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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 10 HANFORD PROJECT OFFICE  
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RICHLAND, WASHINGTON 99352

September 7, 1993

Paul M. Pak  
U.S. Department of Energy  
P.O. Box 550, MS A5-19  
Richland, Washington 99352

Re: 1) White Bluffs Pickling Acid Crib Expedited Response  
Action Proposal (DOE/RL-93-48, Revision 0) - Review and  
Recommendations  
2) White Bluffs Pickling Acid Crib Expedited Response  
Action - Split Sampling Data Comparison

Dear Mr. Pak:

The U.S. Environmental Protection Agency (EPA) and the Washington State Department of Ecology (Ecology) have completed the review of the White Bluffs Pickling Acid Crib (100-IU-5) Expedited Response Action Proposal (DOE/RL-93-48, Revision 0). We agree with the Department of Energy (DOE) assessment of requiring no further action under the removal authority.

In order to continue with the evaluation of 100-IU-5 for a final Record of Decision (ROD) for the operable unit, the DOE must comply with the requirement specified in 40 CFR 430. Specifically, a Remedial Investigation/ Feasibility Study report, including a risk assessment, and a Proposed Plan must be submitted to the regulatory agencies for approval.

We have included the White Bluffs Pickling Acid Crib Split Sampling Data Comparison prepared for EPA by PRC Environmental Management, Inc. (PRC). The split sampling was done as part of the characterization effort for the expedited response action (ERA) for 100-IU-5, the White Bluffs Pickling Acid Crib. In general, the data sets from sampling done by EPA and DOE were comparable.



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Paul M. Pak

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September 7, 1993

If you have any questions or concerns regarding our recommendations or concerning the data comparison report, please contact me at (509) 376-4919.

Sincerely,

  
Pamela S. Innis  
Unit Manager

Enclosure

cc w/Enc: R. Stanley/D.R. Jansen, Ecology  
D.C. Teel, Ecology  
B.A. Austin, WHC  
W.L. Johnson/J.M. Frain, WHC  
B. Drost, USGS  
Administrative Record, Pickling Acid Crib ERA

cc w/o Enc: A. DeAngeles, PRC

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**DATA COMPARISON  
WHITE BLUFFS PICKLING ACID CRIBS ERA**

**HANFORD SITE  
RICHLAND, WASHINGTON**

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## 1.0 INTRODUCTION

PRC Environmental Management, Inc. (PRC) has compared data collected for the White Bluffs Pickling Acid Cribs Expedited Response Action (ERA) for the U.S. Environmental Protection Agency (EPA). This report focuses on the technical accuracy and completeness of Westinghouse Hanford Company's data validation results for soil characterization sampling, which was performed in the fall of 1992 (WHC 1993). The EPA soil samples were extracted and analyzed using the same methods as those used for the WHC soil samples. The WHC data are compared to data collected by PRC on December 1 and 2, 1992, and validated by EPA (EPA 1993a; EPA 1993b; EPA 1993c; and EPA 1993d).

## 2.0 ANALYTICAL DATA

### 2.1 DATA OBTAINED BY WESTINGHOUSE HANFORD

Data from the chemical analysis of 29 samples and their quality assurance samples were reviewed and validated to verify that reported sample results were of sufficient quality to support decisions regarding remedial actions performed at the White Bluffs Pickling Acid Crib Project (WHC 1993). Data for metals and anions were evaluated according to Level IV criteria (EPA 1987). Approximately one-fifth of the volatile organic analytes (VOAs) and semivolatile organic analytes (SVOAs) data were evaluated according to Level IV criteria. The remainder of the VOA and SVOA data were evaluated according to Level II criteria.

#### 2.1.1 Semivolatile Organic Compounds

Five of 29 samples were subjected to Level IV data validation. The remainder of the sample results were evaluated by cursory review (Level II data validation). No data were rejected as unusable in the data validation report. Several blanks contained tentatively identified compounds (TICs.) In addition, several of the matrix spike percent recoveries were outside quality control criteria.

