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7. Abstract

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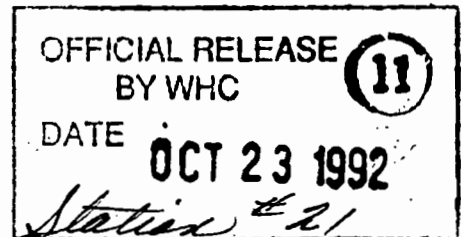
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## 1.0 INTRODUCTION

Data from the chemical analysis of eight samples from the 300-FF-1 Operable Unit Remedial Investigation and their related quality assurance samples were reviewed and validated to verify that reported sample results were of sufficient quality to support decisions regarding remedial actions performed at this site. Four samples collected from the 307-RB-1 borehole were analyzed by Thermo Analytical Laboratories (TMA) using U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) protocols. Four split samples from the 399-1-8A borehole were analyzed by Roy F. Weston, Incorporated using CLP protocols. Sample analyses included:

- ▶ Volatile organics;
- ▶ Pesticides and polychlorinated biphenyls (PCBs);
- ▶ Metals; and
- ▶ General chemical parameters.

Data quality was reviewed and validated using EPA CLP protocols and guidelines and related WHC Hanford Company procedures. Data were qualified based upon their quality and the guidance provided by these sources. Instances where WHC Hanford Company procedures are more restrictive than CLP protocols were noted, particularly with regard to holding times for pesticide/PCB analyses. Data have been initially validated to CLP protocol for holding times in these instances, and the potential difference in results if Westinghouse Hanford Company (WHC) procedures are used have been highlighted.

No volatile organic data or pesticide/PCB data were rejected. Arsenic and selenium data from one sample delivery group were rejected due lack of documentation. Silver data from another sample delivery group were rejected due to very low laboratory control sample (LCS) percent recovery.

Several general quality trends which resulted in data qualification were observed. These included:

- ▶ Initial or continuing calibrations fell outside of established criteria for some volatile organic compounds.
- ▶ Blank contamination was noted in both of the sample delivery groups analyzed for volatile organic compounds.
- ▶ Case-specific matrix spike analyses were not performed for the sample analyzed by Roy F. Weston for pesticide compounds.

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- ▶ Roy F. Weston did not report results for the full target compound list (TCL) for pesticides and PCBs. In addition, some of the quality control requirements were not met. The as-qualified data reporting form lists all compounds, and professional judgement was used to qualify the data (all non-detects) based upon expanded criteria.
- ▶ Excessive holding times between sample collection and analysis, apparent excessive time periods between initial and continuing calibrations, and lack of appropriate quality control analyses for ion chromatography, ammonia, and pH were observed for analyses performed by TMA.
- ▶ Sample preparation methods for general chemical analyses were not described in the data packages; differing preparation methods may also introduce biases in reported results.
- ▶ In addition, several of the general chemistry analyses were not reported on the reporting forms, although requested and analyzed for.

The laboratory reported results for samples B00VRD and B00V50. WHC later indicated to Hart Crowser, Inc. that the sample names should be B00VR0 and B00VS0. The validation forms may still contain a reference the former. This report, however, addresses the latter.

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## 2.0 VOLATILE ORGANIC DATA VALIDATION AND LIMITATIONS

### SUMMARY

#### *Two Sample Delivery Groups*

Sample results from two volatile organic cases (10-160 and 9105L510) are included in this report. Data qualifiers for these cases are summarized in Table 2-1.

#### *Twenty Percent Validation*

A minimum of twenty percent of the sample analyses for Thermo Analytical Case 10-160 and Roy F. Weston Case 9105L510 were fully validated, including recalculation of all results reported by the laboratories. All reported results for quality assurance samples associated with these cases were fully validated.

#### *WHC Validation Guidance Used*

Data validation was performed in accordance with the WHC Draft Data Validation Procedures for Chemical Analyses (WHC 1991a). Additional criteria established for the determination of laboratory performance was obtained from WHC (WHC 1991b), and the EPA's Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses (EPA 1998b).

#### *Samples Analyzed According to CLP Protocols*

Sample analyses were performed using gas chromatograph/mass spectrometer (GC/MS) systems, outfitted with packed columns. Instances where failure to comply with various technical requirements established by CLP protocols resulted in qualification of the data. The specific problems observed during the quality assurance review are detailed in the sections below.

#### *Data Quality Objectives Met*

The analysis was complete and met the method and work plan CRQL requirements (WHC 1991b).

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***No Rejected Data***

No data associated with these two cases were rejected due to quality control results.

***Minor Deficiencies Noted***

There were minor deficiencies associated with the analyses. These include: minor blank contamination; a level of common laboratory contaminants that were not found in the blanks; and compounds detected above the instrument detection limit, but below the quantification limit. These qualifications have been explained in greater detail in the section of this report.

**ANALYTICAL METHOD*****Gas Chromatography/Mass Spectrometer Tuning Criteria Met***

Tuning is performed to ensure that mass resolution, identification, and to some degree sensitivity of the gas chromatography/mass spectrometry (GC/MS) instrument have been established. When analyzing for volatile organics, instrument tuning is performed with bromofluorobenzene (BFB). Instrument tuning must be performed prior to the analysis of either standards or samples and must meet the criteria established by the analytical protocol. The specific criteria for acceptable GC/MS instrument tuning using BFB are outlined in WHC (WHC 1991a) and in EPA (EPA 1988a,b) criteria.

The original data were checked for transcription and calculation errors to verify that tuning criteria were met. Prior to calibration and sample analysis, all tuning criteria were met.

