

JUN 21 1993

11

ENGINEERING DATA TRANSMITTAL

Page 1 of 1

1. EDT

142910

Station # 12

2. To: (Receiving Organization) Environmental Remedial Actions	3. From: (Originating Organization) Environmental Remedial Actions	4. Related EDT No.: NA
5. Proj./Prog./Dept./Div.:	6. Cog. Engr.: J.M. Frain	7. Purchase Order No.: NA
8. Originator Remarks: Document being distributed for release and approval		9. Equip./Component No.: NA
		10. System/Bldg./Facility: NA
11. Receiver Remarks:		12. Major Assm. Dwg. No.: NA
		13. Permit/Permit Application No.: NA
		14. Required Response Date: 6/11/93

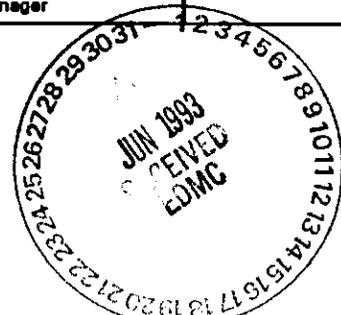
15. DATA TRANSMITTED					(F)	(G)	(H)	(I)
(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	Impact Level	Reason for Transmittal	Originator Disposition	Receiver Disposition
1	WHC-SD-EN-TI-159		0	White Bluffs Pickling Acid Crib Expedited Response Action Data Validation Report	3a	1/2	1	

16. KEY			
Impact Level (F)	Reason for Transmittal (G)		Disposition (H) & (I)
1, 2, 3, or 4 (see MRP 5.43)	1. Approval 2. Release 3. Information	4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)	1. Approved 2. Approved w/comment 3. Disapproved w/comment 4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged

17. SIGNATURE/DISTRIBUTION (See Impact Level for required signatures)											
(G)	(H)	(J) Name (K) Signature (L) Date (M) MSIN				(J) Name (K) Signature (L) Date (M) MSIN				(G)	(H)
Reason	Disp.									Reason	Disp.
1/2	1	Cog. Eng.	J.M. Frain	6/11/93	H6-07	ERC				H6-07	3
1/2	1	Cog. Mgr.	G.C. Henckel	6/14/93	H6-04	EDMC (2)				H6-08	3
1/2	1	QA	D.G. Farwick	6/14/93	H4-16	Central Files (2)				L8-04	3
		Safety									
		Env.									

18. J.M. Frain <i>J.M. Frain</i> Signature of EDT Originator 6/11/93 Date	19. G.C. Henckel <i>G.C. Henckel</i> Authorized Representative for Receiving Organization 6/14/93 Date	20. G.C. Henckel <i>G.C. Henckel</i> Cognizant/Project Engineer's Manager 6/14/93 Date	21. DOE APPROVAL (if required) Ltr. No. <input type="checkbox"/> Approved <input type="checkbox"/> Approved w/comments <input type="checkbox"/> Disapproved w/comments
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BD-7400-172-1 (02/89)

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ACRONYMS

%D	percent difference
BFB	bromofluorobenzene
CCV	continuing calibration verification
CLP	contract Laboratory Program
CRDL	contract Required Detection Limit
CRQL	contract Required Quantitation Limit
CVAA	cold vapor atomic absorption spectrophotometer
DFTPP	decafluorotriphenylphosphine
DOE	U.S. Department of Energy
EPA	U.S. Environmental Protection Agency
ERA	Expedited Response Action
GC/MS	gas chromatography/mass spectrometry
GFAA	graphite furnace atomic absorption spectrometer
ICP	inductively coupled argon plasma emission spectrometer
ICV	initial calibration verification
IDL	instrument detection limit
LCS	laboratory control sample
MDA	minimal detectable amount
MSA	method of standard additions
MS/MSD	matrix spike/matrix spike duplicate
QA	quality assurance
QC	quality control
RPD	relative percent difference
RRF	relative response factor
RSD	relative standard deviation
RRT	relative retention time
SDG	sample delivery group
SOW	statement of work
TAL	target analyte list
TCL	target compound list
TIC	tentatively identified compound
TMA	Thermo Analytical Laboratories
VOC	volatile organic compound
Weston	Roy F. Weston Company
WHC	Westinghouse Hanford Company

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1.0 INTRODUCTION

Laboratory data for soil samples collected during the White Bluffs Pickling Acid Cribs Expedited Response Action (ERA) have been reviewed and validated to ensure that they are of sufficient quality to support decisions regarding further actions to be taken at the White Bluffs Pickling Acid Cribs Operable Unit. This report summarizes the results previously presented to Westinghouse Hanford Company (WHC) in the Preliminary Quality Assurance Reports for the White Bluffs Pickling Acid Cribs ERA soil samples. In some instances, the data qualifiers originally presented in the Preliminary Quality Assurance Reports have been changed based upon further review of the data; these cases are highlighted in the text.

Throughout this report, various standard abbreviations have been used to note the qualifications associated with sample results. These abbreviations are summarized in Table 1-1.

1.1 CHEMICAL ANALYSES

Data from the chemical analysis of 29 samples from the White Bluffs Pickling Acid Cribs ERA and their related quality assurance (QA) 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. Three analytical cases were analyzed by Thermo Analytical Laboratories (TMA) and one was analyzed by Roy F. Weston (Weston) Laboratories using U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) or other EPA-approved protocols. Sample analyses included:

- Semivolatile organics (29 samples)
- Volatile organics (11 samples)
- Metals (29 samples)
- Anions (29 samples)
- Nitrate/Nitrite (29 samples)

A summary indicating the validation level of effort per sample is given in Table 1-2.

1.2 RADIOCHEMICAL ANALYSES

Data from one TMA radiochemical case and one Weston/Ecotek radiochemical case were validated. The laboratory used analytical protocols specified in the White Bluffs Pickling Acid Crib Expedited Response Action Project Plan (WHC 1992c). Radiochemical analyses consisted of gamma spectroscopy performed on 11 soil samples.

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1.3 WHC GUIDANCE USED

Data quality was reviewed and analytical results were validated using EPA CLP protocols and guidelines and related WHC procedures.

1.4 MAJOR DEFICIENCIES

Semivolatile Organic Analyses. No sample data were rejected due to deficiencies in data quality.

Volatile Organic Analyses. No sample data were rejected due to deficiencies in data quality.

Metals Analyses. No sample data were rejected due to deficiencies in data quality.

Anion Analyses. No sample data were rejected due to deficiencies in data quality.

Nitrate/Nitrite Analyses. No sample data were rejected due to deficiencies in data quality.

Radiochemical Analysis. No sample data were rejected due to deficiencies in data quality.

1.5 GENERAL QUALITY TRENDS

Several general quality trends, which resulted in data qualification, were observed. These include the following:

- The semivolatile organics method blanks contained tentatively identified compounds (TIC).
- The Weston semivolatile organics matrix spike percent recoveries were outside the control limits and were not comparable to the TMA data.
- Methylene chloride and acetone were detected in the volatile organic method blanks.
- Trace concentrations of methylene chloride and toluene were detected in several of the samples. Toluene and methylene chloride are common laboratory contaminants. These compounds were not detected in each of the method blanks, however, at the low concentrations detected, it is not possible to verify that the source of the contamination is the site.
- The overall accuracy goal of $\pm 25\%$ was not met for antimony. Despite exceedences and qualifications made based on the validation guidelines, the overall precision and accuracy goals were met for all of the other metals.

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- There were several metals data points qualified as undetected or estimates due to blank contamination or negative blank concentrations.
- There were several holding time exceedences for the anions and nitrate/nitrite analyses.
- The TMA anion initial calibrations were not performed on the dates of the analyses.
- The TMA radiochemical results were acceptable with no qualifications. The Weston/ECOTek radiochemical results were qualified as estimates (J) due to lack of an acceptable laboratory control sample.

Table 1-1. Glossary of Data Qualifiers.

- U** Indicates the compound or analyte was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory.
- UJ** Indicates the compound or analyte was analyzed for and not detected. Due to quality control (QC) deficiencies identified during data validation the value reported may not accurately reflect the sample quantitation limit.
- J** Indicates the compound or analyte was analyzed for and detected. The associated value is estimated but the data are useable for decision making processes.
- R** Indicates the compound or analyte was analyzed for and due to an identified QC deficiency the data are not useable.
- JN** Indicates presumptive evidence of a compound at an estimated value. Data are not useable for decision making purposes.
- UJN** Indicates the compound or analyte was originally identified from presumptive evidence. Due to QC deficiencies identified during data validation the value reported may not accurately reflect the sample quantitation limit. Data are not useable for decision making purposes.

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Table 1-2. White Bluffs Pickling Acid Crib ERA Sample Summary

Laboratory	Case Number	Sample Number	BNA	VOC	METALS	ANIONS	NO2/NO3	GAMMA
Weston	9212L005	B07Q13	C	C	F	F	F	F
TMA	A212011/N212014	B07PY8	C	C	F	F	F	C
TMA	A212011/N212014	B07PZ0	C		F	F	F	
TMA	A212049/N212069	B07Q04	C		F	F	F	
TMA	A212049/N212069	B07Q05	C		F	F	F	
TMA	A212049/N212069	B07Q06	C		F	F	F	
TMA	A212049/N212069	B07Q07	C		F	F	F	
TMA	A212049/N212069	B07Q08	C		F	F	F	
TMA	A212049/N212069	B07Q09	C		F	F	F	
TMA	A212049/N212069	B07Q10	C		F	F	F	
TMA	A212049/N212069	B07Q11	C		F	F	F	
TMA	A212049/N212069	B07Q12	C	C	F	F	F	C
TMA	A212049/N212069	B07Q14	C		F	F	F	
TMA	A212049/N212069	B07Q15	C		F	F	F	
TMA	A212049/N212069	B07Q16	C		F	F	F	
TMA	A212018/N212014	B07PY9	F		F	F	F	
TMA	A212018/N212014	B07PZ1	F	F	F	F	F	C
TMA	A212018/N212014	B07PZ2	F	F	F	F	F	F
TMA	A212018/N212014	B07PZ3	F		F	F	F	
TMA	A212018/N212014	B07PZ4	F	F	F	F	F	C
TMA	A212023/N212014	B07PZ5	C		F	F	F	
TMA	A212023/N212014	B07PZ6	C	C	F	F	F	C
TMA	A212023/N212014	B07PZ7	C	C	F	F	F	C
TMA	A212023/N212014	B07PZ8	C		F	F	F	
TMA	A212023/N212014	B07PZ9	C		F	F	F	
TMA	A212023/N212014	B07Q00	C		F	F	F	
TMA	A212023/N212014	B07Q01	C	C	F	F	F	C
TMA	A212023/N212014	B07Q02	C	C	F	F	F	F
TMA	A212023/N212014	B07Q03	C	C	F	F	F	C

- C** Cursory review (Level II validation) of quality control data, full review of sample calculations.
- F** Full review (Level IV validation) of quality control data and sample data.
- BNA** Base, neutral, and acid extractable organic compounds (semivolatiles).
- VOC** Volatile organic compounds.
- METALS** Metals (aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, zinc, and zirconium).
- ANIONS** Chloride, fluoride, phosphate, and sulfate.
- NO2/NO3** Nitrate plus nitrite.
- GAMMA** Gamma spectroscopy (Cobalt-60, Cesium-137, and Thorium-228).

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2.0 SEMIVOLATILE ORGANIC DATA VALIDATION AND LIMITATIONS

2.1 SUMMARY

2.1.1 Four Sample Delivery Groups

Sample results from four semivolatile organic cases are included in this report:

Laboratory	Case Number	No. of Samples	No. Fully Validated
TMA	A212018/023	14	13
TMA	A212011	2	2
Weston	9212L005	1	1
TMA	A212049	12	12

Data qualifiers assigned to the semivolatile compound data for these cases are summarized in Table 2-1.

2.1.2 All Samples Validated

Results for all the sample analyses for the cases listed above were validated, and data qualifiers assigned as appropriate. All of the reported results for QA samples associated with these cases were reviewed. For Case A212018/023, 100% of the QA sample results were recalculated and quality control (QC) calculations verified. A limited number of samples, specified by WHC, were fully validated (i.e., all sample results were recalculated from the laboratory raw data).

2.1.3 WHC Validation Guidance Used

Data validation was performed in accordance with the WHC Data Validation Procedures for Chemical Analyses (WHC 1992a). Additional criteria established for the determination of laboratory performance were obtained from WHC (WHC 1992c), and the EPA's Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses (EPA 1990c).

2.1.4 Samples Analyzed According to CLP Protocols

Twenty-nine low-level soil samples were submitted for analysis. Analyses were performed according to the 1990 CLP protocol (EPA 1990a).

9212018-0019

Sample analyses were performed using two gas chromatograph/mass spectrometer (GC/MS) systems. Both systems were outfitted with capillary columns. The data were evaluated against equivalent QC requirements and are comparable. Failure to comply with various technical requirements established by CLP protocols resulted in qualification of the data. The specific problems observed during the QA review are detailed in the sections below.

The analysis was complete and met the method and work plan contract required quantification limit (CRQL) requirements (WHC 1992c) in all cases.

2.1.5 Minor Deficiencies Noted

There were minor deficiencies associated with the analyses which resulted in the qualification of data. These included minor blank contamination, minor matrix spike exceedences, and sample concentrations reported below the quantification limit. These deficiencies and the resulting data qualifications are explained in greater detail below.