The method detection limits for SVOAs ranged from 320 to 870 micrograms per kilogram ( $\mu\text{g}/\text{kg}$ ). According to the sampling analysis plan (SAP) the median detection limit was 340  $\mu\text{g}/\text{kg}$ . No compounds on the target analyte list (TAL) were detected above these limits.

#### 2.1.2 Volatile Organic Compounds

Three of 11 samples were subjected to Level IV data validation. The remainder of the sample results were evaluated by cursory review (Level II data validation). No samples were rejected as unusable in the data validation report. There were several minor quality assurance problems. Acetone, 1,1,1-trichloroethane, and methylene chloride were detected in blank samples. In addition, an internal standard exceeded quality control criteria, and sample concentrations were reported below the quantitation limit.

The reported detection limits for VOAs ranged from 3 to 11  $\mu\text{g}/\text{kg}$ . According to the SAP, the median detection limit was 10  $\mu\text{g}/\text{kg}$ . Other than those compounds identified as common laboratory contaminants, no VOAs were detected above these limits.

#### 2.1.3 Metals

Twenty-eight of the 29 samples were subjected to Level IV data validation. No samples were rejected as unusable because of deficiencies in data quality. There was minor blank contamination, as well as matrix spike percent recovery exceedances, analytical spike percent recovery exceedances, and minor problems associated with inductively coupled plasma (ICP) serial dilutions.

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Generally, matrix spike analyses quality control criteria were met for all metals except antimony, suggesting an acceptable level of accuracy. Analytical duplicate sample analysis suggested that laboratory procedure precision was good. Analysis of field duplicates (BO7QO1 and BO7QO2) showed that field sampling procedures were adequate to provide a representative sample. Results from field split analyses (BO7Q12 and BO7Q13) suggested that interlaboratory precision was adequate.

#### 2.1.4 Nonmetals

Twenty-eight of 29 samples were subjected to Level IV data validation. No data were rejected. However, holding times for all of the analyses were exceeded and daily instrument calibration was not completed. As a result, all analyses were qualified as estimated quantities (J or UJ).

Field split analyses showed that laboratory error had a significant effect on sample results. The interlaboratory precision goals were not met for chloride (81 relative percent difference [RPD]), fluoride (78 RPD), and phosphate (126 RPD).

### 2.2 DATA OBTAINED BY THE U.S. ENVIRONMENTAL PROTECTION AGENCY

#### 2.2.1 Semivolatile Organic Compounds

Nine samples were subjected to Level IV data review.

Trace amounts of several common laboratory contaminants including phenol, 2-fluorophenol, benzoic acid, dioctyl phthalate, and bis(2-ethylhexyl) phthalate were detected in the blank samples. Trace concentrations of 2-fluorophenol, in concentrations similar to those found in the blank samples, were detected in several samples. The method detection limits for SVOAs ranged from 80 to 10,900  $\mu\text{g}/\text{kg}$ . The median detection limit was 546  $\mu\text{g}/\text{kg}$ . Beside those analytes found in blank samples, no compounds on the target analyte list were detected.

#### 2.2.2 Volatile Organic Compounds

Eleven samples including two duplicates were subjected to Level IV data review.

Trace amounts of acetone, 2-butanone, 1,2,3-trichlorobenzene, toluene, chloromethane, trichlorofluoromethane, dichlorodifluoromethane, 2-butanone, acetone, and methylene chloride were detected in the blank samples. The method detection limits for sediment VOAs ranged from 1 to 19  $\mu\text{g}/\text{kg}$ , with a median concentration of 3  $\mu\text{g}/\text{kg}$ .

Several analytes associated with laboratory contaminants were detected in one or more sediment samples which were probably present as a result of laboratory contamination. Trichlorofluoromethane was detected in one sample (PC-B-S3-5), but not in the duplicate sample (PC-B-S2-5). Dichlorodifluoromethane was detected in sample PC-B-S2-5, but not in the duplicate sample PC-B-S3-5. All TAL analytes were assigned the data qualifier J because the analysis did not meet quality control criteria for continuing calibration. A J qualifier indicates that the analyte was positively identified, but the associated numerical result is an estimate.