***Acceptable Calibration***

Instrument calibration is performed to establish that the GC/MS instrument is capable of producing acceptable and reliable analytical data over a range of concentrations. The initial and continuing calibrations are to be performed according to CLP protocols. An initial multipoint calibration is performed prior to sample analysis to establish the linear range of the GC/MS instrument. Continuing calibration checks are performed to verify that instrument performance is stable and reproducible on a day-to-day basis.

A detailed description of the results for the initial and continuing calibrations performed is presented below.

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**Acceptable Initial Calibration**

Instrument response is established when the relative response factors (RRFs) for all target compounds are greater than or equal to 0.05 units. Linearity is established when the relative standard deviations (RSDs) of the RRFs are less than or equal to 30 percent.

All initial calibration criteria were met for data associated with these two cases, with the exception of methylene chloride and vinyl acetate in Case 9105L510. There were no detectable levels of these compounds (after evaluation of blank contamination) found in sample B00VQ8. Therefore, their detection limits were qualified as estimates (UJ).

**Minor Continuing Calibration Exceedences**

The criteria for accepting the continuing calibration requires that a 50 ug/L standard be analyzed at least once per 12 hour period and that the RRFs of all target compounds be greater than or equal to 0.05 units. In addition, the percent difference of these RRFs must be less than or equal to 25 percent of the average RRFs calculated for the associated initial calibration.

All continuing calibration criteria were met for Case 10-160 with the exception of chloromethane, whose percent difference was 34%. The out-of-tolerance continuing calibration was associated with the matrix spike analysis only. Therefore, no qualification of sample data was made.

All continuing calibration criteria were met for Case 9105L510 with the exception of 1,2-dichloroethane, vinyl acetate, and 4-methyl-2-pentanone. There were no detectable levels of these compounds found in the associated sample (B00VQ8). Therefore, their detection limits were qualified as estimates (UJ).

**Minor Method Blank Contamination**

Method blank and field blank analyses are performed to determine the extent of laboratory or field contamination of samples.

In Case 10-160, 2-hexanone and methylene chloride were detected in the associated method blank. 2-Hexanone was not detected in any associated samples. Methylene chloride and two common laboratory contaminants (acetone and carbon disulfide) were detected at low concentrations in the associated samples. These appear to be attributable to laboratory contamination, and the associated concentrations were qualified as undetected (U).

In Case 9105L510, acetone and methylene chloride were detected in the associated method blank. These compounds were detected at low concentrations in sample B00VQ8. Therefore, the associated concentrations were qualified as undetected (U).

### **HOLDING TIMES WERE MET**

Analytical holding times were assessed to ascertain whether the CLP holding time requirements for volatile organic analyses were met by the laboratory. The CLP holding time requirements for volatile organic analyses are as follows: soil samples must be analyzed within 14 days of the date of sample collection, and all samples must be shipped on ice to the laboratory and stored at 4°C until analysis.

All holding times were met for samples associated with these two cases.

### **ACCURACY**

Accuracy was assessed by evaluating the recoveries of stable isotopically-labeled surrogate compounds added to all samples and blanks, and by the analysis of a representative sample which was spiked with a variety of volatile organic compounds.

#### ***Surrogate Compound Recoveries Acceptable***

Matrix-specific surrogate compound recovery control windows have been established by the EPA CLP program. When a surrogate compound is out of the control window, all positively identified target compounds associated with the unacceptable surrogate recoveries are qualified as estimates (J). Undetected compounds are qualified as having an estimated detection limit (UJ).

The surrogate compound recoveries calculated for the three stable isotopically labeled surrogate compounds were acceptable in all cases.

#### ***Matrix Spike Recoveries Acceptable***

Matrix spike compounds are added to a sample which is representative of the sample delivery group. Matrix spike analyses are performed in duplicate using five compounds specified by CLP protocols. All recoveries for the five compounds should be within the established quality control limits (EPA 1988b). The matrix spike analyses estimate how much the target compounds are interfered with, either positively or negatively, by the sample matrix.

The matrix spike compound recoveries were all found to be acceptable in all cases.

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## PRECISION ACCEPTABLE

Precision is expressed by the relative percent difference (RPD) between the recoveries of duplicate matrix spike analyses performed on a sample. When the laboratory has not performed duplicate spike analyses, precision may also be assessed using unspiked duplicate sample analyses. Field precision is measured by analyzing duplicate samples taken in the field.

There was one set of matrix spike samples analyzed with Case 10-160, but no project-specific matrix spike samples analyzed with Case 9105L510. For Case 10-160 the precision was acceptable, and ranged from 1 to 19 %RPD. No field duplicate samples were analyzed. No data for Case 9105L510 were qualified based upon the lack of project-specific matrix spike analysis.

## INTERNAL STANDARD PERFORMANCE ACCEPTABLE

Internal standard performance was assessed to determine whether abrupt changes in instrument response and sensitivity occurred that may have affected the reliability of the analytical data. The response (area or height) of the internal standards must not vary by more than +100 percent or -50 percent from the response of the internal standard that was used to calculate the upper and lower bounds. The upper and lower bounds define the range for acceptable internal standard response (area/height) for the sample analyses. The criteria for internal standard performance were met in all cases.

## IDENTIFICATION OF COMPOUNDS

The identity of detected compounds was confirmed to investigate the possibility of false positives. The confirmation of compound identification during the quality assurance review focuses on false positives because only mass spectra for positive identifications are submitted. However, target compounds that are reported as undetected are also evaluated to investigate the possibility of false negatives. Confirmation of possible false negatives is addressed by reviewing other factors relating to analytical sensitivity (e.g., detection limits, linearity, analytical recovery).

