8260B). <u>All criteria that were validated were found to be within method or validation limits.</u>

## 2.1.Sample Receipt and Holding Times

Technical holding times were met for all SDGs and all analytical suites. All samples were received in good condition within the temperature limit validation criteria.

No data were qualified based on sample receipt and holding time criteria.

## 2.2.Initial and Continuing Calibration

Instrument calibration data were included in the laboratory data packages, but not the EDDs (typical of the industry). Review included the instrument setup, operating conditions, initial calibration verifications, and continuing calibration verifications.

All calibrations met method or validation criteria in the mercury analysis (Method 7471A), perchlorate analysis (Method 314.0), hexavalent chromium analysis (Method 7199), total petroleum hydrocarbon analysis (Method 8015B), VOC analysis (Method 8260B), metals/elements (Methods 6010B and 6020), Organochlorine Pesticides (Method 8081), and PCBs (Method 8082).

Continuing calibration issues were identified in the Dioxin/Furans analysis (Method 8290) and SVOC (Method 8270C) analyses. These violations were associated with selected SDGs that had relative response factors or percent differences greater than the limits. In most cases the violations were not significantly outside the criteria. These data have been qualified as described in Appendix J and G respectively.

All data that were qualified due to calibration issues are provided in Table 2-7.

## 2.3. Quantification above Calibration Range

In most cases analyses that resulted in analytes being quantified at a value above the calibration range were diluted and re-analyzed. This diluted value, obtained from a response within the calibration range, is the best value and reported in the EDD. As such, some values are qualified with an "X" in the EDD to indicate a more appropriate value has been chosen and reported.

In the case of dioxin/furan analyses there a numerous instances where the analyte was detected above the calibration range, yet the instrument was not saturated. These are generally considered usable; as such the data have been qualified with an J and reason code "e."

All data that were qualified due to a result that exceeded the calibration range are provided in Table 2-8.

## **2.4.Serial Dilution**

Serial dilutions are used in the ICP-AES (Method 6010B) and ICP-MS (Method 6020) analysis of metals/elements to assess possible chemical or physical interferences. Data qualified due to serial dilutions that exceed the criteria of within ±10% of the original determination were qualified as described in Appendix B. A summary of all data qualified due to serial dilution issues is provided in Table 2-11

## 2.5.Laboratory, Equipment, and Field Blanks

Field and laboratory blanks consisting of contaminant-free water were prepared and analyzed as part of standard quality assurance/quality control (QA/QC) procedures to monitor for potential contamination of field equipment, laboratory process reagents, and sample containers. For this program, two groups of blanks were prepared and analyzed: (1) laboratory blanks (calibration blanks and method blanks), and (2) field QC blanks (including equipment and trip blanks). Each blank type is discussed below. The assignment of validation qualifiers associated with blank contamination is discussed.

#### Laboratory Blanks

Two types of laboratory blanks were prepared and analyzed: calibration blanks and method blanks. Both types were prepared in the laboratory using high-grade, contaminant-free water.

<u>Calibration Blanks</u> – Calibration blanks are comprised of acidified high-grade contaminant-free water analyzed at the beginning (initial calibration blank [ICB]), end (continuing calibration blank [CCB]), and every 10 runs during analysis of metals by ICP, ICP/MS, and CVAA. Their primary function is to initially set the calibration curve (along with calibration standards) and continually monitor the background for possible variations in instrument electronic signal or cross-contamination. ICB and CCB data are included in the laboratory data packages, but not the EDDs. As such, ICB and CCB data were evaluated based solely on hardcopy data (i.e., these results are not available in the database) for samples that underwent full validation only.

<u>Method Blanks</u> – Method blanks are laboratory QC samples that are prepared and analyzed with each batch of environmental samples. Method blanks are comprised of high-grade contaminant-free water that is carried though all preparation procedures in batches with field samples (including the addition of all reagents and QC monitoring compounds). Method blanks monitor potential contaminants in laboratory processes, reagents, and containers, and were analyzed for each analytical method used on field samples.

The discussion of analytes that were detected in one or more calibration or method blank is provided in the individual analytical suite results in the appendices. Note that sample results may or may not have been qualified for all listed analytes based on the comparison of blank concentrations to sample concentrations.

Table 2-2 contains all results that were qualified based on laboratory blank contamination. Details on why the data were qualified are provided in the appendices for each analytical suite.