2.2 ANALYTICAL METHOD

2.2.1 Gas Chromatography/Mass Spectrometer Tuning Criteria Met

Tuning is performed to ensure that mass resolution, identification, and, to some degree, sensitivity of the GC/MS instrument have been established. When analyzing for semivolatile organics, instrument tuning is performed with decafluorotriphenylphosphine (DFTPP). 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 DFTPP are outlined in the WHC data validation guidelines (WHC 1992a) and in the CLP Statement of Work (SOW) (EPA 1990a).

The original tuning data were checked for transcription and calculation errors in one of the packages (Case A212018/023). In the remaining data packages, tuning and mass calibration summary forms (Form V) were evaluated to verify that tuning criteria were met. Prior to calibration and sample analysis, tuning criteria were met and no data were qualified based on the tuning results.

2.2.2 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 during analytical runs.

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

2.2.2.1 No Initial Calibration Exceedences. Instrument response is established when the relative response factors (RRF) for all target compounds are greater than or equal to the minimum criteria specified in the CLP SOW (EPA 1990a). Linearity is established when the relative standard deviations (RSD) of the RRF are less than or equal to 20.5% (EPA 1990a). Only some of the compounds are required to meet these criteria, and minor exceedences of common problem compounds do not cause qualification of the data.

For each of the cases, the initial calibrations met the criteria. Therefore, no data were qualified based on the initial calibration results.

2.2.2.2 No Continuing Calibration Exceedences. The criteria for accepting the continuing calibration require that a mid-range standard be analyzed at least once per 12-hour period and that the RRF of all target compounds be greater than or equal to the minimum criteria specified in the CLP SOW (EPA 1990a). In addition, the percent difference (%D) of these RRFs must be less than or equal to 25% (EPA 1990a) of the average RRF calculated for the associated initial calibration. Only some of the compounds are required to meet these criteria, and minor exceedences of common problem compounds do not cause qualification of the data.

The required analysis frequency and criteria for continuing calibration were met for the cases, and no data were qualified based on the continuing calibration results.

2.2.3 Blanks

Method blank and field blank analyses are performed to determine the extent of laboratory or field contamination of samples. If the sample concentration for a compound is less than 5 times the blank concentration (10 times if the compound is a common laboratory contaminant), the sample concentration is qualified as undetected (U).

2.2.3.1 Minor Method Blank and Equipment Blank Contamination. One method blank was extracted with each sample delivery group. Several compounds were detected in the blanks at low concentrations. These included di-n-butyl phthalate and TICs. Based on the five times and ten times criteria, associated sample data were qualified as undetected (U), as outlined below.

Case A212018. Four TICs were detected in the blank associated with this case. Associated TIC concentrations in samples B07PY9, B07PZ1, B07PZ2, B07PZ3, B07PZ4, B07PZ5, B07PZ6, B07PZ7, B07PZ8, B07PZ9, B07Q00, B07Q01, B07Q02, and B07Q03 that were less than five times the blank concentration were qualified as undetected at estimated concentrations (UJN). A summary of these qualifications can be found on Form B-3 of the data validation documentation found in the WHC project files.

Case A212011. Five TICs were detected in the blank associated with this case. Associated TIC concentrations in samples B07PY8 and B07PZ0 that were less than five times the blank concentration were qualified as undetected at estimated concentrations (UJN). A summary of these qualifications can be found on Form B-3 of the data validation documentation found in the WHC project files.

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One target compound, di-n-butyl phthalate, was detected in the blank associated with this case. The di-n-butyl phthalate concentration in sample B07PY8 was qualified as undetected (U) at the CRQL.

An equipment blank, sample B07PZ0, was analyzed with this case. One TIC that was reported in the equipment blank was also reported in sample B07PY8. The TIC concentration in sample B07PY8 was qualified as undetected at an estimated concentration (UJN).

Case 9212L005. Aldol condensation products (TIC) and three additional TICs were detected in the blank associated with this case. Associated aldol condensation products and one TIC compound whose concentrations (B07Q13) were less than five times the blank concentration were qualified as undetected at estimated concentrations (UJN).

The case narrative stated that a method blank was analyzed with the sample and di-n-butyl phthalate was detected. The laboratory then analyzed another portion of the extract and reported the results. The initial blank results were not reported. Di-n-butyl phthalate was detected in sample B07Q13 and should be qualified as undetected (U) based on the initial blank results. No action was taken, however, as qualifications were made only on the reported data.

Case A212049. Six TICs were detected in the blank associated with this case. Associated TIC concentrations in samples B07Q04, B07Q05, B07Q06, B07Q07, B07Q08, B07Q09, B07Q10, B07Q11, B07Q12, B07Q14, B07Q15, and B07Q16 that were less than five times the blank concentration were qualified as undetected at estimated concentrations (UJN). A summary of these qualifications can be found on Form B-3 of the data validation documentation found in the WHC project files.

2.3 HOLDING TIMES

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

The holding times were acceptable for all of the samples associated with these four cases. Therefore, no qualifiers were assigned based on the holding time.

WHC data validation procedures specify holding times for semivolatiles analyses that 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 1992a). Using WHC guidelines, the following results would also be qualified; based upon EPA guidelines, no qualifiers were assigned.

9212L005

Case A212018/A212023. Using WHC guidelines, data from samples B07PZ1, B07PZ2, and B07PZ4 would be qualified upon holding times, as these samples were extracted 8 days after collection.

Case 9212L005. Data from sample B07Q13 would be qualified based upon holding times, as this sample was extracted 10 days after collection.

Case A212049. Data from sample B07Q12 would be qualified based upon holding times, as this sample was extracted 11 days after collection.

2.4 ACCURACY

The overall accuracy goal for the target compounds is $\pm 25\%$. Accuracy was assessed by evaluating the recoveries of stable isotopically labeled surrogate compounds added to all samples and blanks, matrix spikes, and by the analysis of a representative sample, which was spiked with a variety of semivolatile organic compounds.

2.4.1 Surrogate Compound Recovery Acceptable

Matrix-specific surrogate compound recovery control windows have been established by the EPA CLP program (EPA 1990a). When a surrogate compound recovery is out of the control window, 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 EPA surrogate recovery control windows (EPA 1990) are calculated using the following formula:

$$EPA \text{ Control Limit} = \overline{\%R} \pm (\tau * Std. Dev.)$$

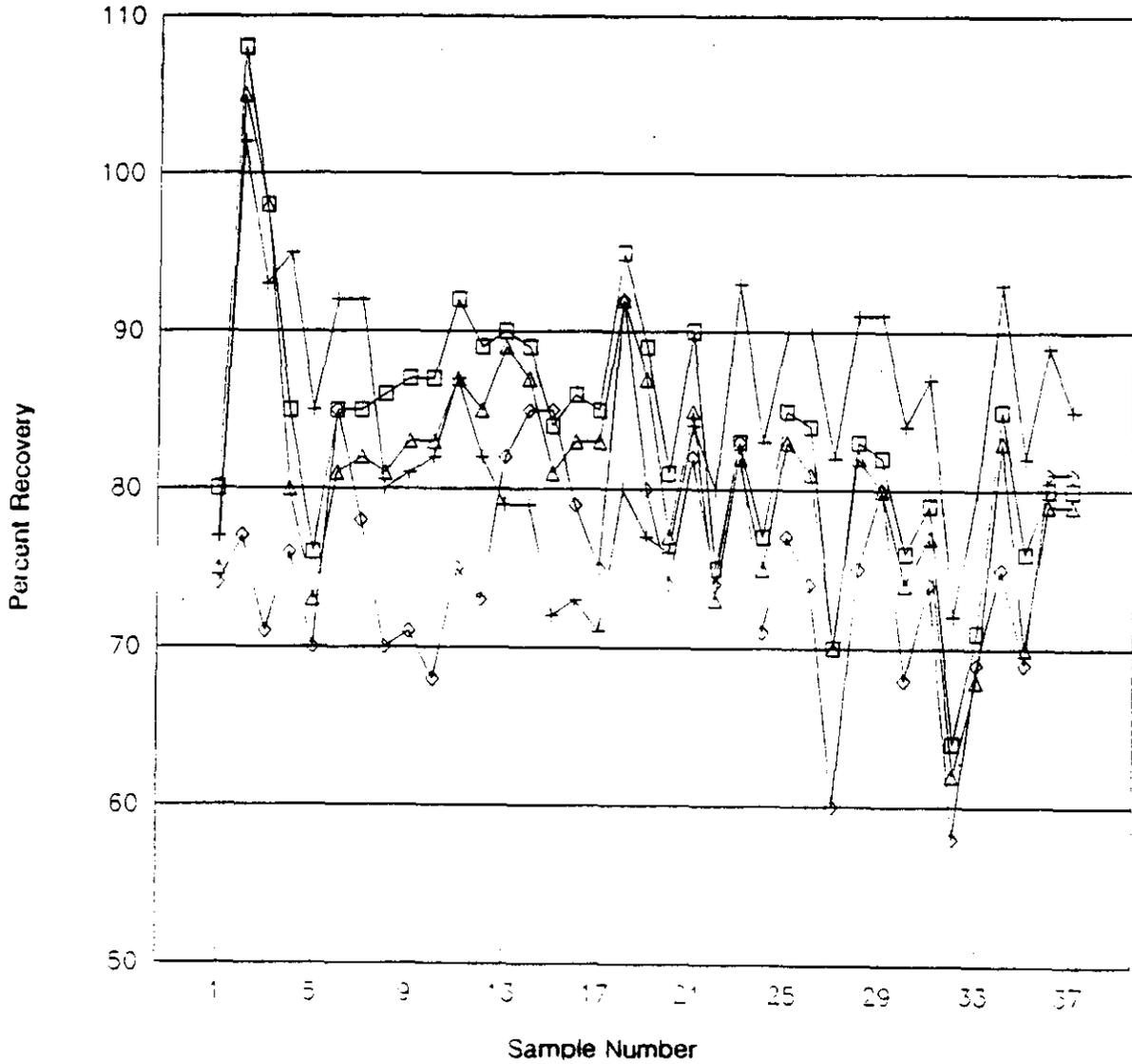
where:

$$\tau = \text{Student } \tau \text{ Value at the 99 Percent Confidence Level}$$

The surrogate recovery distribution for the White Bluffs Pickling Acid Crib ERA data was calculated in the same manner. Figures 2-1 and 2-2 show that the White Bluffs Pickling Acid Crib ERA surrogate recovery distribution is comparable to the EPA general population data for this method. The percent recovery distribution is normal and there are no trends.

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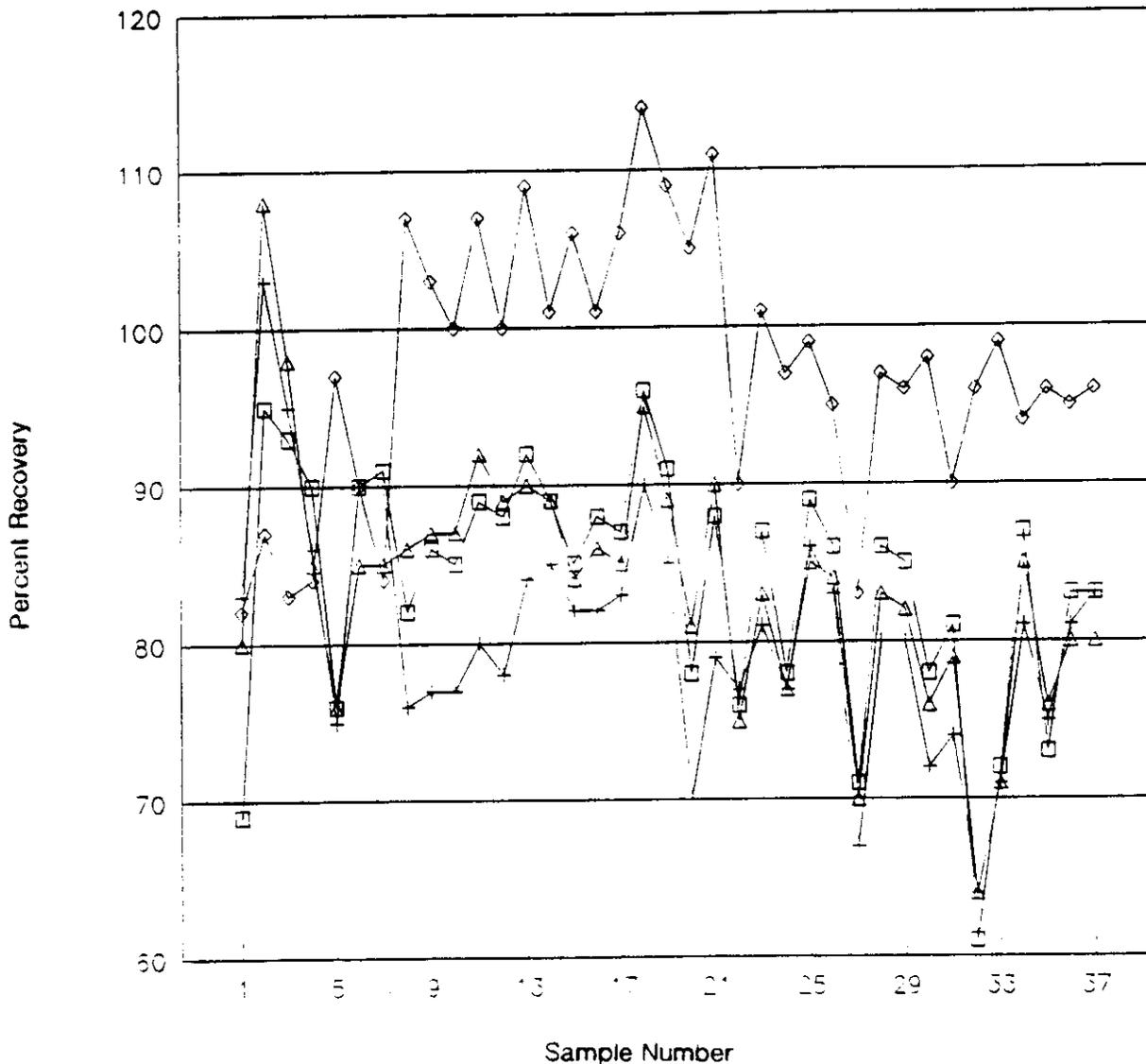
Figure 2-1. White Bluffs Pickling Acid Crib ERA
Acid Semivolatle Surrogate Recovery



	□ Phenol-d5	+ 2-Fluorophenol	o 2,4,6Tribromophenol	Δ 2-Chlorophenol-d4
Mean:	83.7	84.1	75.4	80.9
Variance:	62.4	51.6	45.7	61.6
Unbiased Std. Dev.:	8.0	7.3	6.9	8.0
2.44*Std. Dev.:	19.5	17.8	16.7	19.4
Range (Mean +/- (2.44*Std. Dev.):	64-103	66-102	59-92	62-100
EPA Limits:	24-113	25-121	19-122	20-130

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Figure 2-2. White Bluffs Pickling Acid Crib ERA
Base/Neutral Semivolatile Surrogate Recovery



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	□ Nitrobenzene	+ 2-Fluorobiphenyl	◇ Terphenyl-d14	△ 1,2-Dichlorobenzene
Mean:	83.9	80.6	97.5	79.9
Variance:	59.0	55.1	68.2	43.6
Unbiased Std. Dev.:	7.8	7.5	8.4	6.7
2.44*Std. Dev.:	19.0	18.4	20.4	16.3
Range (Mean +/- (2.44*Std. Dev.):	65-103	62-99	77-118	64-96
EPA Limits:	23-120	30-115	18-137	20-130

The surrogate compound recoveries calculated for the eight stable isotopically labeled surrogate compounds were acceptable. Therefore, no qualifiers were assigned based on surrogate recovery.

