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**200-BP-1 SITE INVESTIGATION
ANALYTICAL CHEMISTRY SUPPORT PROJECT**

TASK 7

DATA PACKAGE/REPORT No. 14

Revision 0



May 20, 1992



Prepared by: B.M. Gillespie

Pacific Northwest Laboratory

(PNL Project #16772)

DON'T SAY IT --- *Write It!*

DATE: January 26, 1993

TO: File B01R19-PNL-059

FROM: S.D.L.A.

Telephone: 2-3206

CC:

SUBJECT:

VALIDATION DOCUMENTATION

Validation documentation for the above mentioned data package is filed with Data Package B019P1-PNL-058.

INTRODUCTION

This data package contains the results obtained by Pacific Northwest Laboratory (PNL) staff in the characterization of samples for the 200-BP-1 Site Investigation Analytical Chemistry Support Project. The samples were submitted for analysis by Westinghouse Hanford Company (WHC) under the Technical Project Plan (TPP) 16772 and the Quality Assurance Project Plan (QAPjP) ALO-001. The samples are water samples collected in support of Task 7. The analytical procedures required for analysis were defined in the Test Instructions (TI) prepared by the PNL 200-BP-1 Project Management Office in accordance with the TPP and the QAPjP ALO-001.

The samples (Table 1) were submitted with the appropriate WHC Chain of Custody (COC) and Sample Analysis Request Forms. The samples were delivered at refrigerated temperature to the 300 Area, 325 Building 200-BP-1 Sample Custodian.

The requested analyses for these samples were cyanide, free cyanide and ferrocyanide. A "ferrocyanide" result is not obtained nor calculated since the amount of the complex cyanide being ferrocyanide is indeterminate. A complex cyanide result is determined by the difference of the total cyanide and the free cyanide results. The quality control (QC) requirements for each sample are defined in the test instructions for each sample. The QC requirements outlined in the procedures and requested in the WHC SOW were followed. Sample duplicates, method blanks, matrix spikes and laboratory control standards were analyzed. All QC data that exist are included in this Data Package/Report.

The data in this package are reported in separate tables (Tables 2 through 4) for each analyte or method. Three appendices are provided; one for Test Instruction, one for Chain of Custody, Sample Analysis Request Forms and Sample Receipt Forms and one that contains the primary inorganic analytical data.

CERTIFICATION STATEMENT

I certify that this data package is in compliance with the terms and conditions of the TPP 16772 and QAPjP ALO-001 for completeness. Release of the data contained in this hard copy data package and in the computer-readable data submitted on floppy diskette has been authorized by the Project Manager or the Project Manager's designee, as verified by the following signature.

B. M. Gillespie

B. M. Gillespie
200-BP-1 Project Manager

5-20-92

Date

TABLE 1: 200-BP-1 Sample Numbers

<u>WHC Sample Number</u>	<u>PNL ALO Sample Number</u>	<u>Sample Type</u>
B01R19	92-03689	Water
B01QY8	92-03690	Water
B01QZ5	92-03691	Water
B01R01	92-03692	Water

CYANIDE ANALYSIS RESULTS

Total cyanide analysis was performed in room 419 of building 325 in the Hanford Site 300 area. This data package includes cyanide results for four water samples. Cyanide results are presented by colorimetric analysis run batch. Data results are summarized in Table 2.

Total cyanide concentrations for samples 92-03689, 92-03690, 92-03691 and 92-03692 were 9.4, 74.1, 16.2 and 18.1 $\mu\text{g}/\text{l}$, respectively. Duplicate RPD for samples 92-03689 and 92-03692 were 12.5% and 3.8%, respectively.

The 12 day hold time specified for cyanide analysis under the CLP protocol was met for all samples in this data package.

Spiked sample cyanide recovery was 103% with a standard deviation of 3%.

Recovery of cyanide for the laboratory control/initial calibration verification standard (ICV-6) was 103% with a standard deviation of 1%. Recovery value for ICV-6 (LCS-0789, prepared by ICF Corporation) is based on the spiking of 2 ml of stock standard ICV-6 to 500 ml of deionized water and recovery back calculated to the original ICV-6 cyanide concentration.

Cyanide found in blanks analyzed for analysis groups within the data package were below the IDL.

Instrument detection limit for the colorimetric cyanide analysis procedure is 2 $\mu\text{g}/\text{L}$ using the EPA approved procedure for determining IDL.