#### Field QC Blanks

Trip blanks are field QC blanks collected and analyzed with field samples and were only collected for the VOC analysis. Trip blanks were prepared at the laboratory by filling a 40-milliliter vial with high-grade, contaminant-free water and sealing it with a Teflon-lined lid. Trip blanks are shipped to the field sampling location accompanying sample containers in the shipping cooler. When samples for VOCs are collected and shipped back to the laboratory for analysis, a trip blank is transported within the shipping container. Trip blanks monitor for potential contamination of sample containers during shipment to the field, as well as monitor for potential contamination of VOC samples during collection and transportation back to the laboratory.

No contaminants were found in the trip blanks analyzed for VOCs in this project. VOC trip blanks are identified in Appendix F.

Equipment blanks are field QC blanks collected in the field by field personnel. These were collected for all analytical suites with the exception of mercury, and hexavalent chromium. In general, no significant contamination was identified in the equipment blanks. Low levels of contaminants were identified in the metals/elements; Organochlorine pesticide; SVOC; and dioxins/furans. Details on the level of contamination and data qualification are described in the associated appendix.

Blank Value	Sample Result	Qualification
Detects	Not detected	No qualification
	< PQL	Report PQL with U
< PQL	≥ PQL	Use professional judgment
		(typically no qualification)
	< PQL	Report PQL with U
> PQL	≥ PQL but < blank value	Report PQL with U
> FQL	≥ PQL and > blank value	Use professional judgment
		(either J+ or no qualifier)
	< PQL	Report PQL with U
= PQL	≥ PQL	Use professional judgment
		(either J+ or no qualifier)
Negative value (often	< PQL	Report sample value with J-
seen with metals)		
Gross contamination	Detects	Qualify as rejected "R"

All organic chemistry results, with the exception of dioxins and furans, were qualified as follows:

For qualification of metals along with Dioxin and furan, the evaluation and validation of blank levels following the BMI (2009, rev 4) SOP-40 algorithms. In these cases the blank and sample levels were compared to the applicable SQL and PQL values.

Table 2-3 contains all results that were qualified based on field blank contamination. Details on why the data were qualified are provided in the appendices for each analytical suite.

## 2.6.Analyte Spike Samples

Spiked samples are environmental matrices spiked with a subset of target compounds at known concentrations. These QC samples were analyzed with project samples to measure laboratory accuracy and potential interference from the matrix. Two types of spike samples were analyzed with the project samples to monitor for potential interferences during analysis:

- Matrix spike (MS) and matrix spike duplicate (MSD) samples; these samples consist of aliquots of environmental samples spiked with a subset of target compounds. MS/MSD samples monitor potential interference from the site-specific sample matrix and its effect on target compounds.
- Blank spike samples, also known as laboratory control samples (LCS); these samples are an aliquot of reagent soil or water spiked with a subset of target compounds. The LCS monitors laboratory accuracy without the bias of a sample matrix. In some cases, the LCS was analyzed in duplicate (LCSD).

At least one MS/MSD sample and one LCS were prepared and analyzed with each batch of environmental samples. Note that for some SDGs, the laboratory conducted MS and MSD analyses on samples that were not specific to this project.

The reviewer evaluated both the spike and duplicate recoveries of the MS and LCS pairs. Data were generally qualified only if both the MS and LCS recovery were outside the QC limits. Though in some instances J+/J- qualifiers were applied to the sample that was spiked if the MS or MSD indicated recovery outside the limits. Data generally are not rejected based solely on MS or MSD recovery unless the exceedance is excessive.

Data qualified based on analyte spiking is provided in the individual appendices for each analytical suite. All results that were qualified due to MS/MSD recoveries are provided in Table 2-4. Table 2-5 contains all data that were qualified due to LCS recoveries. No data were rejected for this project based on spike recoveries.

## 2.7.Surrogate Spikes Samples

Surrogate spikes were prepared by adding compounds similar to target compounds of interest to sample aliquots and associated QC samples for organic analyses only. Surrogate spike recoveries monitor the efficiency of contaminant extraction from the sample medium into the instrument measuring system and measure possible interferences from the sample matrix that may affect the data quality of target compound results. Similarly, tracer isotopes are added to radionuclide analyses to monitor the extraction and analysis of radionuclides.

Surrogate spikes were added to the sample aliquot during preparation of the sample for analysis, and surrogate recoveries were compared with QC acceptance limits. Surrogate recoveries outside of the acceptable limits indicate interference from the sample matrix for the detection of target compounds.

Data qualified based on surrogate recovery is provided in the individual appendices for each analytical suite. One PCB SDG set showed recoveries above the upper range limit. This may be due to native concentrations of decachlorobiphenyl and tetrachloro-m-xylene. Data qualified based on surrogate recovery is provided in the individual appendices for each analytical suite. All data that were qualified due to surrogate issues are provided in Table 2-6.