2.4.2 Matrix Spike Recoveries High for Weston Sample

Matrix spike compounds are added to a sample that is representative of the sample delivery group. Matrix spike analyses are performed in duplicate using eleven compounds specified by CLP protocols. The recoveries for the eleven compounds must be within the sample matrix established QC limits (EPA 1990a). The matrix spike analyses estimate the interference with target compounds, either positively or negatively.

Four soil matrix spike samples and four soil matrix spike duplicate samples were analyzed with the cases addressed in this report. With the exception of minor percent recovery exceedences for the Weston sample, the matrix spike compound recoveries were acceptable. Therefore, no qualifiers were assigned based on matrix spike percent recoveries.

The EPA matrix spike recovery control windows (EPA 1990) are calculated using the following formula:

$$EPA\ Control\ Limit = \overline{\%R} \pm (\tau * Std. Dev.)$$

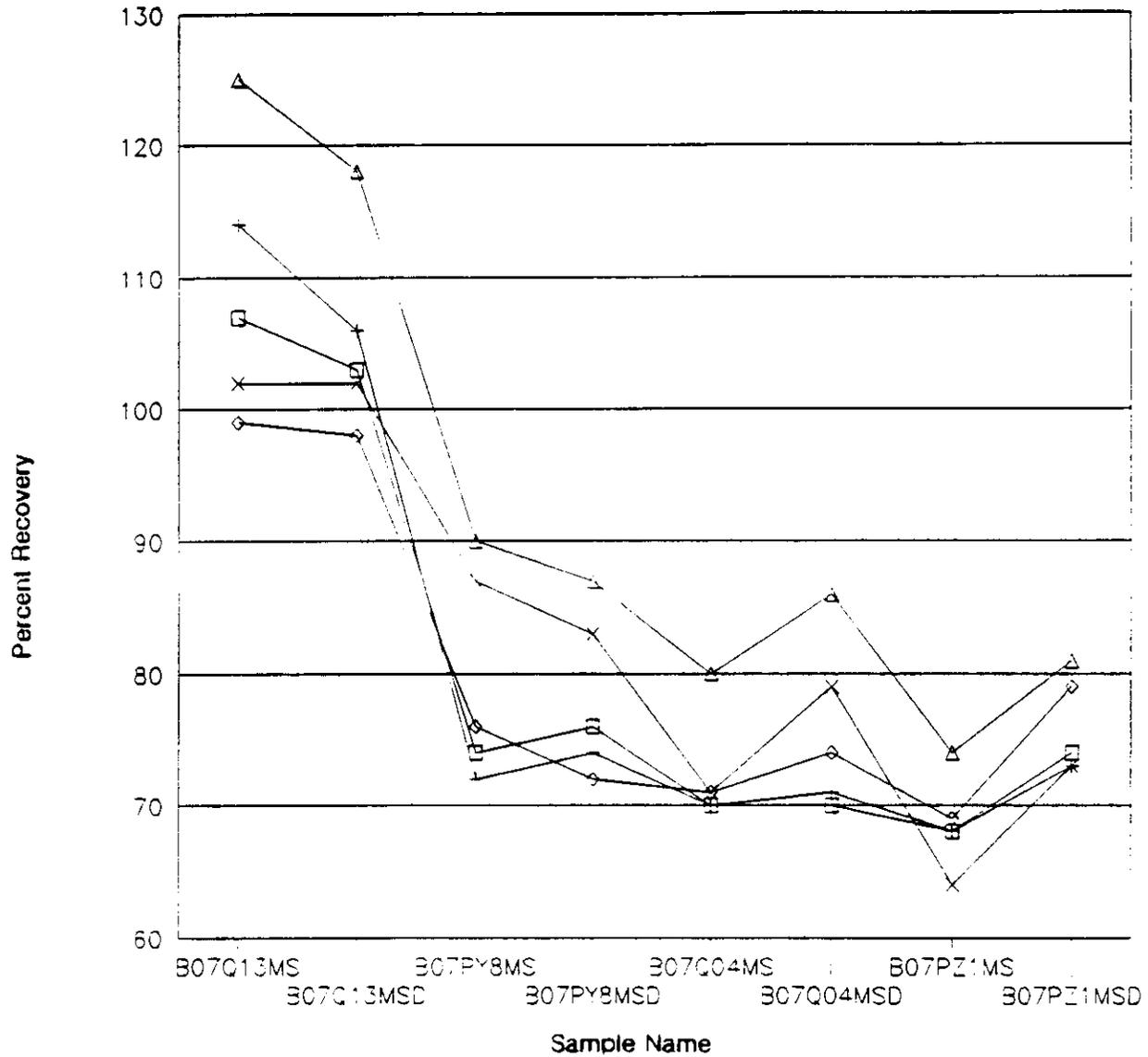
where:

$$\tau = Student\ \tau\ Value\ at\ the\ 99\ Percent\ Confidence\ Level$$

Although the White Bluffs Pickling Acid Crib ERA matrix spike population is small (eight matrix spike samples), the matrix spike recovery distribution was calculated in the same manner. Figures 2-3 and 2-4 show that the matrix spike recovery distribution is comparable to the EPA general population data for this method for the TMA data. When Weston matrix spike recoveries, which were outside the control windows are included, the control limits exceed the EPA limits. As the Weston matrix spike data are associated with only one sample in which no compounds were detected, no qualifiers were assigned.

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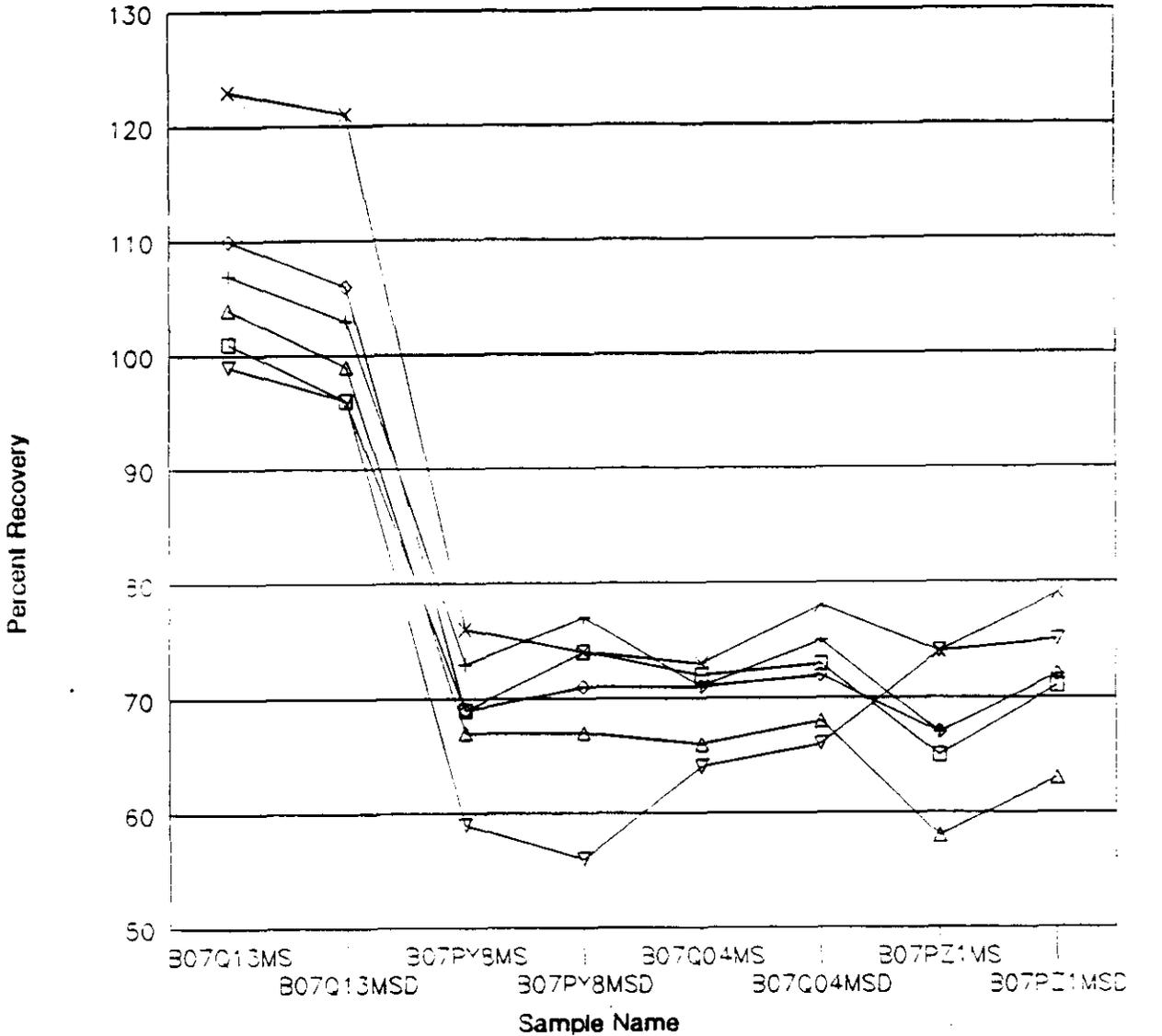
Figure 2-3. White Bluffs Pickling Acid Crib ERA
Acid Matrix Spike Recovery



	□ Phenol	+ 2-Chlorophenol	◇ 4-Chloro-3-methylphenol
Mean:	80.3	81.0	79.8
Variance:	211.2	287.3	125.4
Unbiased Std. Dev.:	15.5	18.1	12.0
2.998*Std. Dev.:	46.6	54.3	35.9
Range (Mean +/- (2.998*Std. Dev.)):	34-127	27-135	44-116
EPA Limits:	26-90	25-102	26-103
	Δ 4-Nitrophenol	X Pentachlorophenol	
Mean:	92.6	82.6	
Variance:	302.0	169.7	
Unbiased Std. Dev.:	18.6	13.9	
2.998*Std. Dev.:	55.7	41.8	
Range (Mean +/- (2.998*Std. Dev.)):	37-148	41-124	
EPA Limits:	11-114	17-109	

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Figure 2-4. White Bluffs Pickling Acid Crib ERA
Base/Neutral Matrix Spike Recovery



1,4-Dichlorobenzene + N-Nitroso-di-n-propylamine ○ 1,2,4 Trichlorobenzene

Mean:	77.6	80.6	79.8
Variance:	153.5	206.5	269.4
Unbiased Std. Dev.:	13.2	15.4	17.5
2.998*Std. Dev.:	39.7	46.1	52.6
Range (Mean +/- (2.998*Std. Dev.)):	38-117	35-127	27-132
EPA Limits:	28-104	41-126	38-107

	△ Acenaphthene	X 2,4-Dinitrotoluene	▽ Pyrene
Mean:	74.0	87.3	73.6
Variance:	262.5	406.4	227.7
Unbiased Std. Dev.:	17.3	21.6	16.1
2.998*Std. Dev.:	51.9	64.6	48.4
Range (Mean +/- (2.998*Std. Dev.)):	22-126	23-152	25-122
EPA Limits:	31-137	28-89	35-142

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2.5 PRECISION ACCEPTABLE

The overall precision goal for the target compounds is $\pm 35\%$. Analytical 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 matrix spike/matrix spike duplicate (MS/MSD) analyses, precision may also be assessed using unspiked duplicate sample analyses. Field precision is measured by analyzing duplicate samples taken in the field. Interlaboratory precision is measured by analyzing duplicate samples ("field splits") by two analytical laboratories.

2.5.1 Matrix Spike Duplicates Acceptable

Four soil matrix spike duplicate samples were analyzed with the cases addressed in this report. The matrix spike duplicate RPDs were acceptable for all four cases. Therefore, no qualifiers were assigned based on the matrix spike duplicate precision.