All compound identifications were confirmed. Compounds found in samples associated with these two cases include chloroform, benzene, toluene, and trichloroethene.

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**COMPOUND QUANTIFICATION AND REPORTED DETECTION LIMITS**

Compound quantifications and reported detection limits were recalculated and verified for a minimum of 20% of the samples in each case to ensure that they were accurate and are consistent with CLP requirements.

Below the contract required quantification limit (CRQL), instrument precision becomes more variable as the instrument detection limit (IDL) is approached. Therefore, the concentration of any compound that was detected below the CRQL was qualified as an estimate (J).

**SYSTEM PERFORMANCE ACCEPTABLE**

A thorough review of ongoing data acquisition and instrument performance criteria was made to assess overall GC/MS instrument performance. No changes in instrument performance were noted that would result in the degradation of data quality. No indications of unacceptable instrument performance (i.e. shifts in baseline stability, retention time shifts, extraneous peaks, sensitivity) were found during the quality assurance review.

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**TABLE 2-1**  
**300-FF-1 Operable Unit Remedial Investigation**  
**Volatile Organic Analysis and Qualifier Summary**  
**Thermo Analytical Case 10-160**  
**Roy F. Weston Case 9105L510**

Compounds	Thermo Analytical Case A1-10-160 Samples								Roy F Weston Case 9105L510 Samples		
	CRQL	B014R2		B014R3		B014R4		B014R5		B00VQ8	
	(ug/Kg)	(ug/Kg)	Q	(ug/Kg)	Q	(ug/Kg)	Q	(ug/Kg)	Q	(ug/Kg)	Q
<b>300-FF-1 Compounds of Concern</b>											
Methylene Chloride	10	5	U	5	U	5	U	5	U	24	U
1,1-Dichloroethene	10	5	U	5	U	5	U	5	U	5	U
1,2-Dichloroethene (total)	10	5	U	5	U	5	U	5	U	5	U
Trichloroethene	10	5	U	5	U	5	U	5	J	5	U
Tetrachloroethene	10	5	U	5	U	5	U	5	U	5	U
<b>Other Reported Compounds</b>											
Acetone	10	22	U	40	U	30	U	42	U	32	U
Carbon Disulfide	10	5	U	6	U	6	U	5	U	NR	
Chloroform	10	2	J	NR		NR		5	U	NR	
Benzene	10	NR		NR		2	J	3	J	NR	
Toluene	10	NR		NR		2	J	4	J	4	J

All 300-FF-1 Operable Unit compounds of concern and those compounds originally reported by the laboratory as present at concentrations above the detection limit are listed

CRQL = Contract Required Quantitation Limit

Q = Data qualifier assigned or verified during data validation

NR = Not reported as present at concentrations above detection limits for this sample

### 3.0 PESTICIDE AND PCB DATA VALIDATION AND LIMITATIONS

#### SUMMARY

##### *Two Sample Delivery Groups*

Two pesticide and PCB cases (10-160 and 9105L510) are included in this report. Data qualifiers for these cases are summarized in Table 3-1.

##### *Twenty Percent Validation*

A minimum of twenty percent of the sample analyses for Thermo Analytical Case 10-160 and Roy F. Weston Case 9105L510 were fully validated, including recalculation of all results reported by the laboratories. All reported results for quality assurance samples associated with these cases were fully validated.

##### *WHC Validation Guidance Used*

Data validation was performed in accordance with the WHC Draft Data Validation Procedures for Chemical Analyses (WHC 1991a). Additional criteria established for the determination of laboratory performance was obtained from WHC (WHC 1991b), and the EPA's Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses (EPA 1998b).

##### *Samples Analyzed According to CLP Protocols*

Sample analyses were performed using gas chromatography/electron capture detection (GC/ECD) systems. Instances where failure to comply with various technical requirements established by CLP protocols resulted in qualification of the data. The specific problems observed during the quality assurance review are detailed in the sections below.

##### *Data Quality Objectives Met*

The analyses were complete and overall met the method and work plan requirements (WHC 1991b). In neither case were work plan CRQL requirements met. Results for

the full list of target analytes were not reported for Case 9105L510; only PCB data were reported.

### ***No Data Rejected***

No data associated with these two cases were rejected.

### ***Deficiencies Associated with Case 9105L510***

There were no significant deficiencies associated with Case 10-160. However, there were deficiencies associated with Case 9105L510. These deficiencies included incomplete reporting of the data and an unacceptable initial calibration. These qualifications have been explained in greater detail in the body of this report.

## **ANALYTICAL METHOD**

### ***Instrument Performance***

Instrument performance was assessed to ensure that adequate chromatographic resolution and instrument sensitivity were achieved by the gas chromatographic system.

The specific criteria for acceptable instrument performance are outlined in EPA guidelines (EPA 1988a,b), including the evaluation and qualification procedures that may be performed on the analytical results.

During the quality assurance review, all indicators for acceptable instrument performance were verified. All criteria established by CLP protocols were met and the results are acceptable, except as noted.

### ***Unacceptable Instrument Calibration - Case 9105L510***

Instrument calibration is performed to ensure that the chromatographic system is capable of producing acceptable and reliable analytical data. The initial and continuing calibrations are to be performed according to procedures established by CLP protocols. An initial calibration is performed prior to sample analysis to establish the linear range of the system, including a demonstration that all target compounds can be detected. Continuing calibration checks are performed to verify that instrument performance is stable and reproducible on a day-to-day basis.

9 3 1 2 9 0 0 0 4 6 1

A detailed description of the results for the initial and continuing calibrations is presented below.