## **2.8.Internal Standards**

Internal standards were prepared for certain organics and ICP/MS analyses by adding compounds similar to target compounds of interest to sample aliquots. Internal standards are used in the quantitation of target compounds in the sample or sample extract. Internal standard responses and retention times were presented in all data packages received from the laboratories in which these compounds were used. The evaluation of internal standards involved comparing the instrument response and retention time from the target compounds in the sample with the response and retention time of specific internal standards added to the sample extract prior to analysis.

For this project, internal standards were utilized in the VOC, SVOC, and dioxin/furan analyses. Data qualified based on internal standard recovery and retention times are provided in the individual appendices for each analytical suite. All data that were qualified due to internal standard issues are provided in Table 2-9.

## **2.9.Duplicate Field Samples**

Duplicate samples involved the preparation and analysis of an additional aliquot of a field sample. Results from duplicate sample analysis measure laboratory precision as well as homogeneity of contaminants in the field matrix. For this investigation, three types of duplicate analyses were conducted: (1) Matrix spike duplicates (MSDs), (2) matrix duplicates (MDs) and (3) field duplicates (FDs). MSDs and MDs measure laboratory precision and sample homogeneity, while field duplicates are used to evaluate field sampling technique precision, laboratory precision, and homogeneity of the sample matrix.

At least one duplicate analysis (MSD or MD) was performed with each batch of environmental samples processed in the laboratory though the duplicate samples did not always include samples from this project. The laboratory calculated the relative percent difference (RPD) between the two detected values for MSD and MD analyses of stable chemistries. RPD values within the acceptable limits indicate both laboratory precision and minimal matrix heterogeneity of compounds detected in the samples. Results associated with elevated RPD values were qualified as estimated to indicate the variability in detected concentrations or poor laboratory precision.

The following duplicate field samples were evaluated for recovery precision in the individual appendices.

#### Field Duplicate Samples

RSAI7009-1.5BR
RSAJ5009-1BR
RSAK5009-1BR
RSAL2009-1.5BR
SA114009-1BR
SA129009-1.5BR
SA155009-1BR
SA175009-1BR
SA182009-1BR
SA49009-1BR

All results qualified due to field duplicate precision not meeting the goal of less than 50% RPD are provided in Table 2-10.

In general, fairly good precision was found for most duplicate pairs. Data qualified based on duplicate precision is provided in the individual appendices for each analytical suite.

## 2.10. Other Qualifications

The laboratory evaluated the SQL and PQL for each sample result. In cases where sample results were greater than the SQL, but less than the PQL, the laboratory qualified the results as estimated. Specifically, results with this scenario were qualified "J" by the laboratory. During data validation, positive results less than the PQL but greater than or equal to the SQL were also qualified as estimated (J). Qualitatively, the results are acceptable; however, these results were considered estimated, because as the value approaches the SQL, the accuracy of the measurement is less certain. All results qualified as estimated (J) for this reason are presented in Table 2-1 and were assigned the validation comment code "sp" in the electronic database (EDD).

Some dioxin/furan results were qualified due to ion abundance ratios as described in Appendix J, Section 11.

In several cases an analyte was detected in two different analysis, an initial and dilution run. If the analyte had exceeded the calibration range it was qualified as described above in Section 2.3. For results that did not exceed the range, a decision on the best value was made during validation. This decision process was based on whether either value was qualified. In cases where there was no apparent data quality issue, the high value was chosen, though in all cases where the calibration range was not exceeded there was very little difference between values. The values that were discarded have a final validation qualifier of X, with a validation reason code of "o."

## 2.11. Summary of Rejected Data

Based upon the data validation summarized above and described in the appendices, <u>no sample results</u> <u>were rejected</u>. Data were qualified but no deficiencies were identified to result in rejection. All data are considered validated.

## 3. Evaluation of PARCCS Parameters

Overall data quality was acceptable based on the critical indicator parameters and no data were rejected. PARCCS parameters were reviewed for laboratory analytical results obtained during the investigation and the sections below discuss the results of the evaluation for each indicated parameter. These sections refer to the data quality indicators from an analytical standpoint only.

## **3.1.Precision**

Precision is the measure of the variability associated with an entire sampling and analysis process. It is the comparison among independent measurements as the result of repeated application of the same process under similar conditions. It is determined by analyzing field duplicate pairs, MSD pairs, and MD pairs. Precision is expressed as the RPD of a pair of values (or results) for stable chemistries.