2.5.2 Field Duplicates Analyzed

One set of field duplicate samples (B07Q02 and B07Q01) was analyzed. The target compounds were not detected or were detected at concentrations less than the CRQL. Therefore, field duplicate precision could not be quantitatively evaluated. No qualifiers were assigned based on field duplicate precision data.

2.5.3 Interlaboratory Precision

One set of field split samples (B07Q12 and B07Q13) was analyzed. Target compounds were not detected or were detected at concentrations less than the CRQL. Therefore, interlaboratory precision could not be quantitatively evaluated. No qualifiers were assigned based on interlaboratory precision data.

2.6 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% or -50% 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. Therefore, no qualifiers were assigned based on internal standard performance.

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Table 2-1. White Bluffs Pickling Acid Crib ERA
Semivolatile Organic Compounds Analysis and Qualifier Summary
(Sheet 1 of 6)

Sample Number:	B07PY8	B07PY9	B07PZ0	B07PZ1	B07PZ2	B07PZ3	B07PZ4	B07PZ5	B07PZ6	B07PZ7
Units:	µg/kg									
Phenol	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
bis(2-Chloroethyl)Ether	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2-Chlorophenol	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
1,3-Dichlorobenzene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
1,4-Dichlorobenzene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2-Methylphenol	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2,2'-oxybis(1-Chloropropane)	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
4-Methylphenol	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
N-Nitroso-Di-n-Propylamine	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Hexachloroethane	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Nitrobenzene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Isophorone	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2-Nitrophenol	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2,4-Dimethylphenol	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
bis(2-Chloroethoxy)Methane	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2,4-Dichlorophenol	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
1,2,4-Trichlorobenzene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Naphthalene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
4-Chloroaniline	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Hexachlorobutadiene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
4-Chloro-3-Methylphenol	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2-Methylnaphthalene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Hexachlorocyclopentadiene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2,4,6-Trichlorophenol	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2,4,5-Trichlorophenol	870 U	800 U	780 U	840 U	810 U	840 U	850 U	840 U	820 U	820 U
2-Chloronaphthalene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2-Nitroaniline	870 U	800 U	780 U	840 U	810 U	840 U	850 U	840 U	820 U	820 U
Dimethylphthalate	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Acenaphthylene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
3-Nitroaniline	870 U	800 U	780 U	840 U	810 U	840 U	850 U	840 U	820 U	820 U
Acenaphthene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2,4-Dinitrophenol	870 U	800 U	780 U	840 U	810 U	840 U	850 U	840 U	820 U	820 U

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Table 2-1. White Bluffs Pickling Acid Crib ERA
Semi-volatile Organic Compounds Analysis and Qualifier Summary
(Sheet 2 of 6)

Sample Number: Units:	B07PY8 µg/kg	B07PY9 µg/kg	B07PZ0 µg/kg	B07PZ1 µg/kg	B07PZ2 µg/kg	B07PZ3 µg/kg	B07PZ4 µg/kg	B07PZ5 µg/kg	B07PZ6 µg/kg	B07PZ7 µg/kg
4-Nitrophenol	870 U	800 U	780 U	840 U	810 U	840 U	850 U	840 U	820 U	820 U
Dibenzofuran	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2,4-Dinitrotoluene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
2,6-Dinitrotoluene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Diethylphthalate	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
4-Chlorophenyl-phenylether	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Fluorene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
4-Nitroaniline	870 U	800 U	780 U	840 U	810 U	840 U	850 U	840 U	820 U	820 U
4,6-Dinitro-2-methylphenol	870 U	800 U	780 U	840 U	810 U	840 U	850 U	840 U	820 U	820 U
N-Nitrosodiphenylamine (1)	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
4-Bromophenyl-phenylether	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Hexachlorobenzene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Pentachlorophenol	870 U	800 U	780 U	840 U	810 U	840 U	850 U	840 U	820 U	820 U
Phenanthrene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Anthracene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Carbazole	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Di-n-Butylphthalate	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Fluoranthene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Pyrene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Butylbenzylphthalate	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
3,3'-Dichlorobenzidine	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Benzo(a)Anthracene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
bis(2-Ethylhexyl)Phthalate	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Chrysene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Di-n-Octyl Phthalate	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Benzo(b)Fluoranthene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Benzo(k)Fluoranthene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Benzo(a)Pyrene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Indeno(1,2,3-cd)Pyrene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Dibenz(a,h)Anthracene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U
Benzo(g,h,i)Perylene	360 U	330 U	320 U	350 U	330 U	340 U	350 U	340 U	340 U	340 U

Table 2-1. White Bluffs Pickling Acid Crib ERA
Semi-volatile Organic Compounds Analysis and Qualifier Summary
(Sheet 3 of 6)

Sample Number:	B07PZ8	B07PZ9	B07Q00	B07Q01	B07Q02	B07Q03	B07Q04	B07Q05	B07Q06	B07Q07
Units:	µg/kg									
Phenol	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
bis(2-Chloroethyl)Ether	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2-Chlorophenol	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
1,3-Dichlorobenzene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
1,4-Dichlorobenzene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
1,2-Dichlorobenzene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2-Methylphenol	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2,2'-oxybis(1-Chloropropane)	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
4-Methylphenol	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
N-Nitroso-Di-n-Propylamine	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Hexachloroethane	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Nitrobenzene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Isophorone	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2-Nitrophenol	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2,4-Dimethylphenol	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
bis(2-Chloroethoxy)Methane	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2,4-Dichlorophenol	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
1,2,4-Trichlorobenzene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Naphthalene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
4-Chloroaniline	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Hexachlorobutadiene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
4-Chloro-3-Methylphenol	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2-Methylnaphthalene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Hexachlorocyclopentadiene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2,4,6-Trichlorophenol	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2,4,5-Trichlorophenol	810 U	920 U	820 U	860 U	860 U	830 U	860 U	820 U	810 U	800 U
2-Chloronaphthalene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2-Nitroaniline	810 U	920 U	820 U	860 U	860 U	830 U	860 U	820 U	810 U	800 U
Dimethylphthalate	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Acenaphthylene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
3-Nitroaniline	810 U	920 U	820 U	860 U	860 U	830 U	860 U	820 U	810 U	800 U
Acenaphthene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2,4-Dinitrophenol	810 U	920 U	820 U	860 U	860 U	830 U	860 U	820 U	810 U	800 U

Table 2-1. White Bluffs Pickling Acid Crib ERA
Semi-volatile Organic Compounds Analysis and Qualifier Summary
(Sheet 4 of 6)

Sample Number:	B07PZ8	B07PZ9	B07Q00	B07Q01	B07Q02	B07Q03	B07Q04	B07Q05	B07Q06	B07Q07
Units:	µg/kg									
4-Nitrophenol	810 U	920 U	820 U	860 U	860 U	830 U	860 U	820 U	810 U	800 U
Dibenzofuran	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2,4-Dinitrotoluene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
2,6-Dinitrotoluene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Diethylphthalate	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
4-Chlorophenyl-phenylether	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Fluorene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
4-Nitroaniline	810 U	920 U	820 U	860 U	860 U	830 U	860 U	820 U	810 U	800 U
4,6-Dinitro-2-methylphenol	810 U	920 U	820 U	860 U	860 U	830 U	860 U	820 U	810 U	800 U
N-Nitrosodipheylamine (1)	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
4-Bromophenyl-phenylether	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Hexachlorobenzene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Pentachlorophenol	810 U	920 U	820 U	860 U	860 U	830 U	860 U	820 U	810 U	800 U
Phenanthrene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Anthracene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Carbazole	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Di-n-Butylphthalate	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Fluoranthene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Pyrene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Butylbenzylphthalate	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
3,3'-Dichlorobenzidine	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Benzo(a)Anthracene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
bis(2-Ethylhexyl)Phthalate	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Chrysene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Di-n-Octyl Phthalate	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Benzo(b)Fluoranthene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Benzo(k)Fluoranthene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Benzo(a)Pyrene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Indeno(1,2,3-cd)Pyrene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Dibenz(a,h)Anthracene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U
Benzo(g,h,i)Perylene	340 U	380 U	340 U	360 U	350 U	340 U	360 U	340 U	330 U	330 U

Table 2-1. White Bluffs Pickling Acid Crib ERA
Semivolatile Organic Compounds Analysis and Qualifier Summary
(Sheet 5 of 6)

Sample Number:	B07Q08	B07Q09	B07Q10	B07Q11	B07Q12	B07Q13	B07Q14	B07Q15	B07Q16
Units:	µg/kg								
Phenol	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
bis(2-Chloroethyl)Ether	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2-Chlorophenol	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
1,3-Dichlorobenzene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
1,4-Dichlorobenzene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
1,2-Dichlorobenzene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2-Methylphenol	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2,2'-oxybis(1-Chloropropane)	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
4-Methylphenol	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
N-Nitroso-Di-n-Propylamine	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Hexachloroethane	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Nitrobenzene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Isophorone	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2-Nitrophenol	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2,4-Dimethylphenol	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
bis(2-Chloroethoxy)Methane	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2,4-Dichlorophenol	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
1,2,4-Trichlorobenzene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Naphthalene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
4-Chloroaniline	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Hexachlorobutadiene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
4-Chloro-3-Methylphenol	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2-Methylnaphthalene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Hexachlorocyclopentadiene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2,4,6-Trichlorophenol	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2,4,5-Trichlorophenol	810 U	820 U	840 U	820 U	810 U	840 U	840 U	830 U	840 U
2-Chloronaphthalene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2-Nitroaniline	810 U	820 U	840 U	820 U	810 U	840 U	840 U	830 U	840 U
Dimethylphthalate	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Acenaphthylene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
3-Nitroaniline	810 U	820 U	840 U	820 U	810 U	840 U	840 U	830 U	840 U
Acenaphthene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2,4-Dinitrophenol	810 U	820 U	840 U	820 U	810 U	840 U	840 U	830 U	840 U

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Sample Number:	B07Q08	B07Q09	B07Q10	B07Q11	B07Q12	B07Q13	B07Q14	B07Q15	B07Q16
Units:	µg/kg								
4-Nitrophenol	810 U	820 U	840 U	820 U	810 U	840 U	840 U	830 U	840 U
Dibenzofuran	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2,4-Dinitrotoluene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
2,6-Dinitrotoluene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Diethylphthalate	330 U	110 J	350 U	340 U	330 U	340 U	350 U	340 U	350 U
4-Chlorophenyl-phenylether	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Fluorene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
4-Nitroaniline	810 U	820 U	840 U	820 U	810 U	840 U	840 U	830 U	840 U
4,6-Dinitro-2-methylphenol	810 U	820 U	840 U	820 U	810 U	840 U	840 U	830 U	840 U
N-Nitrosodipheylamine (1)	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
4-Bromophenyl-phenylether	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Hexachlorobenzene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Pentachlorophenol	810 U	820 U	840 U	820 U	810 U	840 U	840 U	830 U	840 U
Phenanthrene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Anthracene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Carbazole	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Di-n-Butylphthalate	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Fluoranthene	330 U	340 U	350 U	340 U	330 U	100 J	350 U	340 U	350 U
Pyrene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Butylbenzylphthalate	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
3,3'-Dichlorobenzidine	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Benzo(a)Anthracene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
bis(2-Ethylhexyl)Phthalate	330 U	340 U	350 U	340 U	330 U	36 J	350 U	340 U	350 U
Chrysene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Di-n-Octyl Phthalate	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Benzo(b)Fluoranthene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Benzo(k)Fluoranthene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Benzo(a)Pyrene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Indeno(1,2,3-cd)Pyrene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Dibenz(a,h)Anthracene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U
Benzo(g,h,i)Perylene	330 U	340 U	350 U	340 U	330 U	340 U	350 U	340 U	350 U

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Table 2-1. White Bluffs Pickling Acid Crib ERA
Semi-volatile Organic Compounds Analysis and Qualifier Summary
(Sheet 6 of 6)

3.0 VOLATILE ORGANIC DATA VALIDATION AND LIMITATIONS

3.1 SUMMARY

3.1.1 Four Sample Delivery Groups

Sample results from four volatile organic cases are included in this report:

Laboratory	Case Number	No. of Samples	No. Fully Validated
TMA	A212018/023	8	7
TMA	A212011	1	1
Weston	9212L005	1	1
TMA	A2120149	1	1

Data qualifiers assigned to the sample data for these cases are summarized in Table 3-1.

3.1.2 All Samples Validated

Results for all the sample analyses for the cases listed above were validated, and data qualifiers assigned as appropriate. All of the reported results for QA samples associated with these cases were reviewed. For Case A212018/023, 100% of the QA sample results were recalculated and QC calculations verified. A limited number of samples, specified by WHC, were fully validated (i.e., all sample results were recalculated from the laboratory raw data).

3.1.3 WHC Validation Guidance Used

Data validation was performed in accordance with the WHC Data Validation Procedures for Chemical Analyses (WHC 1992a). Additional criteria established for the determination of laboratory performance were obtained from WHC (WHC 1992c), and the EPA's Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses (EPA 1990c).

3.1.4 Samples Analyzed According to CLP Protocols

Ten low level soil samples were submitted for analysis. Analyses were performed according to the 1990 CLP protocol (EPA 1990a).

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Sample analyses were performed using two GC/MS systems. One system was outfitted with a packed column, the other with a capillary column. The data were evaluated against equivalent QC requirements and are comparable. Failure to comply with various technical requirements established by CLP protocols resulted in qualification of the data. The specific problems observed during the QA review are detailed in the sections below.

The analysis was complete and met the method and work plan CRQL requirements (WHC 1992c) in all cases.

3.1.5 Minor Deficiencies Noted

There were minor deficiencies associated with the analyses which resulted in the qualification of data. These included minor blank contamination, an internal standard exceedence, and sample concentrations reported below the quantification limit. These deficiencies and the resulting data qualifications are explained in greater detail below.