### **Initial Calibration**

The laboratory performed an initial multipoint calibration for the four compounds specified at the concentrations required by CLP protocols. The linearity of the initial calibration is established when the percent relative standard deviation (RSD) of the calibration factors is less than or equal to 10 percent.

For Case 10-160 all initial calibration criteria were met. For Case 9105L510 the linearity exceeded the 10 percent criteria. Values ranged from 21 to 25 percent. According to WHC validation procedures, the data should be rejected. It is reasonable to accept data where the initial calibration RSD ranges from 10 and 35 percent and qualify results as estimates, or as having estimated detection limits. As the sample associated with Case 9105L510 had no detectable levels of the compounds of concern, the detection limits were qualified as estimates (UJ).

### **Continuing Calibration Acceptable**

The criteria for acceptable continuing calibrations requires that the calibration factors for all target compounds have a percent difference of less than or equal to 15 percent of the average calibration factor calculated for the associated initial calibration standard. The 15 percent difference value is required for results calculated using the chromatographic column which is used for quantitative purposes. In addition, the percent difference of the calibration factors calculated for the chromatographic column that is used for confirmation must be less than or equal to 20 percent.

The results for continuing calibration are acceptable for Case 10-160. For Case 9105L510 numerous compounds exceeded the criteria for the last pair of standards (INDA and INDB). As these were analyzed after the sample analysis, and because the results were already qualified as having estimated detection limits, further qualification of the data was not necessary.

### **Analytical Sequence Acceptable**

Sample analysis and instrument calibration must be performed in a 72-hour sequence established by CLP protocols. In both cases, the laboratory performed the standard and sample analyses according to this sequence.



### ***Method Blank Analysis***

Method blank samples are analyzed to determine the extent of laboratory contamination of samples. There were no compounds of concern detected in the method blanks.

### **HOLDING TIMES MET**

Analytical holding times were assessed to ascertain whether the CLP holding time requirements for pesticide/PCB analyses were met by the laboratory. The CLP holding time requirements for pesticide/PCB analyses are as follows: soil samples must be extracted within 14 days of the date of sample collection; all extracts must be analyzed within 40 days of extraction; all samples must be shipped on ice to the laboratory and stored at 4°C until extraction; and the extracts must be stored at 4°C until analysis.

The CLP criteria were met for both cases, and no data have been qualified based upon holding times.

WHC data validation procedures specify holding times for pesticide and PCB analyses which are more restrictive than those established by CLP protocols. WHC procedures require that samples be extracted within 7 days of collection and analyzed within 40 days of extraction (WHC 1991a).

Using WHC guidelines, data from samples B014R2 and B014R3 in Case 10-160 would be qualified based upon holding times, as these samples were extracted 10 days after collection.

### **ACCURACY**

Accuracy was assessed by evaluating the recoveries of the surrogate compounds and the matrix spike recoveries calculated for the sample analyses. Both sets of results are presented below.

### ***Surrogate Compound Recoveries Acceptable***

The CLP specified surrogate, dibutyl chlorendate (DBC), was used for all sample analyses. The recovery for DBC must be within the established CLP acceptable quality control limits of 24 to 150 percent (EPA 1988a). All surrogate recoveries for DBC were recalculated and verified during the quality assurance review, using the average of the corresponding calibration factors from Evaluation Mixes A, B, and C.

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The recoveries for DBC were acceptable for all samples associated with these two cases.

### ***Matrix Spike Recoveries Acceptable***

Matrix spike analyses are performed in duplicate using six compounds specified by CLP protocols. The recoveries for the six compounds must be within the acceptable quality control limits established by CLP protocols.

Case 10-160 matrix spike recoveries were acceptable. For Case 9105L510 there was no project-specific matrix spike sample analysis.

### **PRECISION ACCEPTABLE**

Precision is expressed as the relative percent difference (RPD) between the recoveries of the matrix spike and the matrix spike duplicate analyses performed on a sample. The RPDs were all acceptable for the MS/MSD analyses associated with Case 10-160. Project-specific matrix spike analyses were not performed for Case 9105L510.

### **IDENTIFICATION OF COMPOUNDS**

There were no target compounds detected in any of the samples associated with these two cases. The data were evaluated to investigate the possibility of false negatives. Confirmation of possible false negatives is addressed by reviewing other factors relating to analytical sensitivity (e.g., detection limits, instrument linearity, analytical recovery). The data do not appear to be false negatives.

### **COMPOUND QUANTIFICATION AND REPORTED DETECTION LIMITS**

Compound quantifications and reported detection limits were recalculated and verified for a minimum of 20% of the samples in each case to ensure that they were accurate and are consistent with CLP requirements (EPA 1988a). The reported detection limits must be in accordance with the CRQLs specified in the applicable CLP statement of work.

The CRQLs reported were calculated correctly and were acceptable.

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**TABLE 3-1**  
**300-FF-1 Operable Unit Remedial Investigation**  
**Pesticide/PCB Analysis and Qualifier Summary**  
**Thermo Analytical Case 10-160**  
**Roy F. Weston Case 9105L510**

Compounds	CRQL (ug/Kg)	Thermo Analytical Case 10-160 Samples								Roy F Weston Case 9105L510 Samples	
		B014R2		B014R3		B014R4		B014R5		B00VQ8	
		(ug/Kg)	Q	(ug/Kg)	Q	(ug/Kg)	Q	(ug/Kg)	Q	(ug/Kg)	Q
300-FF-1 Compounds of Concern											
Arochlor-1248	33.0	82.0 U	83 U	84 U	84 U	81 U					

All 300-FF-1 Operable Unit compounds of concern and those compounds originally reported by the laboratory as present at concentrations above the detection limit are listed

CRQL = Contract Required Quantitation Limit

Q = Data qualifier assigned or verified during data validation

NR = Not reported as present at concentrations above the detection limits for this sample

9 3 1 2 9 0 0 6 2 1 2 6

## 4.0 METALS DATA VALIDATION AND LIMITATIONS

### SUMMARY

#### *Two Sample Delivery Groups*

Two metals sample delivery groups are included in this report. Data qualifiers for these cases are summarized in Table 4-1.