#### 2.2.3 Metals

Eleven samples including duplicates were subjected to Level IV data validation. Selenium, manganese, and antimony data were rejected as unusable because of deficiencies in data quality.

Duplicate sample analysis suggested that laboratory procedure precision was good. Analysis of field duplicates (PC-B-S2-5/BO7QO1 and PC-B-S3-5/BO7QO2) showed that field sampling procedures were adequate to provide

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a representative sample. The RPD for the metals analysis ranged from 0 for arsenic to 199 for calcium, with an average of 13.1 all of metals, meeting the quality control criteria.

#### 2.2.4 Nonmetals

Nine samples were subjected to Level IV data validation. No data were rejected. However, holding times for nitrate, nitrite, and ammonia were exceeded. As a result, all nitrate, nitrite, and ammonia analyses were qualified as estimated quantities (J or UJ). Several pH analyses were completed outside of the 72-hour holding time and were qualified as estimated quantities (J).

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## 2.3 COMPARISON OF WHC AND EPA RESULTS

### 2.3.1 Semivolatile Organic Compounds

No TAL compounds were detected in either the WHC or the EPA analyses. The median reported detection limits for sample analysis were comparable for both data sets. Though WHC matrix spike recoveries exceeded WHC and EPA quality control criteria, none was qualified as an estimated value. The EPA data set contained several qualified results because of several minor quality assurance deficiencies such as exceedances of quality control criteria for response factors during instrument calibration.

In general, the quality of both data sets was good, and no data were rejected. The median reported detection limits for analytes were similar. However, EPA presented data for approximately 20 more compounds and/or isomers than did the WHC report, indicating a more complete analysis.

### 2.3.2 Volatile Organic Compounds

No TAL compounds were detected in the WHC and EPA samples that were not also detected in blank samples. The median reported detection limits for sample analysis were comparable for both data sets.

In general, the quality of both data sets was good, and no data were rejected. The detection limits for analytes were similar. However, EPA presented data for approximately 30 more compounds and/or isomers than did the WHC report, indicating a more complete analysis.

### 2.3.3 Metals

Though there were some differences between the EPA and the WHC data set for metals, in general the data are comparable (Table 1). The matrix spike recoveries for each data set are also comparable, suggesting that the accuracy of the EPA and WHC data sets are similar. The results from duplicate analyses by EPA and WHC suggest that the effects of sample heterogeneity and laboratory error are similar in both the EPA and WHC analytical procedures.

The RPDs between EPA and WHC metals data from six soil samples are compared in Table 2. The RPD for each analyte ranges from 6.5 for aluminum to 198 for magnesium. Generally, because of dilution problems associated with most metals analyses, the results of common constituents of soil, which are normally present in high concentrations, are less precise than results from trace elements such as arsenic, lead, copper, and other heavy metals.

However, there is not a good correlation between EPA and WHC data for individual analytes. Correlation coefficients ( $r^2$ ) ranged from 0.02 for chromium to 0.86 for magnesium. Generally, the  $r^2$  values are greater for common constituents of soils that are normally present in high concentrations, and lower for trace elements. Trace metal analysis is more precise, but, because much of the data are detected at concentrations near the reported detection limits, sample and instrument error affect the absolute value of the data more than they affect data from common soil constituents.

### 2.3.4 Non-Metals

The results from the analysis of anions are similar, though the reported detection limits for the EPA analysis were somewhat lower (Table 3). The WHC data sets contained many data that are qualified as estimated because sample holding time quality assurance criteria were not met. The WHC quality assurance criteria for holding times was, in some cases, shorter than the EPA criteria. For example, the holding time criteria for phosphate was 28 days for EPA and 48 hours for WHC, which caused more WHC than EPA phosphate data to be qualified as estimated values. In most cases, results were comparable and analyte results are well correlated.