3.2 ANALYTICAL METHOD

3.2.1 Gas Chromatography/Mass Spectrometer Tuning Criteria Met

Tuning is performed to ensure that mass resolution, identification, and, to some degree, sensitivity of the 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 the WHC data validation guidelines (WHC 1992a) and in the CLP SOW (EPA 1990a).

The original tuning data were checked for transcription and calculation errors in one of the packages (Case A212018/023). In the remaining data packages, tuning and mass calibration summary forms (Form V) were evaluated to verify that tuning criteria were met. Prior to calibration and sample analysis, all tuning criteria were met and no data were qualified based on the tuning results.

3.2.2 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 during analytical runs.

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

3.2.2.1 No Initial Calibration Exceedences. Instrument response is established when the RRFs for all target compounds are greater than or equal to the minimum criteria specified in the CLP SOW (EPA 1990a). Linearity is established when the RSDs of the RRFs are less than or equal to 20.5% (EPA 1990a). Only some of the compounds are required to meet these criteria, and minor exceedences of common problem compounds do not cause qualification of the data.

For each of the cases, the initial calibrations met the criteria. Therefore, no data were qualified based on the initial calibration results.

3.2.2.2 No Continuing Calibration Exceedences. The criteria for accepting the continuing calibration require that a 50 µg/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 the minimum criteria specified in the CLP SOW (EPA 1990a). In addition, the %D of these RRFs must be less than or equal to 25% (EPA 1990a) of the average RRFs calculated for the associated initial calibration. Only some of the compounds are required to meet these criteria, and minor exceedences of common problem compounds do not cause qualification of the data.

The required analysis frequency and criteria for continuing calibration were met for these cases and no data were qualified based on the continuing calibration results.

3.2.3 Blanks

Method blank and field blank analyses are performed to determine the extent of laboratory or field contamination of samples. If the sample concentration for a compound is less than five times the blank concentration (ten times if the compound is a common laboratory contaminant), the sample concentration is qualified as undetected (U).

3.2.3.1 Minor Method Blank Contamination. One method blank was analyzed during each 12-hour period on each instrument. Several compounds were detected in the blanks at low concentrations. These included acetone, 1,1,1-trichloroethane, and methylene chloride. Based on the 5 times (1,1,1-trichloroethane) and 10 times (acetone and methylene chloride) criteria, all associated sample data were qualified as undetected (U), as outlined below.

Case A212018/023. Acetone was detected in the blank associated with this case. Acetone data in samples B07PZ1, B07PZ2, B07PZ4, B07PZ6, B07PZ7, B07Q01, B07Q02, and B07Q03 were qualified as undetected (U) at the CRQL.

Case A212011. Methylene chloride and acetone were detected in the blank associated with this case. Acetone and methylene chloride data in sample B07PY8 were qualified as undetected (U) at the CRQL, and at the concentration reported, respectively.

Case 9212L005. Acetone was detected in the blank associated with this case. The acetone data for sample B07Q13 was qualified as undetected (U) at the CRQL.

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Case A212049. Acetone was detected in the blank associated with this case. The acetone data for samples B07Q12 was qualified as undetected (U) at the CRQL.

3.3 HOLDING TIMES

Analytical holding times were assessed to ascertain whether the Westinhouse Hanford holding time requirements for volatile organic analyses were met by the laboratory. The WHC 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.

The holding times were acceptable for all of the samples associated with these four cases. Therefore, no qualifiers were assigned based on the holding time.

3.4 ACCURACY

The overall accuracy goal for the target compounds is ± 25%. Accuracy was assessed by evaluating the recoveries of stable isotopically labeled surrogate compounds added to all samples and blanks, matrix spikes, and by the analysis of a representative sample, which was spiked with a variety of volatile organic compounds.

3.4.1 Surrogate Compound Recovery Acceptable

Matrix-specific surrogate compound recovery control windows have been established by the EPA CLP program (EPA 1990a). When a surrogate compound recovery 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 EPA surrogate recovery control windows (EPA 1990) are calculated using the following formula:

$$EPA \text{ Control Limit} = \overline{\%R} \pm (\tau * Std. Dev.)$$

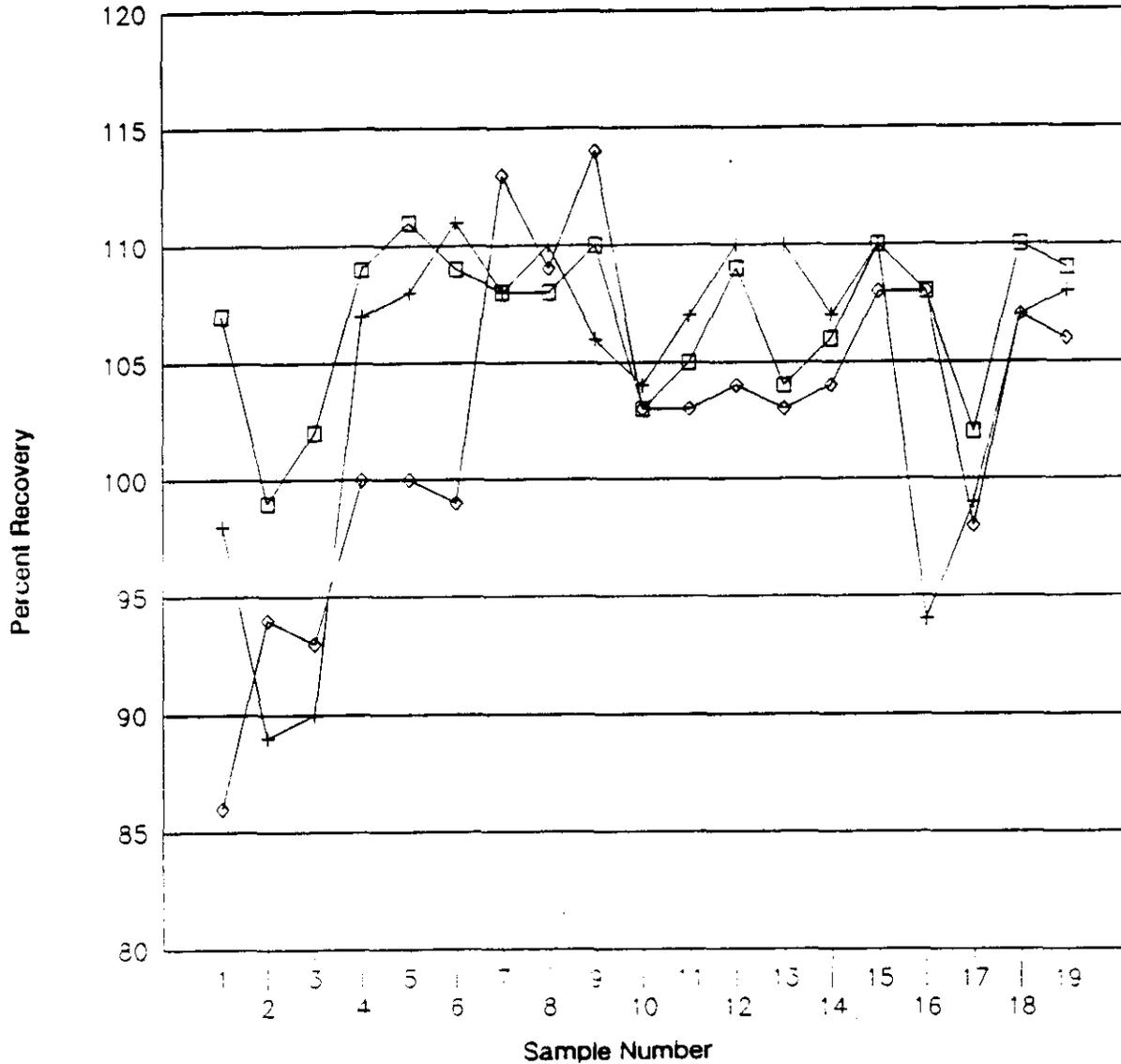
Where:

τ = Student τ Value at the 99 Percent Confidence Level

The surrogate recovery distribution was calculated in the same manner. Figure 3-1 shows that the White Bluffs Pickling Acid Crib ERA surrogate recovery distribution is comparable to the EPA general population data for this method. The percent recovery distribution is normal and there are no trends.

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Figure 3-1. White Bluffs Pickling Acid Crib ERA
Volatile Surrogate Recovery



	□ Toluene-d8	+ Bromofluorobenze	◇ 1,2-Dichloroethane-d4
Mean:	106.8	104.4	102.7
Variance:	10.8	45.3	45.4
Unbiased Std. Dev.:	3.4	6.9	6.9
2.998*Std. Dev.:	8.5	17.5	17.5
Range (Mean +/- (2.998*Std. Dev.)):	98-115	87-122	85-120
EPA Limits:	23-120	30-115	18-137

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The surrogate compound recoveries calculated for the three stable isotopically labeled surrogate compounds were acceptable. Therefore, no qualifiers were assigned based on surrogate recovery.

3.4.2 Matrix Spike Recoveries Acceptable

Matrix spike compounds are added to a sample that is representative of the sample delivery group. Matrix spike analyses are performed in duplicate using five compounds specified by CLP protocols. The recoveries for the five compounds must be within the sample matrix established QC limits (EPA 1990a). The matrix spike analyses estimate the interference with target compounds either positively or negatively.

Four matrix spike samples and four matrix spike duplicate samples were analyzed with the cases addressed in this report. The matrix spike compound recoveries were acceptable. Therefore, no qualifiers were assigned based on matrix spike percent recoveries.

The EPA matrix spike recovery control windows (EPA 1990a) are calculated using the following formula:

$$\text{EPA Control Limit} = \overline{\%R} \pm (\tau * \text{Std. Dev.})$$

Where:

$$\tau = \text{Student } \tau \text{ Value at the 99 Percent Confidence Level}$$

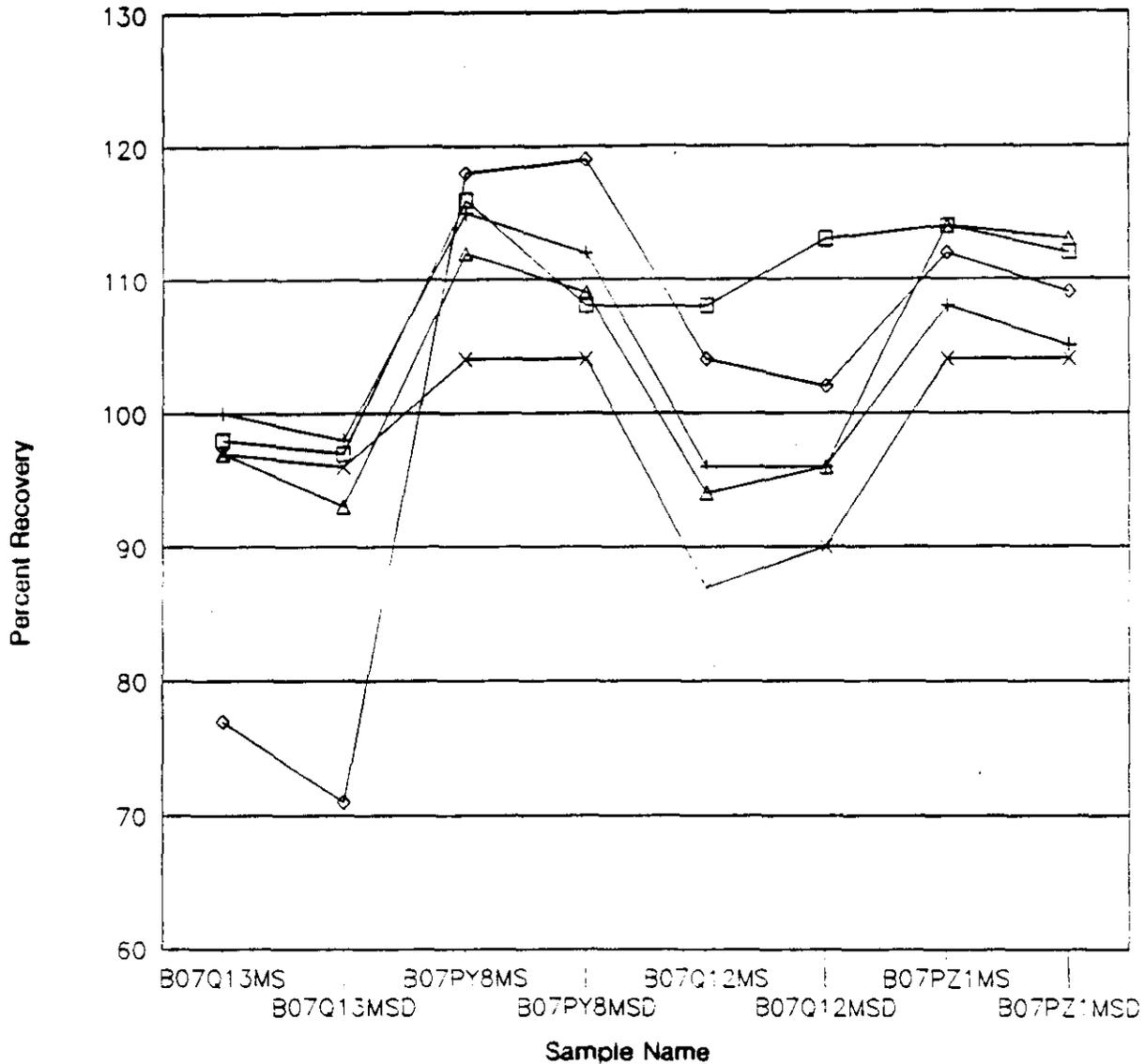
Although the White Bluffs Pickling Acid Crib ERA matrix spike population is small (eight matrix spike samples), the matrix spike recovery distribution was calculated in the same manner. Figure 3-2 shows that the matrix spike recovery distribution is comparable to the EPA general population data for this method.