#### *Twenty Percent Validation*

A minimum of twenty percent of the samples from each of the four cases were fully validated, including recalculation of all results reported by the laboratories. All reported results for quality assurance samples associated with these cases were fully validated.

#### *WHC Validation Guidance Used*

Data validation was performed in accordance with the WHC Draft Data Validation Procedures for Chemical Analyses (WHC 1991a). Additional criteria established for the determination of laboratory performance was obtained from WHC (WHC 1991b) guidelines, and the EPA's Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses (EPA 1988d).

#### *Samples Analyzed According to CLP Protocols*

Eight low level soil samples were submitted for analysis for inorganic target analyte list (TAL) metals. A total of fourteen analyses were performed (e.g., submitted samples, reanalyses, method blanks, matrix spike samples, duplicate samples). Thermo Analytical performed the analyses according to the 1988 CLP protocol (EPA 1988c). Roy F. Weston performed the analyses according to the 1990 CLP protocol (EPA 1990).

Samples were analyzed using an inductively coupled argon plasma emission spectrometer (ICP), a graphite furnace atomic absorption spectrometer (GFAA), and a cold vapor atomic absorption spectrophotometer (CVAA). Instances where failure to comply with various technical requirements established by CLP protocols resulted in qualification of

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the data. The specific problems observed during the quality assurance review are detailed in the sections below.

### ***Most Data Quality Objectives Met***

The analyses were complete and met the method and work plan contract required detection limit (CRDL) requirements (WHC 1991b), with the exception of the arsenic, selenium and silver data which were rejected.

### ***Major Deficiencies for Arsenic, Selenium, and Silver***

Arsenic and selenium data for Case A1-10-158 were rejected; the affected samples include B014R2, B014R3, B014R4 and B014R5. Reported sample results were rejected because supporting raw data were missing from the data package.

Silver data for Case 9105L510, 9105L529, and 9105L597 were rejected; the affected samples included B00VQ8, B00VQ9, B00VR0, and B00VS0. The data were rejected due to an unacceptable laboratory control sample (LCS) percent recovery of 1.5%.

### ***Minor Deficiencies (Other Qualified Data)***

There were minor deficiencies associated with the analysis of these samples.

Minor deficiencies for Case A1-10-158 included low percent recovery of antimony and silver in the matrix spike sample; minor blank contamination; elements reported near the instrument detection limit (IDL); and minor calculation errors.

Minor deficiencies for Case 9105L510, 9105L529, and 9105L597 included unacceptable duplicate analysis precision; minor blank contamination; elements reported near the IDL; low matrix spike percent recoveries; unacceptable ICP serial dilution precision; low analytical spike recovery; missed holding times; transcription errors; and the absence of some quality control samples.

These qualifications are explained in greater detail in the following sections.

## **ANALYTICAL METHODS**

Performance of specific instrument quality assurance and quality control procedures, including deficiencies noted during the quality assurance review, are outlined below.

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***Instrument Calibration Acceptable***

Three calibration standards and a blank were analyzed for arsenic, selenium, thallium, and lead by GFAA. The correlation coefficient of a least squares linear regression met the requirements for calibration.

Up to five calibration standards and a blank were analyzed for mercury by CVAA. The correlation coefficient of a least squares linear regression met the requirements for calibration.

At least one standard and a blank were analyzed by ICP for all other elements.

***Calibration Verification Acceptable***

The above calibrations were each immediately verified with an initial calibration verification (ICV) standard and a calibration blank. The ICV was prepared from a source independent of the calibration standards, and was prepared at a mid-calibration range concentration. The ICV percent recovery must fall within the control limits of 90-110 percent for metals analyzed by ICP and GFAA, and 80-120 percent for mercury. Calibration linearity near the detection limit was verified with a standard prepared at a concentration near the CRDL. The ICVs met the recommended control limits in all cases.

The calibrations were subsequently verified at regular intervals using a continuing calibration verification (CCV) standard. The control windows for percent recovery of CCV standards are the same as the ICV windows described above. The CCVs met the recommended control limits in all cases.

***Low Level Blank Contamination***

Initial calibration blank (ICB), continuing calibration blank (CCB) and preparation or method blank (PB or MB) results were reviewed to determine the extent of variability of the detection limit and the existence and magnitude of blank contamination.

Several elements were detected in blanks associated with Case A1-10-158. Listed below is the maximum concentration of elements found in all associated laboratory blanks. No field blanks were submitted.

Element Detected	Concentration in ug/L		
	CRDL	IDL	Highest Blank
Manganese	15	2	8.1
Aluminum	200	31	47.8
Calcium	5000	15	99.3
Iron	100	12	56.0
Sodium	5000	57	140.6
Barium	200	2	3.9
Copper	25	5	11.2

Two elements were detected in blanks associated with Cases 9105L510, 9105L529, and 9105L597. Listed below is the maximum concentration of elements found in all associated laboratory blanks. No field blanks were submitted.

Element Detected	Concentrations in ug/L		
	CRDL	IDL	Highest Blank
Sodium	5000	71	117.4
Zinc	50	3	3.9

Samples with digestate concentrations (in ug/L) of less than five times (<5x) the highest amount found in any of the associated blanks have had their associated values qualified as non-detected (U). Samples with concentrations of greater than five times (>5x) the highest amount found in any of the associated blanks do not require qualification.