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The major differences between the two data sets were the choice of analytes and analytical methods. The methods of analysis for fluoride, chloride, phosphate, and sulfate were similar. However, WHC analyzed for nitrate/nitrite, whereas EPA presents results for the relevant environmental species of nitrogen, including nitrate, nitrite, and ammonia. In addition, EPA analyzed for hydrogen activity (pH), whereas WHC did not.

### 3.0 CONCLUSIONS AND RECOMMENDATIONS

The analytical results from EPA and WHC data sets were comparable for semivolatile organic compounds, volatile organic compounds, metals, and non-metals. Generally, the main differences between the data sets involved the number of organic analytes and the chosen analytical methods. The EPA data set presents results for approximately 20 more semivolatile compounds and 30 more volatile compounds. In addition, EPA presents results for all of the environmentally relevant nitrogen species whereas WHC presents results for only nitrate/nitrite. The EPA data set presents pH results, which is a very significant environmental parameter, whereas the WHC omits the hydrogen ion as an analyte.

### 4.0 REFERENCES

EPA 1987. Data Quality Objectives for Remedial Response Activities: Development Process. EPA/540/G-87/003. U.S. Environmental Protection Agency. May.

EPA 1993a. Memorandum from Joe Blazeovich (Environmental Chemistry Program) to Pam Innis. U.S. Environmental Protection Agency. January 28.

EPA 1993b. Memorandum from Joe Blazeovich (Environmental Chemistry Program) to Pam Innis. U. S. Environmental Protection Agency. February 9.

EPA 1993c. Memorandum from Isabel Chamberlain to Pam Innis. U.S. Environmental Protection Agency. February 1.

EPA 1993d. Memorandum from Phil Davis to Pam Innis. U.S. Environmental Protection Agency. January 5.

WHC 1993. White Bluffs Pickling Acid Cribs Expedited Response Action Data Validation Report. WHC-SD-EN-TI-159. Westinghouse Hanford Company.

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TABLE 1

EPA AND WHC ANALYTICAL RESULTS COMPARISON:  
TOTAL METALS (mg/kg)

SAMPLE NUMBER (EPA/WHC)							
Analyte	PC-A-SI-10/B07PY8	PC-A-SI-15/B07P21	PC-A-S2-8/B07PY9	PC-E-SI-12/B07P24	PC-B-SI-11/B07P26	PC-B-S2-5/B07Q01	EPA and WHC Duplicate of PC-B-S2-5/B07Q01 PC-B-S2-5/B07Q01 PC-B-S3-5/B07Q02
Arsenic	0.74/ 1.5 J	1.23/ 1.2 J	1.51/ 1.6J	1.78/ 2.3 J	1.03/ 1.2 J	1.35/ 1.3	1.35/ 1.3 J
Lead	4.45/ 3.9	3.94/ 4.1	3.83/ 3.4	3.84/ 4	3.30/ 3.5	4.27/ 3.3	3.99/ 3.3
Chromium	11.9/ 9.1	11.4/ 11.2	10.8/ 9.4	13.1/ 10	12.2/ 7.7	12.6/ 10.2	10.7/ 10.2
Copper	15.8/ 23.5	13.8/ 20.7	15.4/ 16.7 U	14.0/ 17.6 U	15.2/ 15.2 U	14.1/ 14.6	16./ 7.3
Nickel	10.1/ 62	8.52/ 9.5	16.4/ 8.3	11.6/ 10.3	9.54/ 7.9	13.6/ 10.8	11.9/ 10.8
Zinc	90.6/ 71.8	52.9/ 50.7	71.7/ 13.7	38.9/ 30.3	38.3/ 30.5	49.5/ 35.6	50.2/ 35.6
Barium	51.6/ 44.1	40.4/ 36.8	50.6/ 41.2 U	39.0/ 50.8	42.1/ 41	57.8/ 58.3	65.1/ 58.3
Calcium	3,140/ 2,600	3,230/ 2,870	3,730/ 2,810	7,970/ 8,010	3,640/ 2,850	7,190/ 6,410	10,600/ 6,410
Iron	15,500/ 14,600	13,800/ 13,500	16,900/ 14,200	13,000/ 13,200	15,900/ 12,900	17,800/ 15,000	17,700/ 15,000
Magnesium	3,850/ 3,310	4,500/ 4,080	4,490/ 3,610	4,480/ 4,350	4,000/ 2,960	5,270/ 4,620	5,420/ 4,620
Potassium	815/ 820	725/ 763	816/ 862	486/ 794	238/ 542	940/ 1,010	1,090/ 1,010
Sodium	267/ 139 U	210/ 171	298/ 166	235/ 166	241/ 158	267/ 142	344/ 142 U
Vanadium	38.7/ 41.4	31.6/ 34.1	37.2/ 37	28.9/ 32.4	36.5/ 39.2	38.8/ 34.9	37.9/ 34.9
Aluminum	6,804/ 5,360	6,233/ 5,700	7,270/ 5,650	5,920/ 5,550	5,340/ 4,310	7,220/ 5,800	7,570/ 5,730