3.5 PRECISION ACCEPTABLE

The overall precision goal for the target compounds is $\pm 35\%$. Analytical precision is expressed by the RPD between the recoveries of duplicate matrix spike analyses performed on a sample. When the laboratory has not performed MS/MSD analyses, precision may also be assessed using unspiked duplicate sample analyses. Field precision is measured by analyzing duplicate samples taken in the field. Interlaboratory precision is measured by analyzing duplicate samples ("field splits") by two analytical laboratories.

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Figure 3-2. White Bluffs Pickling Acid Crib ERA Matrix Spike Recovery



	□ 1,1-Dichloroethene	+ Trichloroethene	◇ Benzene	Δ Toluene	X Chlorobenzene
Mean:	108.3	103.8	101.5	103.5	98.3
Variance:	45.2	47.7	285.3	75.3	41.7
Unbiased Std. Dev.:	7.2	7.4	18.1	9.3	6.9
2.998*Std. Dev.:	21.5	22.1	54.1	27.8	20.7
Range (Mean +/- (2.998*Std. Dev.)):	87-130	82-126	47-156	76-131	78-119
EPA Limits:	59-172	62-137	66-142	59-139	60-133

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concentrations, the source of the methylene chloride and toluene cannot be confirmed to be from the site.

There were no TICs detected in these samples.

3.8 COMPOUND QUANTIFICATION AND REPORTED DETECTION LIMITS

Compound quantifications and reported detection limits were recalculated for the samples specified by WHC for each case to verify that they were accurate and consistent with CLP requirements. The calculations were consistent with the reported results. Therefore, no changes or qualifications were made based on the calculations.

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

3.9 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 QA review.

3.10 CHANGES MADE SINCE PRELIMINARY REPORT

Data qualifier assignments and documentation were reviewed by a senior validator and a technical reviewer. No changes were made after the submittal of the Preliminary QA Report.

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Sample Number:	B07PY8	B07PZ1	B07PZ2	B07PZ4	B07PZ6	B07PZ7	B07Q01	B07Q02	B07Q03
Units:	µg/kg								
Chloromethane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Bromomethane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Vinyl Chloride	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Chloroethane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Methylene Chloride	11 U	11 U	10 U	11 U	10 U	10 U	2 J	3 J	2 J
Acetone	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Carbon Disulfide	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
1,1-Dichloroethene	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
1,1-Dichloroethane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
1,2-Dichloroethene (total)	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Chloroform	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
1,2-Dichloroethane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
2-Butanone	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
1,1,1-Trichloroethane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Carbon Tetrachloride	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Bromodichloromethane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
1,2-Dichloropropane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
cis-1,3-Dichloropropene	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Trichloroethene	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Dibromochloromethane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
1,1,2-Trichloroethane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Benzene	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
trans-1,3-Dichloropropene	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Bromoform	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
4-Methyl-2-Pentanone	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
2-Hexanone	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Tetrachloroethene	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
1,1,2,2-Tetrachloroethane	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Toluene	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Chlorobenzene	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Ethylbenzene	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Styrene	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U
Xylene (total)	11 U	11 U	10 U	11 U	10 U	10 U	11 U	11 U	10 U

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Table 3-1. White Bluffs Pickling Acid Crib ERA
 Volatile Organic Compounds Analysis and Qualifier Summary
 (Sheet 1 of 2)

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4.0 METALS DATA VALIDATION AND LIMITATIONS

4.1 SUMMARY

4.1.1 Three Sample Delivery Groups

Sample results from three metals cases are included in this report:

Laboratory	Case Number	No. of Samples	No. Fully Validated
TMA/Skinner	N212014	16	15
TMA/Skinner	N212069	12	12
Weston	9212L005	1	1

Data qualifiers assigned to the analytes reported for these cases are summarized in Table 4-1.

4.1.2 All Samples Validated

Results for all the sample analyses for the cases listed above were validated, and data qualifiers assigned as appropriate. All of the reported results for QA samples associated with these cases were reviewed. For all of the cases, 100% of the QA sample results were recalculated, and QC calculations verified. A limited number of samples, specified by WHC, were fully validated (i.e., all sample results were recalculated from the laboratory raw data).

4.1.3 WHC Validation Guidance Used

Data validation was performed in accordance with the WHC Data Validation Procedures for Chemical Analyses (WHC 1992a). Additional criteria established for the determination of laboratory performance were obtained from WHC (WHC 1992c).

4.1.4 Samples Analyzed According to CLP Protocols

Twenty-eight low level soil samples were submitted for analysis for inorganic target analyte list (TAL) metals and zirconium. Analyses were performed according to the 1990 CLP protocol (EPA 1990b).

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). Failure to comply with various technical requirements established by CLP protocols resulted in qualification of some of the data.

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4.1.5 Most Data Quality Objectives Met

The analyses were complete and met the method and work plan contract required detection limit (CRDL) requirements (WHC 1992c), except as noted below.

4.1.6 No Data Rejected

No sample data were rejected due to deficiencies in data quality.

4.1.7 Minor Deficiencies in Other Qualified Data

There were minor deficiencies associated with the analysis of these samples. Minor deficiencies included minor blank contamination, matrix spike percent recovery exceedences, analytical spike percent recovery exceedences, and ICP serial dilution %D exceedences.

These deficiencies and the resulting data qualifications are explained in greater detail in the following sections.

4.2 ANALYTICAL METHODS

Performance of specific instrument QA and QC procedures, including deficiencies noted during the QA review, are discussed below.

4.2.1 Instrument Calibration Acceptable

4.2.1.1 GFAA. Four 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.

4.2.1.2 CVAA. 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.

4.2.1.3 ICP. At least one standard and a blank were analyzed by ICP for all other elements, and the calibration was acceptable.

4.2.2 Calibration Verification Acceptable

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

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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 required control limits in all cases.

4.2.3 Low-Level Blank Contamination

Initial calibration blank, continuing calibration blank, and preparation or method blank results were reviewed to determine the extent of variability of the sample detection limit and the existence and magnitude of blank contamination.

Samples with digestate concentrations of less than five times the highest amount found in any of the associated blanks are qualified as non-detected (U). Samples with concentrations of greater than five times the highest amount found in any of the associated blanks do not require qualification.

The laboratory reports any negative values acquired during blank sample analyses that are greater than the IDL in magnitude. When this occurs, sample detection limits are qualified as estimates (UJ) because they potentially could be positive concentrations reported as non-detects by the laboratory. Any positive sample concentrations reported near the IDL (i.e., less than 5 times the absolute value of the blank) are also qualified as estimates (J). Any sample concentration greater than 5 times the absolute value of the blank is not qualified.

Several elements were detected in blanks associated with these cases. Qualifications made as a result of the blank contamination are outlined below.

Case N212014. Calcium in sample B07PZ0; copper in samples B07PZ8, B07PZ9, B07Q00, B07Q01, B07Q02, B07Q03, B07PZ7, B07PZ6, B07PZ5, B07PZ4, B07PY9, B07PZ0, B07PZ2, and B07PZ3; sodium in samples B07PZ7, B07PZ8, B07Q01, B07Q02, B07PY8, B07PZ0, B07PZ2, B07PZ3, and B07Q03; and zinc in sample B07PZ0 were qualified as undetected (U) due to blank contamination.

Negative blanks, as discussed on the preceding page, were reported for arsenic, calcium, chromium, cobalt, nickel, and sodium. These elements were not detected in the associated sample (B07PZ0). Therefore, the associated arsenic, calcium, chromium, cobalt, nickel, and sodium detection limits for sample B07PZ0 were qualified as estimates (UJ). Arsenic was detected in samples B07PY8, B07PY9, B07PZ1, B07PZ2, B07PZ3, B07PZ4, B07PZ5, B07PZ6, B07PZ7, B07PZ8, B07PZ9, B07Q00, B07Q01, B07Q02, and B07Q03 at concentrations near the detection limit. Therefore, the associated arsenic concentrations for these samples were qualified as estimates (J).

Case 9212L005. Nickel and potassium in sample B07Q13 were qualified as undetected (U) due to blank contamination. A negative blank was reported for sodium. Sodium was detected at a concentration near the detection limit. Therefore the sodium concentration for sample B07Q13 was qualified as an estimate (J).

Case N212069. Copper in samples B07Q08, B07Q14, and B07Q15 were qualified as undetected (U) due to blank contamination.

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4.2.4 Holding Times Met

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 laboratory. The holding time requirements are as follows: samples must be analyzed within 28 days for mercury; and within 6 months for all other metals.

Required holding times were met for the samples in each of these cases.

4.2.5 Instrument-Specific QC Procedures

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

The interference check samples analyzed with these cases were acceptable.

Serial Dilutions. 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 $\pm 10\%$.

A serial dilution was required for many of the ICP metals in these four cases. The dilution concentrations were found to be within 10% of the initial analysis except for the following:

Case N212069. The serial dilution criteria were exceeded for potassium (12.1%) in sample B07Q16L. Therefore, the potassium concentrations for samples B07Q04, B07Q05, B07Q06, B07Q07, B07Q08, B07Q09, B07Q10, B07Q11, B07Q12, B07Q14, B07Q15, and B07Q16 were qualified as estimates (J).

4.2.5.2 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.

Duplicate injection frequency and precision requirements were met.

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 exceed the control window of 85 to 115% recovery and the absorbance of the sample is greater than 50% of the analytical spike absorbance, the sample must be reanalyzed using the method of standard additions (MSA).

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Analytical spike percent recovery and MSA requirements were met with the following exceptions:

Case N212014. The arsenic analytical spike percent recovery for sample B07Q00 (119%) exceeded the control limits. Arsenic was detected in the sample. Therefore, the associated concentration was qualified as an estimate (J).

Selenium analytical spike percent recoveries in samples B07PZ4 (117%), B07PZ6 (129%), B07Q03 (79%), B07Q00 (84%), and B07PY8 (126%) exceeded the control limits. Selenium was not detected in samples B07PZ4, B07PZ6, and B07PY8. Because the percent recoveries were high for these samples, no qualifiers were assigned. Selenium was detected in samples B07Q03 and B07Q00. Therefore, the selenium concentrations for these samples were qualified as estimates (J).

One sample (B07PZ3) was analyzed for selenium using the MSA technique. The MSA correlation coefficient was <0.995 (0.992). Therefore, the associated selenium concentration was qualified as an estimate (J).

Case 9212L005. The selenium analytical spike recovery in sample B07Q13 (74 percent) exceeded the control limits. Selenium was not detected in the sample. Therefore, the sample detection limit was qualified as an estimate (UJ).

Case N212069. Selenium analytical spike percent recoveries in samples B07Q06 (81%), B07Q07 (81%), B07Q11 (79%), B07Q05 (82%), B07Q09 (40%), and B07Q16 (83%) exceeded the control limits. Selenium was detected in sample B07Q09, and the associated concentration was qualified as an estimate (J). Selenium was not detected in the remaining samples, and the associated detection limits were qualified as estimates (UJ).

4.3 ACCURACY

The overall accuracy goal for the metals of concern is $\pm 25\%$. Although there were exceedences for other metals, only one of the metals of concern (antimony) did not meet this goal.

4.3.1 Matrix Spike Exceedences for Antimony, Manganese, Selenium, and Mercury

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 to 125%.

The matrix spike results were acceptable with the following exceptions:

Case N212014. In sample B07Q00, the matrix spike percent recoveries were outside the control limits for antimony (69%), manganese (71%), and selenium (63%). Antimony was not detected in the associated samples (B07PY8, B07PY9, B07PZ0, B07PZ1, B07PZ2, B07PZ3, B07PZ4, B07PZ5, B07PZ6, B07PZ7, B07PZ8, B07PZ9, B07Q00, B07Q01, B07Q02, and B07Q03). Therefore, the antimony detection limits were qualified as estimates (UJ). Manganese was detected in

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the associated samples (listed above), and the sample concentrations were qualified as estimates (J). Selenium data for the samples listed above were qualified as estimates (J or UJ).

Case 9212L005. In sample B07Q13, the matrix spike percent recovery was outside the control limit for mercury (129%). As mercury was not detected in the associated sample and the matrix spike recovery was high, no qualifier was assigned.

Case N212069. In sample B07Q16, the matrix spike percent recovery was outside the control limit for antimony (56%). Antimony was detected in samples B07Q11 and B07Q16, and the associated antimony concentrations were qualified as estimates (J). Antimony was not detected in samples B07Q04, B07Q05, B07Q06, B07Q07, B07Q08, B07Q09, B07Q10, B07Q12, B07Q14, and B07Q16; the associated antimony detection limits were qualified as estimates (UJ).

4.3.2 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 and analyzed with every group of samples that have been prepared together. The performance criteria for soil LCS samples are specified by the certifying agency that provides them.

One LCS was digested and analyzed with each case. The results were compared against the control windows and were found to be acceptable. Therefore, no data were qualified based on the laboratory control samples analyses.

4.4 PRECISION

The overall precision goal for the metals of concern is $\leq 35\%$ RPD. 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. Field split analyses are used to measure interlaboratory precision.

4.4.1 Analytical Duplicates Acceptable

The CLP RPD goal for analytical duplicates in a soil matrix is less than or equal to 35% for concentrations greater than five times the CRDL and \pm two times the CRDL for concentrations less than five times the CRDL.

One set of analytical duplicates was analyzed for all analytes with each sample delivery group (SDG) for each matrix. The laboratory duplicate precision goals were met, and no qualifiers were assigned.