Accuracy and precision of the colorimetric cyanide analysis can be inferred from cumulative recovery data for the distilled cyanide laboratory control standard (ICV-6). Average recovery of ICV-6, for the period of 10-91 to 2-92, was 103% with a standard deviation of 7%.

TABLE 2: TOTAL CYANIDE ANALYSIS DATA FOR TASK 7
SDG #14

WATER SAMPLES

Sample ID#	PNL Log#	Sample G1 (µg/L)	C	Sample dup. G2 (µg/L)	C	%RPD	Blank G5 (µg/L)	C	Spike added (µg)	Sample+ spike G3 (µg/L)	sample G4 (ICV) (mg/L)	Sample+ spike G3 recovery(%)	sample G4 (ICV) recovery(%)	Flags Q	Footnote# 1,2,3,4,5 (ALL)	
B01R19	92-03689	9.4	B	10.6	B	12.5	2	U	49.25	215.8	9.7	105	104			
B01QY8	92-03690	74.2														
B01QZ5	92-03691	16.2	B	16.9	B	3.8	2	U	49.25	212.7	9.6	100	102			
B01R01	92-03692	18.1	B													
												Mean	103	103		
												Std. Dev.	3	1		

Footnotes

1. Concentration of stock ICV-6=9.4 mg/L (18.8 µg of cyanide is added to each distillation flask and recovered in 250 mL of NaOH).
2. Contract required detection limit for water = 10 µg/L.
3. Used 250 mL of sample per distillation due to limited sample size of 1.5L of total sample. [Except Blank G5]
4. Instrument detection limit is 2 µg/l based on the EPA CLP method.
5. Duplicate precision under the CLP protocol must be within one CRDL when either sample or duplicate are below 5X CRDL.

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FREE CYANIDE ANALYSIS RESULTS

The liquid samples in this sample delivery group were analyzed using procedure PNL-AL0-271, which was derived from Dionex Application Update 107. The sample preparation and analysis were performed in the 325 building in the 300 area.

Each sample is presented on a separate line with sample, sample duplicate, matrix spike, duplicate matrix spike and control standard information in Table 3.

The relative percent difference values for samples analyzed in duplicate, spike recoveries for spiked samples and control standard recoveries are also reported. For soil samples, all analyte values, spike levels, and recoveries are based on dry weights. The control standard is the spiked blank. CLP flags (C) and Quality control flags (Q) are also appended, where appropriate. The CRDL and IDL values are 10 ug/L, & 5 ug/L respectively. Values are reported in ug/L for liquids.

Hold Times

The hold time, analysis within 12 days following receipt, was missed by 2 days. No significant effect on data quality is expected.

Accuracy and Precision in results

The accuracy of reported values between 20-80% of the calibration maximum is estimated to be +10%, unless otherwise noted in the Problems section of this report. The accuracy decreases and errors increase for lower analyte levels and may be 100% at the instrument detection limit.

Quality Control

The criterion for the acceptance of data, that the spiked blank value has been quantitated within $\pm 20\%$, has been met. As this blank was made up in new

eluant (prepared 2/4/92) and a matrix related bias has been observed, further discussion of this phenomenon is included in the sections on spike recoveries and under the Problems section.

A duplicate set of analyses, performed on 92-3690/B01QY8, was recovered within the appropriate CLP window. No analyte was found in this sample. However, the two spike recoveries in another water sample, though low, indicate high precision with almost 0% RPD.

The matrix spike and duplicate matrix spike recoveries, for sample 92-03691/B01QZ5 were not within $\pm 25\%$. However, it appears that acceptable recoveries ($100 \pm 25\%$) are possible if corrections are made for the lack of matrix matching between the sample spikes and the eluant used for standard preparation.

Specifically, if the area defined by the positive y-intercept were added to the areas found for the spiked samples, the recoveries for both the MS and DMS runs would be considered satisfactory. Alternately, the recoveries are acceptable if the y-intercept area is subtracted from all points in the calibration curve and the sample spike peak areas re-quantitated using this new calibration curve.

Problems

Fresh eluant was prepared for these runs and old (prepared 12/91) eluant was used for preparing the calibration standards and for the extractions of solid samples. Based on the evidence available, it appears that the quantitation of free CN is sensitive to matrix matching with the eluant. It appears that it is especially important that calibration standards be prepared in a matrix which is identical to the eluant in use on a particular day.

Upon evaluation of the calibration data it is suggested that the +y-intercept is a reasonable indicator of the lack of matrix matching of the old versus the new eluant. The cause of this aging effect is not understood

at this time and further evaluation of the merits of correcting peak areas of unmatched samples containing CN by adding this term is not possible.