U Indicates that the analyte was analyzed for, but not detected.

J Indicates an estimated concentration value. Identification of the detected analyte has been confirmed; however, the associated numerical value may not accurately reflect the actual sample concentration.

TABLE 2

STATISTICAL COMPARISON OF EPA AND WHC  
SAMPLE RESULTS FOR METALS

Analyte	Relative Percent Difference	Correlation Coefficient ( $r^2$ )
Arsenic	17.8	0.29
Lead	11.9	0.38
Chromium	20.0	0.02
Copper	23.4	0.037
Nickel	23.7	0.04
Zinc	21.4	0.91
Barium	13.6	0.18
Calcium	20.5	0.78
Cobalt	17.7	0.01
Iron	11.6	0.54
Magnesium	198	0.78
Potassium	21.7	0.86
Sodium	66.1	0.40
Vanadium	7.8	0.36
Aluminum	6.5	0.47

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TABLE 3

EPA AND WHC ANALYTICAL RESULTS COMPARISON:  
NONMETALS (mg/kg)

SAMPLE NUMBER (EPA/WHC)								
Analyte	PC-A-S1-10/ B07PY8	PC-A-S1-15/ B07BZ1	PC-A-SZ-8/ B07PY9	PC-E-S1-12/ B07B24	PC-B-S1-11/ B07B26	PC-B-S2-5/ B07Q01	PC-E-S1-7/ B07Q02	PC-B-S3-5/ B07PZZ
Chloride	0.93/ 1.8 J	0.51/ 1.4 J	0.65/ 2.3 J	0.33/ 2.1 J	0.33/ 2	0.38/ 2 J	0.27/ 2 J	0.32/ 2.1 J
Fluoride	0.94/ 0.3 J	0.68/ 0.6 J	0.79/ 0.4 J	0.18/ 0.8 J	0.62/ 0.4	2.30/ 1 J	0.86/ 1 J	2.13/ 1.1 J
Phosphate	0.13 J/ 0.8 UJ	2.06 J/1 J	2.19 J/ 0.8 J	1.02 J/ 1 J	0.22 J/ 0.8 UJ	1.65/ 1 J	0.68 J/ 1 J	1.63 J/ 1.0 J
Sulfate	42.9/ 25 J	9.05/ 13 J	12.6/ 15 UJ	7.66/ 11 J	7.48/ 8 J	5.44/ 10 J	5.51/ 10 J	11.9/ 11 J

U  
J

Indicates that the analyte was analyzed for, but not detected.

Indicates an estimated concentration value. Identification of the detected analyte has been confirmed; however, the associated numerical value may not accurately reflect the actual sample concentration.

# CORRESPONDENCE DISTRIBUTION COVERSHEET

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Subject: White Bluffs Pickling Acid Crib Expedited Response Action Proposal  
(DOE/RL-93-48, Revision 0) - Review and Recommendations

White Bluffs Pickling Acid Crib Expedited Response Action - Split  
Sampling Data Comparison

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