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4.4.2 Field Duplicates Acceptable

One set of field duplicate samples (B07Q02 and B07Q01) was analyzed. Field duplicate precision goals were met, and no qualifiers were assigned.

4.4.3 Interlaboratory Precision Data Acceptable

One set of field split samples (B07Q12 and B07Q13) was analyzed. With the exception of chromium (128% RPD), interlaboratory precision goals were met. No qualifiers were assigned based on the interlaboratory precision data.

4.5 SAMPLE RESULT VERIFICATION

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

Sample calculations were acceptable. No transcription errors or other anomalies were found.

4.6 CHANGES MADE SINCE PRELIMINARY REPORT

Data qualifier assignments and documentation were reviewed by a senior validator and a technical reviewer. No changes were made after the submittal of the Preliminary QA Report.

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Table 4-1. White Bluffs Pickling Acid Crib ERA
 Metals Analysis and Qualifier Summary
 (Sheet 1 of 3)

Sample Number:	B07PY8	B07PY9	B07PZ0	B07PZ1	B07PZ2	B07PZ3	B07PZ4	B07PZ5	B07PZ6
Units:	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Aluminum	5360	5650	33.9	5700	5010	5020	5550	6810	4310
Antimony	2.9 UJ	2.9 UJ	2.9 UJ	3 UJ	2.9 UJ	3.1 UJ	3.1 UJ	3.1 UJ	3 UJ
Arsenic	1.5 J	1.6 J	0.36 UJ	1.2 J	1.1 J	1 J	2.3 J	1.9 J	1.2 J
Barium	44.1	41.2	0.14 U	36.8	44.7	39.4	50.8	56.1	41
Beryllium	0.17	0.21	0.06 U	0.19	0.22	0.18	0.19	0.28	0.16
Cadmium	0.29 U	0.3 U	0.29 U	0.31 U	0.3 U	0.31 U	0.32 U	0.31 U	0.31 U
Calcium	2600	2810	4 UJ	2870	2800	3010	8010	4650	2850
Chromium	9.1	9.4	0.51 UJ	11.2	9.3	8	10	14	7.7
Cobalt	6.4	6	0.25 UJ	6.6	6.1	6.8	6.2	9.1	7.3
Copper	23.5	16.7 U	8.4 U	20.7	17.8 U	13.6 U	17.6 U	17.6 U	15.2 U
Iron	14600	14200	451	13500	12700	15300	13200	15900	12900
Lead	3.9	3.4	0.77	4.1	3.1	3.1	4	4.2	3.5
Magnesium	3310	3610	7.3	4080	3720	3460	4350	5130	2960
Manganese	138 J	142 J	0.23 J	175 J	156 J	149 J	213 J	226 J	144 J
Mercury	0.05 U	0.05 U	0.04 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U
Nickel	6.2	8.3	0.51 UJ	9.5	8.8	7.1	10.3	14.3	7.9
Potassium	820	862	15.5 U	763	824	784	794	1030	542
Selenium	0.59 UJ	0.54 UJ	0.75 J	0.62 J	0.6 UJ	1.1 J	0.61 UJ	0.59 UJ	0.57 UJ
Silver	0.7 U	0.89	0.7 U	0.74 U	0.96	0.98	0.76 U	1.2	0.86
Sodium	139 U	166	22.1 UJ	171	136 U	151 U	166	189	158
Thallium	0.27 U	0.24 U	0.25 U	0.27 U	0.27 U	0.26 U	0.27 U	0.26 U	0.25 U
Vanadium	41.4	37	0.49 U	34.1	30.1	41.6	32.4	36.7	39.2
Zinc	71.8	63.7	1.8 U	50.7	30.3	60.5	31.3	43	30.5
Zirconium	17.1 U	17.5 U	17.2 U	18 U	17.4 U	18.3 U	18.6 U	18.2 U	17.9 U

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Table 4-1. White Bluffs Pickling Acid Crib ERA
Metals Analysis and Qualifier Summary
(Sheet 2 of 3)

Sample Number:	B07PZ7	B07PZ8	B07PZ9	B07Q00	B07Q01	B07Q02	B07Q03	B07Q04	B07Q05	B07Q06
Units:	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Aluminum	4630	4640	7000	4140	5800	5730	4320	5930	4170	5730
Antimony	3 UJ	2.9 UJ	3.1 UJ	2.9 UJ	2.9 UJ	3.1 UJ	2.8 UJ	3.9 UJ	4.4 UJ	3.6 UJ
Arsenic	1.2 J	1.2 J	2 J	1.3 J	1.3 J	1.5 J	1 J	2	1.3	1.7
Barium	29.5	29.7	73.8	43.1	58.3	54	38.1	67	39.7	55.6
Beryllium	0.13	0.14	0.24	0.2	0.31	0.24	0.18	0.23	0.18	0.26
Cadmium	0.31 U	0.29 U	0.32 U	0.3 U	0.3 U	0.31 U	0.29 U	0.34 U	0.38 U	0.32 U
Calcium	2800	2590	22400	3530	6410	6330	5170	9130	4310	6750
Chromium	8.7	9.1	13.6	7.5	10.2	9.6	9.3	11	7.2	10
Cobalt	5.9	5.7	8.7	7.5	7.3	8.3	6.5	7.1	7.2	6.9
Copper	13.7 U	11 U	16.9 U	13.7 U	14.6 U	14.7 U	11.8 U	10.5	13.2	9.7
Iron	12300	11600	15600	14900	15000	15300	12600	16000	15900	17600
Lead	2.6	2.5	6.5	2.5	3.3	5.3	2.9	3.4	2.5	2.9
Magnesium	3570	3520	6500	3420	4620	4910	3560	4920	3470	4390
Manganese	177 J	149 J	265 J	183 J	190 J	200 J	178 J	212	218	240
Mercury	0.05 U	0.05 U	0.06 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U
Nickel	8	8.7	13.3	8.8	10.8	11.8	8.8	10.7	9.6	9.8
Potassium	555	630	1140	504	1010	1010	551	1230 J	546 J	1260 J
Selenium	0.54 UJ	0.64 J	0.95 J	0.67 J	0.63 UJ	0.6 UJ	0.68 J	0.63 U	0.72 UJ	0.58 UJ
Silver	0.74 U	0.95	0.76 U	0.81	0.75	0.79	0.69 U	0.99	0.97	1.3
Sodium	149 U	124 U	194	173	142 U	145 U	129 U	154	200	362
Thallium	0.24 U	0.24 U	0.3 U	0.26 U	0.28 U	0.27 U	0.26 U	0.53 U	0.6 U	0.48 U
Vanadium	33.6	27.1	33.9	40.5	34.9	35.4	34.2	34.1	39.7	35.9
Zinc	28.8	28	40.9	30.6	35.6	38	28	38.2	33.6	35
Zirconium	18.1 U	17.3 U	18.6 U	17.4	17.5 U	18.4 U	16.9 U	18.7 U	20.8 U	17.3 U

Sample Number:	B07Q07	B07Q08	B07Q09	B07Q10	B07Q11	B07Q12	B07Q13	B07Q14	B07Q15	B07Q16
Units:	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Aluminum	6010	4070	5720	5730	8060	7370	5370	6090	6090	7220
Antimony	4.4 UJ	3.7 UJ	3.7 UJ	4 UJ	4.1 J	3.6 UJ	12.2 U	3.5 UJ	4.3 UJ	4.8 J
Arsenic	1.7	1.6	1.2	0.87	1	3.6	2.4	0.87	0.9 U	1.2
Barium	58.1	46.8	75.1	50.8	64.3	57.9	52.3	72.8	68.2	79.6
Beryllium	0.29	0.19	0.31	0.19	0.36	0.3	0.29	0.31	0.26	0.37
Cadmium	0.39 U	0.9	0.32 U	0.35 U	0.35 U	0.31 U	1.43 U	0.31 U	0.37 U	0.35 U
Calcium	5220	4230	3900	3400	4940	3460	3250	3420	3390	3760
Chromium	9.9	6.5	7.9	10.2	13.3	43.1	9.5	8.5	8.8	9.8
Cobalt	7.6	5.9	10.9	6.7	10	9.3	8.4	9.7	8.4	11
Copper	10.4	6.6 U	10.7	18.7	14.2	11.4	13.2	9.3 U	9.1 U	10.1
Iron	19100	12900	20800	16300	23400	19200	14600	20500	17900	23300
Lead	3.6	4.3	3.4	6.7	5.1	3.9	3.6	3.5	3.1	3.5
Magnesium	4410	3220	4320	3740	5210	4040	3670	3850	3680	4180
Manganese	257	196	376	190	263	177	143	347	317	372
Mercury	0.06 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	0.06 U	0.05 U
Nickel	10.6	7.4	11.3	9.2	12.5	27.8	13.6 U	8.7	8.9	9.9
Potassium	1140 J	866 J	1020 J	1430 J	1980 J	1710 J	1410 U	1490 J	1710 J	1620 J
Selenium	0.74 UJ	0.61 U	0.65 J	0.68 U	0.7 UJ	0.67 U	0.41 UJ	0.6 U	0.71 U	0.67 UJ
Silver	1.8	0.81	1.3	1	1.5	0.93	2.04 U	1.4	1.3	2.1
Sodium	543	750	178	136	493	165	165 J	131	140	176
Thallium	0.62 U	0.51 U	0.52 U	0.57 U	0.59 U	0.56 U	0.41 U	0.51 U	0.6 U	0.56 U
Vanadium	40.9	30.1	52.6	39.8	55.9	51.5	36	48.5	42.7	58.8
Zinc	1020	1070	46.6	68.7	554	50.5	40.4	46.6	43.3	49.4
Zirconium	25.9	17.9 U	17.7 U	19.2 U	19.4 U	17.2 U	40.8 U	20.9	20.4 U	30.7

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 Table 4-1. White Bluffs Pickling Acid Crib ERA
 Metals Analysis and Qualifier Summary
 (Sheet 3 of 3)

5.0 ANIONS AND NITRATE/NITRITE DATA VALIDATION AND LIMITATIONS

5.1 SUMMARY

5.1.1 Four Anion and Three Nitrate/Nitrite Sample Delivery Groups

Sample results from four anion cases are included in this report:

Laboratory	Case Number	No. of Samples	No. Fully Validated
TMA	A212018/023	14	13
TMA	A212011	2	2
Weston	9212L005	1	1
TMA	A212049	12	12

Sample results from three nitrate/nitrate cases are included in this report:

Laboratory	Case Number	No. of Samples	No. Fully Validated
TMA/Skinner	N212014	16	15
Weston	9212L005	1	1
TMA/Skinner	N212069	12	12

Data qualifiers assigned to anion and nitrate/nitrite data are summarized in Table 5-1.

5.1.2 All Samples Validated

Results for all the sample analyses for the cases listed above were validated and data qualifiers assigned as appropriate. All of the reported results for QA samples associated with these cases were reviewed. For all of the cases, 100% of the QA sample results were recalculated, and QC calculations verified. A limited number of samples, specified by WHC, were fully validated (i.e., all sample results were recalculated from the raw laboratory data).

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5.1.3 WHC Validation Guidance Used

Data validation was performed in accordance with the WHC Data Validation Procedures for Chemical Analyses (WHC 1992a). Additional criteria established for the determination of laboratory performance were obtained from WHC (WHC 1992c).

5.1.4 Samples Analyzed According to Non-CLP Protocols

A total of 28 soil samples were submitted for analysis for general chemistry analytes (chloride, fluoride, phosphate, sulfate, and nitrate/nitrite). Samples were analyzed by EPA methods for wastewater analysis (EPA 1983) modified for soil (leachate) analysis.

5.1.5 Majority of Data Quality Objectives Met

The analyses were complete and CRDL requirements were met. Many of the results were qualified as estimates due to QC exceedences. The overall data quality objectives were met, however, the interlaboratory precision goals were not met.

5.1.6 Minor Deficiencies in Other Qualified Data

There were minor deficiencies associated with the analysis of these samples. These included holding time exceedences and some lack of daily calibrations.

These deficiencies and the resulting data qualifications are explained in greater detail below.

5.2 ANALYTICAL METHODS

Performance of specific instrumental QA and QC procedures, including deficiencies noted during the QA review, are outlined below.

5.2.1 Instrument Calibration and Verification Criteria

5.2.1.1 Nitrate/Nitrite by Autoanalyzer. The autoanalyzer used for the analysis of nitrate/nitrite must be calibrated on each day of use using a minimum of three standards and a calibration blank. The correlation coefficient of a least-squares linear regression must be equal to or greater than 0.995.

Two different methods were used for the determination of nitrate/nitrite. TMA analyzed the samples using Method 353.2 (automated cadmium reduction). Weston analyzed the samples using Method 353.1 (automated hydrazine reduction). The initial calibrations were acceptable for nitrate/nitrite.

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5.2.1.2 Anions by Ion Chromatography. The ion chromatograph used for the analysis of phosphate must be calibrated on each day of use using a minimum of three standards and a calibration blank. The correlation coefficient of a least squares linear regression must be equal to or greater than 0.995.

The ion chromatograph initial calibrations were not acceptable for anions in Cases A212018/023, A212011, and A212049, as the calibration were performed 25 to 49 days prior to the day of analysis. All anion data associated with these cases were qualified as estimates (J or UJ).

5.2.1.3 Calibration Verifications Acceptable. The above calibrations are each immediately verified with an ICV standard analysis. The ICV standard is prepared from a source independent of the calibration standards, at a mid-calibration range concentration. The ICV percent recovery must fall within the control limits of 90 to 110%.

The calibrations are subsequently verified at regular intervals using a CCV standard. The control limits for percent recovery of CCV standards are the same as the ICV control limits.