Such a correction should be applied only when CN is found in unmatched samples and the quantitation appears to be suspect. For the case of the sample in this SDG, no chromatographic evidence of CN was observed and therefore no such correction would be required. Recent studies indicate that though quantitation may be affected by matrix effects, the method will clearly indicate the presence of CN at a 5 ug/L level in deionized water or in the eluant. The sample 92-03690/B01QY8 can be considered to contain less than 5 ug/L free CN.

A chemical component, derived from the aging of ethylenediamine, which elutes at the CN retention time is probably responsible for our observations. This may be an addition product of ethylenediamine with CO₂ or an ethylenediamine polymerization product. Other investigative techniques will be used to identify this compound. The results will be reported to the client by the ALO project office as it becomes available.

TABLE 3: FREE CYANIDE ANALYSIS TASK 7
SDG # 14 TASK 7

Sample ID#	PNL Log#	Sample J1		Sample dup. J2		%RPD	--J5-- Matrix Blank		-----J3----- Sample+ Spike		-----J4----- Control Std. Added		-----J6----- Dup. + Spike		----- % Recovery -----			Flags
		(ug/L)	C	(ug/L)	C		(ug/L)	C	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	Spike Rec.	Dup. + Spike Rec.	Control Std. Rec.	
B01QY8	92-03690	5	U	5	U		5	U			46	50					92	H,N
B01QZ5	92-3691	5	U						32	50			32	50	64(a)	64(a)		H,N

CLP FLAGS

U = Analyzed but not detected (less than IDL)
N = Spiked sample recovery not within control limits
H = Holdtime not met

CRDL = 10 ug/L
Estimated IDL = 5 ug/L

NOTE

(a) See the run narrative for additional information on spike recovery calculations.

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COMPLEX CYANIDE ANALYSIS RESULTS

The complex cyanide results are calculated from the difference in the total cyanide results and the free cyanide results. A "ferrocyanide" result is not obtained nor calculated since the amount of the complex cyanide being ferrocyanide is indeterminate.

Samples are analyzed for free cyanide based on first determining that the total cyanide result is greater than 20 ug/L. Samples below 20 ug/L total cyanide are typically not analyzed for free cyanide to save on analysis cost. Attempting to perform free cyanide analysis near the Contract Required Detection Limit of total cyanide is not meaningful.

The free cyanide results for the samples were determined to be below the instrument detection level, therefore treated as zero when calculating the complex cyanide results. The total cyanide concentration is considered to be complex cyanide.

TABLE 4: COMPLEX CYANIDE DETERMINATION

See Table 2 for complex cyanide results, as total cyanide results equal complex cyanide results when free cyanide results is equal to zero.

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Westinghouse
Hanford Company

CHAIN OF CUSTODY

Custody Form Initiator PH BUTCHER

Company Contact PH BUTCHER

Project Designation/Sampling Locations 200-BP-1

Ice Chest No. Delta 2

Bill of Lading/Airbill No. NA

Method of Shipment HAND DELIVER

Shipped to PNL/325

Possible Sample Hazards/Remarks N/A

Telephone (509)376-5045

Collection Date 1-17-92

Field Logbook No. WHC-N-385 #11

Offsite Property No. N/A

Sample Identification

B01R19

1, 1L, P, WATER, FREE CYANIDE

1, 1L, P, WATER, TOTAL CYANIDE

Field Transfer of Custody

Chain of Possession

(Sign and Print Names)

Relinquished by: <i>L.D. Walker</i> L.D. Walker	Received by: <i>PH Butcher</i> PH Butcher	Date/Time: 1/20/92 0715
Relinquished by: <i>PH Butcher</i> PH Butcher	Received by: <i>J. Robbins</i> J. ROBBINS	Date/Time: 1-22-92 1100
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:
Comments:		

B01-002

9713524.0017

Westinghouse
Hanford Company

CHAIN OF CUSTODY

Custody Form Initiator PH BUTCHER
 Company Contact PH BUTCHER
 Project Designation/Sampling Locations 200-BP-1
 Ice Chest No. Delta 2
 Bill of Lading/Airbill No. N/A
 Method of Shipment HAND DELIVER
 Shipped to PNL/325
 Possible Sample Hazards/Remarks N/A

Telephone (509)376-5045
 Collection Date 1/15/92
 Field Logbook No. WHC-NR 4461
 Offsite Property No. N/A