Therefore, sodium data for sample B014R2 were qualified as undetected (U). No other data were qualified based on the low level concentrations detected in the blanks.

**MERCURY RECOMMENDED HOLDING TIME EXCEEDED**

Analytical holding times for ICP metals, GFAA metals, and CVAA mercury analyses were assessed to ascertain whether the holding time requirements were met by the

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laboratory. The holding time requirements are as follows: samples must be analyzed within twenty-eight days for mercury, and within six months for all other metals.

All required holding times were met, with the exception of mercury (samples B00VR0, B00VS0, B00VQ8 and B00VQ9) which were prepared 2 to 8 days after the recommended holding time had expired. As mercury was not detected in any of these samples their detection limits were qualified as estimates (UJ).

### *Instrument-Specific Quality Control Procedures*

#### ICP

Interference check samples (ICS) were analyzed at the beginning and end of each ICP sample run to verify the laboratory interelement and background correction factors. Results for the ICS solution must fall within the control limit of  $\pm 20\%$  of the true value.

The ICS samples analyzed with each of these cases were acceptable.

A five-fold serial dilution is required for all elements analyzed by ICP whose concentrations are greater than 50 times the IDL. The subsequent concentrations of the reanalysis are compared with the original analysis. The concentration values must agree within a percent difference (%D) of 10%.

A serial dilution was required for many of the ICP metals. In Case A1-10-158 all dilution concentrations were found to be within ten percent difference of the initial analysis. Therefore, no qualifications were made based upon the serial dilution results.

In Cases 9105L510, 9105L529, and 9105L597 all dilution concentrations were found to be within ten percent difference of the initial analysis with the exception of zinc. Therefore, zinc results for all four of the associated samples were qualified as estimates (J).

#### GFAA

Duplicate injections are required for all GFAA analyses. The duplicate injections establish the precision of the individual analytical determinations. For sample concentrations greater than the CRDL, duplicate injections must agree within  $\pm 20\%$  RSD.

All duplicate injection quality control requirements were met.

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The post-digestion analytical spike is analyzed to determine the extent of interference in the digestate matrix. When the results of the analytical spike analyses exceeds the control window of 85 to 115 percent recovery and the absorbance of the sample is less than fifty percent of the analytical spike absorbance then the sample must be reanalyzed using the method of standard additions (MSA).

All analytical spike control requirements were met, with the exception of selenium in sample B00VS0. Analytical spike recovery was 82% and there was no selenium found in the sample. Therefore, an MSA analysis was not required or performed. The selenium detection limit for sample B00VS0 was qualified as an estimate (UJ).

## ACCURACY

### *Matrix Spike Exceedences for Antimony, Lead, Manganese, and Silver*

Matrix spike analyses are used to assess the analytical accuracy of the reported data and the effect of the matrix on the ability to accurately quantify sample concentrations. Matrix spike recoveries must generally fall within the range of 75-125%.

Matrix spike recoveries were out of control for antimony and silver in sample B014R2 (49% and 69%, respectively). Antimony and silver were not detected in the associated samples (B014R2, B014R3, B014R4, and B014R5). Therefore, their detection limits were qualified as estimates (UJ).

Matrix spike recoveries were out of control for antimony, lead, and manganese in sample B00VQ8 (57%, 59%, and 67%, respectively). Sample results for these elements in the associated samples (B00VQ8, B00VQ9, B00VS0 and B00VR0) were qualified as estimates (J).

### *Laboratory Control Sample Results Acceptable*

The laboratory control sample (LCS) monitors the overall performance of the analysis, including the sample preparation. An LCS should be digested or distilled and analyzed with every group of samples which have been prepared together. The performance criteria for solid LCS samples are established through interlaboratory studies coordinated by a certifying agency (e.g. EPA or an independent commercial supplier).

One solid LCS was digested and analyzed with Case A1-10-158. The results were compared against the control windows established by the EPA and were found to be acceptable.

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Two solid LCS samples were digested and analyzed with Case 9105L510, 9105L529, and 9105L597. The results were compared against the control windows established by the EPA and were found to be acceptable.

### *The Case for Silver*

The percent recovery for silver in one of the LCS samples was 1.5%. Silver recovery is quite variable by this method, and is largely dependent on analyst technique. Because of this the EPA has not been able to establish a control window, and is considering another method for future use. The very low percent recovery indicates that there was potentially a considerable loss of silver from the sample during the digestion procedure. Therefore, we recommend that the silver results be rejected R. There was no silver detected in any of the associated samples.

## PRECISION

### *Duplicate Analyses*

Analytical duplicate sample analyses are used to measure laboratory precision and sample homogeneity. Field duplicate analyses are used to measure both the laboratory and the field sampling procedure precision.

One set of duplicate analyses were analyzed for all analytes in Case A1-10-158. Relative percent differences (RPD) were greater than 20%, but less than 35% for lead. As the EPA Functional Guidelines recommends the 35% control window (EPA 1988d), the results were not qualified.

One set of analytical duplicates were analyzed for all analytes in Cases 9105L510, 9105L529, and 9105L597 except for mercury. The RPD for chromium (139%) exceeded the control limit of 35%. The associated sample results for chromium were qualified as estimates (J).

## SAMPLE RESULT VERIFICATION

Sample results and reported detection limits were recalculated to ensure that the reported results were accurate. Raw data was examined for anomalies, transcription errors and reduction errors. In addition the reviewer verified that the results fell within the linear range of the instrument.