## **3.2.Accuracy**

Accuracy is the degree to which a measurement agrees with its true value and is expressed as percent recovery. Accuracy is assessed by evaluating instrument calibrations and comparing MS, LCS, surrogate recoveries, and carrier/tracer yields with associated QC limits. Sample conditions and holding times can also affect accuracy and these parameters were assessed in the data validation. Each of these parameters is discussed in Section 2, with details provided in the individual appendices. Stage 4 validation entails more in-depth assessment of these same general elements as well as compound or peak identification, calculations, and chromatogram evaluation. This additional assessment under Stage 4 did not result in any rejection of data but did provide insight into the potential interferences observed in the PCB analysis.

For the data validation, positive or negative signs(+ or -) were assigned to the qualifiers to indicate the expected bias, as indicated by the QC result, when possible. In some cases, the MS and MSD had different biases for the same sample; in which case no bias could be assigned. In other instances several DQIs resulted in qualification resulting in a final qualifier assignment with no bias direction (J).

#### 3.3.Representativeness

Representativeness is a qualitative parameter and is defined by the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, or a process or environmental condition. Sample results were evaluated for representativeness by

examining items related to sample collection, including COC documentation, sample labeling, collection dates, and condition of the samples upon receipt at the laboratory. Laboratory procedures also were examined, including anomalies reported by the laboratory, either upon receipt of the samples at the laboratory or during analytical processes, adherence to recommended holding times of samples prior to analysis, calibration of laboratory instruments, adherence to analytical methods, and completeness of data package documentation.

A number of issues that were observed during sample receipt at the laboratory included clarifying sample identification. Each one appeared to be resolved and it was determined that no significant affect on data quality was observed.

In addition to the issues discussed above, representativeness is evaluated by reviewing blanks (laboratory method blanks, equipment blanks, and trip blanks. Generally, concentrations detected in the blanks were considerably less than reported results for the field samples; therefore, these concentrations did not affect overall data quality. Common laboratory contaminants were qualified as estimated in sample results when detected in associated blanks. Some data points were qualified based on field blank results and other results were qualified based on laboratory and field blanks. In some cases results were censored (reported as non-detects at an elevated reporting limit). The affected data were discussed within individual analytical suites in the appendices.

## 3.4.Comparability

Comparability of the data is a qualitative parameter that expresses the confidence with which one data set may be compared with another. Comparability of the data is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions (including dry weight basis), and using standardized reporting formats and data validation procedures.

## **3.5.Completeness**

Completeness is defined as the percentage of measurements judged to be valid. The validity of sample results is determined through the data validation process. All rejected sample results are considered to be incomplete. Data that are qualified as undetected (U), undetected at estimated reporting limits (UJ), and estimated (J) are considered to be valid and usable. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. Since no data were rejected, the completeness is 100%.

#### 3.6.Sensitivity

Sensitivity is the capability of a method to discriminate an actual deflection or response above instrument noise. For the EPA methods employed in this project, sensitivity is measured by the Method Detection Limit (MDL) and Practical Quantitation Limit (PQL). Both nominal MDLs and PQLs (identified as an MRL) were provided by the laboratories in the laboratory data packages and were verified during validation. MDLs in general were adjusted for each soil and EB sample to include the necessary dilution factors, preparation factors, and dry-weight factors of an individual sample as the SQL. The sensitivity requirements were based on the laboratory's ability to detect and report consistent and reliable limits.

Scenarios involving dilutions, high moisture content, and matrix interference affect the SQL by raising it according to the dilution factor or percent moisture content. Dilutions were required for several analyses because of high concentrations. Whenever the concentration exceeded the linear range of the instrumentation, dilutions were analyzed with the exception of the dioxin/furan analyses. In the case of Method 8290 (dioxins/furans), if the instrument was not saturated and all other QC parameters were acceptable, dilutions are generally not required.

It is expected that when a direct comparison to approved applicable screening levels is conducted, some SQLs will exceed the corresponding levels. Usability for other scenarios will be determined on a case-by-case basis. Procedures for handling non-detects (whether above the screening level or not) will be addressed in future work plans.

## 4. Conclusions and Recommendations

Based on the evaluation of each data set, 100 percent of the data obtained for this project are valid (that is, not rejected) and are acceptable for their intended use. All data were validated to EPA Stage 2B level, approximately 10% was validated to EPA Stage 4 level. Data that was qualified during this validation process are summarized in Tables 2-1 through 2-10. Data qualifiers are shown in Table 1-3 with the qualifier reason codes that were added to the electronic data base (EDD) are provided in Table 1-4.

Limitations on data usability for future purposes may arise, but are not addressed in the scope of this document. These limitations will be identified through subsequent data evaluations and mitigated where possible by collecting additional data in future investigations.

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