All cases had acceptable calibration verification analyses.

5.2.2 Acceptable Blank Analyses

Blanks were analyzed for all of the analytes and were found to be acceptable, with no detectable contamination.

5.3 HOLDING TIMES EXCEEDED FOR ANIONS

Analytical holding times for the general chemistry analyses were assessed to ascertain whether the holding time requirements were met by the laboratory. Samples must be analyzed within 28 days for anions and nitrate/nitrite.

The following case had holding time exceedences.

Case A212011. Anion analyses exceeded the holding time by 1 day. Because this exceedence was minor, no qualifiers were assigned.

Case 9212L005. The nitrate/nitrite analysis exceeded the holding time by 9 days. The nitrate/nitrite concentration for sample B07Q13 was therefore qualified as an estimate (J).

Case A212049. Anion analyses exceeded the holding time by 18 to 21 days for all samples associated with this case (B07Q04, B07Q07, B07Q10, B07Q14, B07Q05, B07Q08, B07Q11, B07Q15, B07Q06, B07Q09, B07Q12, and B07Q16). Therefore the sample data (listed above) were qualified as estimates (J or UJ).

Case N212069. The leaching procedure for these nitrate/nitrite analyses exceeded the holding time by 2 to 5 days. In addition, the leachate was held unpreserved for 14 days until analysis. The EPA (1983) holding time for nitrate/nitrite in unpreserved samples is 48 hours. Because the matrix spike

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percent recoveries (which were spiked at the time of leaching) were acceptable, the data were not rejected. However, all nitrate/nitrite data associated with this case were qualified as estimates (J or UJ).

WHC data validation procedures specify holding times for phosphate analyses (based on those for the analysis of orthophosphate as phosphate) that are more restrictive than those established by EPA protocols. WHC procedures require that samples be analyzed within 2 days of collection (WHC 1992a). Using WHC guidelines, the following results would be rejected; based upon the 28 day holding time, no qualifiers were assigned.

Case A212018/A212023, 9212L005, A212049, and A212011. Using WHC guidelines, data from all samples would be rejected based upon holding times, as these samples were analyzed after the 2 day holding time.

5.4 ACCURACY

The overall accuracy goals for the analytes of concern are $\pm 25\%$. Accuracy is evaluated through the analysis of spiked samples and standard reference materials.

5.4.1 Matrix Spike Exceedence for Nitrate/Nitrite

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 fall within the range of 75 to 125%.

Matrix spike analyses for these cases met the percent recovery criteria.

5.4.2 Laboratory Control Sample

The LCS monitors the overall performance of the analysis, including the sample preparation. An LCS should be prepared (e.g., digested) and analyzed with every group of samples that have been prepared together. The performance criteria for aqueous LCS percent recovery is 80 to 120%.

Because there are no available soil LCS samples aqueous LCS samples are analyzed with the leachates. Aqueous LCS (blank spikes) analyzed for the analytes in these cases were found to be acceptable.

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5.5 PRECISION

The overall precision goals for the analytes of concern are $\leq 35\%$ RPD.

5.5.1 Acceptable Analytical Duplicates

Analytical (laboratory) duplicate sample analyses are used to measure laboratory precision and sample homogeneity. Analytical duplicate RPD values must be less than or equal to 35%.

The analytical duplicates met the RPD criteria. Therefore, no qualifiers were assigned based on the duplicate data.

5.5.2 Field Duplicates Acceptable

Field duplicate analyses are used to measure precision of both the laboratory and the field sampling procedure. One set of field duplicate samples was analyzed (B07Q01 and B07Q02). This set was analyzed for nitrate/nitrite only. Nitrate/nitrite was not detected in these samples. Therefore, field duplicate precision could not be quantitatively evaluated. No qualifiers were assigned based on field duplicate precision.

5.5.3 Field Split Samples Do Not Meet Objectives

Field split analyses are used to measure interlaboratory precision. One set of field split samples was analyzed (B07Q12 and B07Q13). The interlaboratory precision goals were not met for chloride (81% RPD), fluoride (78% RPD), and phosphate (126% RPD). No qualifiers were assigned based on the interlaboratory precision data, but this needs an additional evaluation.

5.6 SAMPLE RESULT VERIFICATION

Sample results and reported detection limits were recalculated to ensure that the reported results were accurate. Raw data were examined for anomalies, transcription errors, and reduction errors. In addition, the reviewer verified that the results fell within the linear range of the instrument. There were no discrepancies found. The data are acceptable for use as qualified.

5.7 CHANGES MADE SINCE PRELIMINARY REPORT

Data qualifier assignments and documentation were reviewed by a senior validator and a technical reviewer. The following changes were made after the submittal of the Preliminary QA Report.

- Previously several samples in case N212069 were qualified as rejected due to holding time exceedences. Due to re-evaluation of the data, the qualifiers were changed to undetected at estimated detection limits (UJ).

Sample Number:	B07PY8	B07PY9	B07PZ0	B07PZ1	B07PZ2	B07PZ3	B07PZ4	B07PZ5	B07PZ6	B07PZ7
Units:	mg/kg									
Chloride	1.8 J	2.3 J	3 J	1.4 J	2.1 J	1.8 J	2.1 J	2.2 J	2 J	1.8 J
Fluoride	0.3 J	0.4 J	0.2 J	0.6 J	1.1 J	0.4 J	0.8 J	0.5 J	0.4 J	0.3 J
Phosphate	0.8 UJ	0.8 UJ	0.8 UJ	1 J	1 J	1 J	1 J	2 J	0.8 UJ	1 J
Sulfate	25 J	15 J	3 J	13 J	11 J	10 J	11 J	6 J	8 J	6 J
Nitrate/Nitrite	7.41	3.83	2.43 U	3.89	2.42 U	2.52	2.44 U	2.43 U	2.53 U	2.48 U

Sample Number:	B07Q08	B07Q09	B07Q10	B07Q11	B07Q12	B07Q13	B07Q14	B07Q15	B07Q16
Units:	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Chloride	7.8 J	2.3 J	5.1 J	3.4 J	11.5 J	27	2.3 J	3 J	3 J
Fluoride	1.9 J	1.4 J	0.7 J	1 J	1.4 J	3.2	0.6 J	0.3 J	0.7 J
Phosphate	2 J	1 J	2 J	2 J	1 J	4.4	2 J	2 J	2 J
Sulfate	44 J	4 J	95 J	42 J	23 J	23.2	4 J	54 J	4 J
Nitrate/Nitrite	2.42 UJ	2.5 UJ	16.3 J	3.7 J	3.52 J	2.7 J	3.24 J	5.81 J	2.51 UJ

Sample Number:	B07PZ8	B07PZ9	B07Q00	B07Q01	B07Q02	B07Q03	B07Q04	B07Q05	B07Q06	B07Q07
Units:	mg/kg	mg/kg	mg/kg	mg/kg						
Chloride	2.2 J	2.2 J	1.8 J	2 J	2.2 J	2.1 J	2.3 J	2.1 J	12 J	181 J
Fluoride	0.3 J	0.7 J	0.3 J	1 J	1.1 J	0.3 J	1 J	0.5 J	1.5 J	2.5 J
Phosphate	1 J	0.8 UJ	1 J	1 J	1 J	1 J	1 J	0.8 UJ	0.8 UJ	0.8 UJ
Sulfate	5 J	10 J	6 J	10 J	10 J	6 J	6 J	5 J	292 J	329 J
Nitrate/Nitrite	2.59 U	2.46 U	2.46 U	2.54 U	2.46 U	2.57 U	2.55 UJ	2.52 UJ	2.47 UJ	2.51 UJ

Table 5-1. White Bluffs Pickling Acid Crib ERA
Anions and Nitrite/Nitrite Analysis and Qualifier Summary

6.0 GAMMA SPECTROSCOPY DATA VALIDATION AND LIMITATIONS

6.1 SUMMARY

6.1.1 Two Sample Analytical Cases

Sample results for gamma spectroscopy analyses for the following radiochemistry analytical cases are included in this report:

Laboratory	Case No.	No. of Samples	No. Fully Validated
TMA	N2-12-018-7133	10	2
Weston/Ecotek	9212L005	1	1

Data qualifiers assigned to the gamma spectroscopy results for targeted radionuclides for these radiochemical cases are summarized in Table 6-1.

6.1.2 Identified Samples Validated

Analytical data for cobalt-60, cesium-137, radium-226, and thorium-228 for all of the samples in the cases listed above were reviewed, and data qualifiers assigned as appropriate. One-hundred percent of the QA sample results were recalculated and QC calculations were verified for all sample delivery groups. Results for a limited number of samples, specified by WHC, were fully validated (e.g., all sample results for the radionuclides listed above were recalculated from the laboratory raw data).

6.1.3 WHC Guidance Used

Data validation was performed in accordance with the WHC Data Validation Procedure for Radiological Analyses (WHC 1992b). Additional criteria established for the determination of laboratory performance were obtained from WHC (WHC 1992c) and professional judgement.

6.1.4 Data Quality Objectives Generally Met

In general, data quality objectives were met for gamma spectroscopy. The TMA samples were acceptable without qualifiers. The Weston/Ecotek sample was qualified due to lack of an acceptable LCS.

6.2 ANALYTICAL METHOD

Instrument calibration is performed to establish that the gamma spectroscopy system used is capable of producing acceptable and reliable analytical data. The initial calibration is performed according to the manufacturer's recommendations and consists of determining the instrument detection efficiency for each gamma energy, system resolution, and the full-width at half maximum for each peak. Initial calibration is performed for

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each counting geometry used during analysis of WHC samples. Continuing calibration checks are performed to verify that instrument performance is stable and reproducible on a day to day basis. No data were qualified as a result of instrument calibration deficiencies.

In the TMA case, efficiency curves for the initial calibration were not included but were referenced on the certificates of calibration. This referenced information was considered acceptable.

In the Weston/Ecotek case, the Certificate of Calibration for the standard used did not include an expiration date as required in the WHC validation procedures (WHC 1992b). Because the standard date was within one and a half years of the sample analysis date and the half lives of the radionuclides of concern are relatively long compared to this date, the standard was considered acceptable.

6.3 ACCURACY

Accuracy was evaluated by analyzing samples spiked with known amounts of gamma-emitting radionuclides. The sample activity as determined by sample analysis is compared to the known activity to assess method accuracy. The analytical result must be within 80 to 120% of the true value to be acceptable. Data for samples with spiked sample results outside this range are qualified as estimates (J or UJ). Accuracy was acceptable for all TMA gamma spectroscopy results.

The efficiency checks performed on the gamma spectroscopy instruments by Weston/Ecotek also served for the LCS. Because this efficiency check is used for continuing calibration, it cannot be used as the LCS. Based on this deficiency, the Weston/Ecotek data were qualified as estimates (J).

6.4 PRECISION

Analytical precision is expressed by the 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 samples, provided that the analyte activity is greater than the minimum detectable amount (MDA). If the analyte activity is less than the MDA for either the original or duplicate sample, precision cannot be determined. The control limit defining acceptable method precision is an RPD <35% for replicates with activity levels five times the MDA or greater. If either replicate sample is less than five times the MDA, the difference between the two replicate values must be less than 2 times the MDA. Precision was acceptable for both the TMA and the Weston/Ecotek cases.

6.5 BLANKS

Blank samples are used to determine the presence of contaminants in the analytical system. Blank samples that indicate the presence of contaminants result in an estimated (J) qualification for any associated sample results that are greater than the MDA but less than 10 times the contaminant level. The blanks for both TMA and Weston/Ecotek were acceptable. For Weston/Ecotek,

the background was evaluated in lieu of a blank sample. The background values and sample errors were considered acceptable when compared to the sample results.

6.6 COMPOUND QUANTIFICATION AND REPORTED DETECTION LIMITS

Compound quantifications and detection limits were recalculated for all validated samples in each analytical case to verify that they were accurate and were consistent with 100-IU-5 requirements. Results below the MDA were qualified as nondetects (U) except in cases where the MDA was greater than the CRDL. Compound quantification and reported detection limits were acceptable for both TMA and Weston/Ecotek.

In the TMA case, the iron-59 MDA was greater than the CRDL. Because no iron-59 was detected and all TMA gamma scan results were very low, this was considered acceptable.

6.7 SYSTEM PERFORMANCE ACCEPTABLE

A review of TMA's and Weston/Ecotek's instrument continuing calibration information and QC data indicates that instrument performance was adequate for these analyses.

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Table 6-1. White Bluffs Pickling Acid Crib ERA
Radiochemical Analysis and Qualifier Summary

Customer ID No.	Cobalt-60		Cesium-137		Radium-226		Thorium-228	
	Reported Results (pCi/L)	Qualifier						
TMA N2-12-018-7133								
B07PY8	< 0.05	U	< 0.04	U	0.47		0.71	
B07PZ1	< 0.10	U	< 0.10	U	0.45		0.69	
*B07PZ2	< 0.03	U	< 0.03	U	0.49		0.73	
B07PZ4	< 0.04	U	< 0.03	U	0.51		0.99	
B07PZ6	< 0.05	U	< 0.04	U	0.48		0.83	
B07PZ7	< 0.05	U	< 0.04	U	0.42		0.63	
B07Q01	< 0.04	U	< 0.04	U	0.57		0.93	
*B07Q02	< 0.06	U	< 0.05	U	0.56		0.81	
B07Q03	< 0.05	U	< 0.04	U	0.50		0.70	
B07Q12	< 0.05	U	< 0.05	U	0.56		1.00	
Weston 9212L005								
*B07Q13	< 0.00591	UJ	0.0156	J	0.497	J	N/A	

* Fully validated sample

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7.0 REFERENCES

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