Case A1-10-158 sample calculations were acceptable. No transcription errors or other anomalies were found.

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Sample calculations for Cases 9105L510, 9105L529, and 9105L597 were acceptable, with the exception of lead. Results for lead in sample B00VQ8 were reported from the method of standard additions. The laboratory multiplied the result by a factor of two. No explanation for this correction factor was provided in the laboratory's case narrative, and no reason for its use could be determined by review of the data. Therefore, the results were recalculated and new values were reported.

### *Reporting Near the Detection Limit*

Due to the variability of measurements at or near the detection limit all values less than five times ( $<5x$ ) the IDL were qualified as estimates (J).

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**TABLE 4-1**  
**300-FF-1 Operable Unit Remedial Investigation**  
**Metals Analysis and Qualifier Summary**  
**Thermo Analytical Case A1-10-158**  
**Roy F. Weston Cases 9105L510, 9015L529 and 9105L597**

Analyte	CRQL (mg/Kg)	Thermo Analytical Samples								Roy F. Weston Samples							
		B014R2		B014R3		B014R4		B014R5		B00VQ8		B00VQ9		B00VR0		B00VS0	
		(mg/Kg)	Q	(mg/Kg)	Q	(mg/Kg)	Q	(mg/Kg)	Q	(mg/Kg)	Q	(mg/Kg)	Q	(mg/Kg)	Q	(mg/Kg)	Q
Aluminum	200	5010		4440		4960		4590		3410		3470		3230		9600	
Antimony	60	9.8	UJ	9.7	UJ	9.8	UJ	9.7	UJ	2.62	UJ	2.76	UJ	2.85	UJ	3.46	UJ
Arsenic	10		R		R		R		R	1.1	J	0.92	J	1.1	J	1.9	
Barium	200	56.3		59.9		58.6		126		53		53.2		48.5		162	
Beryllium	5	0.21	U	0.21	U	0.34	J	0.21	U	0.26	J	0.29	J	0.28	J	1	
Cadmium	5	0.62	U	0.62	U	0.63	U	0.62	U	0.55	U	0.58	U	0.6	U	0.73	U
Calcium	5000	3560		5270		5340		19200		5430		4360		5190		4970	
Chromium	10	6.8		6		7.1		3.9		0.84	J	0.44	UJ	0.45	UJ	11.8	J
Cobalt	50	6.3	J	9.4		12.2		8		6.6		8.6		8.1		410.7	
Copper	25	13.9		14.4		17.5		14.1		11		9.3		8.9		10.4	
Iron	100	14400		19100		22700		16100		11000		14200		13900		19300	
Lead	3	4.3		3.3		3.6		3.7		1.7	J	1.3	J	1.2	J	4.8	J
Magnesium	5000	3270		3730		4010		3350		2640		2780		2860		5390	
Manganese	15	216		279		277		193		193	J	211	J	209	J	485	J
Mercury	0.2	0.09	U	0.1	U	0.1	U	0.1	U	0.1	UJ	0.1	UJ	0.1	UJ	0.12	UJ
Nickel	40	7.7	J	7.9	J	8.7		6.8	J	6.8		6.2		5.3		11.5	
Potassium	5000	950		904		781		838		700		557	J	384	J	3260	
Selenium	5		R		R		R		R	0.15	J	0.17	U	0.14	U	0.21	UJ
Silver	10	0.83	UJ	0.83	UJ	0.84	UJ	0.82	UJ		R		R		R		R
Sodium	5000	142	U	237		353		298		213		242		206		190	
Thallium	10	0.21	U	0.21	U	0.21	U	0.21	U	0.69	U	0.82	U	0.69	U	1.04	U
Vanadium	50	32.5		45.3		58.2		34		16.7		28.3		19.7		36.5	
Zinc	20	29		36.1		38.9		27.3		22.6	J	27.1	J	24.8	J	55.3	J
Cyanide	10	NA		NA		NA		NA		NA		NA		NA		NA	

CRQL = Contract Required Quantitation Limit  
 Q = Data qualifier assigned or verified during data validation  
 NA = Sample not analyzed for this parameter

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## 5.0 GENERAL CHEMISTRY DATA VALIDATION AND LIMITATIONS

### SUMMARY

#### *One Sample Delivery Group (SDG)*

One general chemistry case (10-160) is included in this report. Data qualifiers for this case is summarized in Table 5-1.

#### *Twenty Percent Validation*

A minimum of twenty percent of the sample results for Case 10-160 were validated. All reported results for quality assurance samples associated with this case were fully validated.

#### *WHC Validation Guidance Used*

Data validation was performed in accordance with the WHC Draft Data Validation Procedures for Chemical Analyses (WHC 1991a). Additional criteria established for the determination of laboratory performance was obtained from WHC (WHC 1991b), and the EPA's Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses (EPA 1988d).

#### *Samples Analyzed According to Non-CLP Protocols*

Four low level soil samples were submitted for analysis for general chemistry parameters (fluoride, nitrite, nitrate, sulfate, pH, percent moisture, and ammonia) by methods established by the EPA for wastewater analysis, modified for use on soil samples.

#### *Data Quality Objectives Not Defined*

The analyses were complete, but work plan CRDL requirements (WHC 1991b) were not defined, and could not be evaluated. With the exception of the analyses for pH and percent moisture, the methods used are modifications of EPA wastewater analyses which have not been standardized nor officially approved by EPA. Laboratory-specific sample

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preparation procedures were not described in the case documentation; however, reported results appear to be leachable versus total concentrations.

### ***No Data Rejected***

Although there were many deficiencies associated with these analyses, none of the data were rejected.