Sample Identification

BOI/QY8
 1, 1L, P, WATER, FREE CYANIDE
 1, 1L, P, WATER, TOTAL CYANIDE

[] Field Transfer of Custody		Chain of Possession	(Sign and Print Names)
Relinquished by: <i>PH Butcher</i> <i>PH Butcher</i>	Received by: <i>PH Butcher</i> <i>PH Butcher</i>	Date/Time: 1/16/92	0710
Relinquished by: <i>PH Butcher</i> <i>PH Butcher</i>	Received by: J. ROBBINS <i>J. Robbins</i>	Date/Time: 1-22-92	
Relinquished by:	Received by:	Date/Time:	
Relinquished by:	Received by:	Date/Time:	
Final Sample Disposition			
Disposal Method:	Disposed by:	Date/Time:	
Comments:			

B01-004

9713524.0019

Westinghouse
Hanford Company

CHAIN OF CUSTODY

Custody Form Initiator PH BUTCHER
 Company Contact PH BUTCHER
 Project Designation/Sampling Locations 200-BP-1
 Ice Chest No. Delta 2
 Bill of Lading/Airbill No. N/A
 Method of Shipment HAND DELIVER
 Shipped to PNL/325
 Possible Sample Hazards/Remarks N/A

Telephone (509)376-5045
 Collection Date 1/15/92
 Field Logbook No. WHC-N-4461
 Offsite Property No. N/A

Sample Identification

B01Q25
 1, 1L, P, WATER, FREE CYANIDE
 1, 1L, P, WATER, TOTAL CYANIDE

[] Field Transfer of Custody		Chain of Possession	(Sign and Print Names)
Relinquished by: <i>K.D. Lee</i> K.D. Lee	Received by: <i>PH Butcher</i> PH Butcher	Date/Time: 1/15/92	11/6/92 0710
Relinquished by: <i>PH Butcher</i> PH Butcher	Received by: <i>J. Robbins</i> J. ROBBINS	Date/Time: 1-22-92	1100
Relinquished by:	Received by:	Date/Time:	
Relinquished by:	Received by:	Date/Time:	
Final Sample Disposition			
Disposal Method:	Disposed by:	Date/Time:	
Comments:			

B01-006

9713524.0021

Westinghouse
Hanford Company

CHAIN OF CUSTODY

Custody Form Initiator PH BUTCHER

Company Contact PH BUTCHER

Telephone (509)376-5045

Project Designation/Sampling Locations 200-BP-1

Collection Date 1-14-92

Ice Chest No. Delta 2

Field Logbook No. W14 C-N-4461

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment HAND DELIVER

Shipped to PNL/325

Possible Sample Hazards/Remarks N/A

Sample Identification

B01R01

- 1, 1L, P, WATER, FREE CYANIDE
- 1, 1L, P, WATER, TOTAL CYANIDE

Field Transfer of Custody

Chain of Possession

(Sign and Print Names)

Relinquished by:	Received by:	Date/Time:
<i>L.D. Walker</i> L.D. Walker	<i>PH Butcher</i> PH Butcher	11/5/92 0828
<i>PH Butcher</i> PH Butcher	<i>J. Robbins</i> J. Robbins	1-22-92 1100
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:
Comments:		

SAMPLE RECEIPT FORMDelivered by: DUSTY BUTCHER Date/Time: 1-22-92 1100Received by: J. ROBBINSCustomer Name or Project: TASK 7, 200BP-1Customer Sample Number(s): BØIR19, BØIQY8, BØIQZ5, BØIRØ1ALO Sample Number(s): 92-03689, 92-03690, 92-03691, 92-036921. Customer Chain-of-Custody Form: Present Absent _____

2. Additional Shipping Forms (list):

SAR

3. Custody Seals on Shipping and/or Sample Containers and their Conditions.

Present Absent _____If Present, Condition: INTACT

4. Sample Tag(s) ID Numbers if not Recorded on the Chain-of-Custody Record or on Sample Vial.

Notes: N/A

5. Condition of Shipping Container (i.e., broken container, dented, breached plastic bag, temperature of sample container as defined in Section 3.0 in PNL-ALO-051, etc.)

OK - 2°C

6. Condition of Sample Vials.

OK

7. Verification of Agreement or Nonagreement of Information on Receiving Documents.

OK

8. Resolution of Problems or Discrepancies.

OK

RETURN COMPLETED FORM TO PROJECT MANAGER

B01-010

9713524.0024

Samples were delivered directly to the Analysts. Therefore, no other PNL Chain of Custody forms were needed.

B02=002