### ***Minor Deficiencies (Other Qualified Data)***

There were numerous minor deficiencies associated with the analysis of these samples. These include insufficient soil leaching procedure information; holding time exceedences; no LCS analyzed for ammonia or pH; and no daily calibration curve for ion chromatography analyses.

These qualifications have been explained in greater detail in the body of this report.

## **ANALYTICAL METHODS**

Performance of specific instrumental quality assurance and quality control procedures, including deficiencies noted during the quality assurance review are outlined below.

### ***Instrument Calibration***

Calibration was performed and was acceptable for analytes analyzed by ion chromatography (fluoride, nitrite, nitrate, and sulfate). The calibration was verified by analyzing an initial calibration verification standard, which was within the control window of 90 to 110 percent. However, the initial calibration was performed nearly one month prior to the sample analyses, and daily continuing calibration information was not provided, except on the day of analysis. As there was no daily linearity information, the results were qualified as estimates (J).

There was no calibration standard analyzed for pH, and the results were qualified as estimates (J).

### ***Blanks***

A blank was analyzed for the ion chromatography analytes and ammonia; results were found to be acceptable.

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**HOLDING TIMES EXCEEDED**

The samples were not analyzed within the holding times recommended by EPA for pH, fluoride, nitrite, nitrate, and sulfate. The associated data were qualified as estimates (UJ or J).

**ACCURACY*****Matrix Spike Analysis***

Matrix spike analyses are used to assess the analytical accuracy of the reported data and the effect of the matrix on the ability to accurately quantify sample concentrations.

All matrix spikes for ion chromatography analytes were added after the leaching step. These matrix spikes met the percent recovery criteria of 80 to 120 percent.

***Laboratory Control Sample***

The LCS monitors the overall performance of the analysis, including the sample preparation. An LCS should be prepared (e.g. digested or distilled) and analyzed with every group of samples which have been prepared together. The performance criteria for aqueous LCS percent recovery is 80-120 percent. The performance criteria for solid LCS samples are established through interlaboratory studies coordinated by a certifying agency (e.g. EPA or an independent commercial supplier).

A laboratory control sample was analyzed for the ion chromatography analytes, and was found to be acceptable. There were no laboratory control samples analyzed for pH or ammonia. The pH and ammonia sample concentrations were qualified as estimates (J).

**PRECISION*****Duplicate Analyses***

Analytical duplicate sample analyses are used to measure laboratory precision and sample homogeneity. Field duplicate analyses are used to measure both the laboratory and the field sampling procedure precision.

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Analytical duplicate results were acceptable and met the criteria of having less than 35 percent relative percent difference between measurements. There were transcription errors made on the duplicate reporting form, and the results were originally reported as being unacceptable. These were recalculated, and reported. There were no field duplicates included with these samples.

**SAMPLE RESULT VERIFICATION**

Sample results and reported detection limits were recalculated to ensure that the reported results were accurate. Raw data was examined for anomalies, transcription errors and reduction errors. In addition the reviewer verified that the results fell within the linear range of the instrument.

9 3 1 2 9 0 0 4 7 8



**TABLE 5-1**  
**300-FF-1 Operable Unit Remedial Investigation**  
**Conventional Wet Chemistry Analyses**  
**Thermo Analytical Case A1-10-158**

Analyte	Thermo Analytical Samples							
	B014R2		B014R3		B014R4		B014R5	
	Result	Q	Result	Q	Result	Q	Result	Q
Fluoride (mg/Kg)	0.45	J	10.72	J	10.78	J	15.59	J
Nitrite (mg/Kg)	0.5	UJ	0.5	UJ	0.5	UJ	0.58	J
Nitrate (mg/Kg)	2.19	J	2.254	J	2.24	J	9.23	J
Sulfate (mg/Kg)	26.49	J	35.13	J	32.43	J	95.8	J
Ammonia (mg/Kg)	15.96	J	10.64	J	9.31	J	9.42	J
Moisture (%)	4.64		5.51		5.1		6.64	
pH (dimensionless)	8.89	J	9.45	J	9.29	J	9.42	J

CRQL = Contract Required Quantitation Limit  
 Q = Data qualifier assigned or verified during data validation  
 NA = Sample not analyzed for this parameter

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**REFERENCES**

EPA 1988a. EPA Contract Laboratory Program Statement of Work for Organics Analyses, Multi-media, Multi-concentration. U.S. Environmental Protection Agency, Washington, D.C.

EPA 1988b. Laboratory data validation functional guidelines for evaluating organics analyses. U.S. Environmental Protection Agency, Washington D.C.

WHC 1991a. Draft Data Validation Procedures for Chemical Analyses (Draft 7/91). WHC Hanford Company, Washington

EPA 1988c. EPA Contract Laboratory Program Statement of Work for Inorganics Analyses, Multi-media, Multi-concentration. U.S. Environmental Protection Agency, Washington, D.C.

EPA 1988d. Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses. U.S. Environmental Protection Agency, Washington D.C.

EPA. 1989. Code of regulations, protection of the environment, 40 CFR part 136b, definition and procedure for the determination of the method detection limit.

EPA 1990. EPA Contract Laboratory Program Statement of Work for Inorganics Analyses, Multi-media, Multi-concentration. U.S. Environmental Protection Agency, Washington, D.C.

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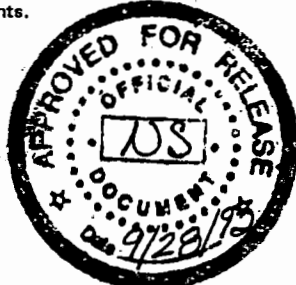
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