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200-BP-1 SITE INVESTIGATION
ANALYTICAL CHEMISTRY SUPPORT PROJECT

TASKS 2 & 4

DATA PACKAGE/REPORT No. 17

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Pacific Northwest Laboratory

(PNL Project #16772)

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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 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 (i.e., packed in ice in coolers) to the 300 Area, 325 Building and 329 Building 200-BP-1 Sample Custodians.

The requested analyses for sample numbers 92-05877, 92-05953 and 92-05954 are the full suite as specified in the WHC SOW. The full suite of analyses parameters of interest are; nitrate, nitrite, phosphate, sulfate, cyanide, free cyanide, complex cyanide, bismuth, the EPA Contract Laboratory Program inorganic target analytes, total alpha, total beta, cesium-137, cobalt-60, ruthenium-106, plutonium-239/240, plutonium-238, strontium-90, technetium-99, total uranium activity, tritium, total organic carbon and the EPA Contract Laboratory Program organic target compound list. The other eight samples were submitted for total and free cyanide analyses. Weight percent solid is also determined for soil samples (Table 2). All data are corrected to dry weight except where otherwise stated. The quality control (QC) requirements for the samples are defined in the test instructions for each sample. The QC requirements outlined in the procedures and requested in the WHC SOW were followed. Method blanks, matrix spikes, sample duplicates and/or matrix spike duplicates were analyzed. All QC data that were required are included in the appendices of this Data Package/Report.

The data in this package are reported in separate tables or CLP Forms (Tables 2 through 16 and CLP Forms) for each analyte or method. Five appendices are provided; one for Test Instruction, one for Chain of Custody, Sample Analysis Request Forms and Sample Receipt Forms, one that contains the

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primary inorganic analytical data, one that contains the primary radiochemistry analytical data, and one that contains the primary organic analytical data and full CLP data tables.

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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.

MW Thim for BMG

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

10-9-92
Date

TABLE 1: 200-BP-1 Sample Numbers

WHC Sample Number	PNL ALO Sample Number	Sample Type	*Analyses Requested
B01SH7	92-05877	Soil	Full Suite
B01SJ0	92-05878	Soil	CN Suite
B01SJ2	92-05879	Soil	CN Suite
B01SJ4	92-05880	Soil	CN Suite
B015S2	92-05881	Water	CN Suite
B01SB0	92-05882	Water	CN Suite
B01SB4	92-05953	Soil	Full Suite
B01SC8	92-05954	Soil	Full Suite
B01SF0	92-06336	Soil	CN Suite
B01SG0	92-06337	Soil	CN Suite
B01SG8	92-06338	Soil	CN Suite

* CN Suite - Total CN, Free CN and Complex CN
Full Suite - Anions, Bismuth, CLP Inorganic Target Analytes, Radiochemistry Suite, TOC and CLP Organic Target Compounds (as defined in Introduction, pg 2.)

TABLE 2:

WEIGHT PERCENT SOLIDS SUMMARY SHEET

SAMPLE ID#	PNL LOG#	SAMPLE WT %	DUPLICATE WT %	AVERAGE SOLIDS WT %
B01SH7	92-05877	96.28	96.29	96.3
B01SJ0	92-05878	97.49	97.67	97.6
B01SJ2	92-05879	96.68	96.58	96.6
B01SJ4	92-05880	95.42	95.43	95.4
B01SB4	92-05953	97.72	97.68	97.7
B01SC8	92-05954	97.94	97.86	97.9
B01SF0	92-06336	94.18	94.25	94.2
B01SG0	92-06337	94.14	93.91	94.0
B01SG8	92-06338	97.18	97.34	97.3

Note: Weight Percent Solids were determined following the method outlined in PNL-ALO-504.

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ANION ANALYSIS RESULTS

Ion Chromatography

The samples were extracted using procedure PNL-ALO-108 and were analyzed using procedure PNL-ALO-212, in accordance with EPA Method 300.0. The samples were analyzed on 6/02/92. The sample preparation and analyses were performed in the 325 buiding in the 300 area.

Data presentation (see Table 3a-3d, and the notes below)

The anion analysis results have been listed on a separate page with sample, sample duplicate, matrix spike, duplicate matrix spike, and control standard information.

RPD values for samples analyzed in duplicate, spike recoveries for spiked samples, and control standard recoveries have also been reported. For soil samples, all analyte values, spike levels, and recoveries were based on dry weights.

The control standard for all anions has been defined as the spiked blank.

Concentration flags (C) and quality flags (Q) have also been appended, where appropriate.

CRDL and IDL values

Analyte	CRDL µg/L	CRDL mg/kg	IDL µg/L	IDL mg/kg
Nitrite-N	15	1	8	0.8
Nitrate-N	15	1	7	0.8
Phosphate-P	60	5	30	1.7
Sulfate	250	20	51	4.0

Reported values for solids in mg/Kg. The IDL values for liquid samples have been derived from the standard deviations of analyses of multiple sets of the lowest calibration standard, CAL 1. For solid samples, at an extract buffer weight to sample weight ratio of 10, the IDL values have been estimated to be 0.8xCRDL for NO_2-N and NO_3-N, 0.33xCRDL for PO_4-P, and 0.2xCRDL for SO_4.

Hold Times

The hold time of 48 hours after extraction (for soils) was met for all of the samples analyzed.

Accuracy and Precision in IC Results

The accuracy of reported values between 20-80% of the calibration maximum has been estimated to be ±10%, unless otherwise noted in this report. This level of accuracy may be considered achievable throughout the calibration range unless otherwise specified. The accuracy decreases and errors increase for lower analyte levels and may be 100% at the instrument detection limit.

Quality Control

The criteria for the acceptance of data, that the spiked blank values for the anions of interest are quantitated within ±20%, has been met for this SDG. The retention time (R.T.) window for the anions of interest, set at ±10%, has been met.

Spike and duplicate spike recoveries on sample 92-05954/B01SC8 were within the acceptable limits of ±25% for nitrite and phosphate. Variation in spike recovery and duplicate analysis of PO_4-P could be caused by heterogeneity of these very coarse soils. The spikes for nitrate and sulfate were significantly less than 25% of the anion concentration; therefore, the calculated spike recoveries are meaningless.

Sample 92-05877/B01SH7 was analyzed in duplicate with RPD values within specified limits for all analytes. Recoveries on all analytes in the control

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standard (spiked method blank) were within the ±10% window; except for NO_2-N which was at 89% recovery.

TABLE 3A: ANION IC ANALYSIS DATA TASK 2 & 4 SDG 17
NITRITE (NO2-N)

CUST ID	ALO #	C1 SAMPLE (mg/Kg)	C	C2 DUP (mg/Kg)	C	RPD	C5 BLANK (mg/Kg)	C	C3 SMPL+ SPIKE (mg/Kg)	C3 SPIKE (mg/Kg)	C6 DUP+ SPIKE (mg/Kg)	C6 SPIKE (mg/Kg)	% RECOVERY C3 SMPL SPIKE	% RECOVERY C6 DUP SPIKE	% RECOVERY C4 CNTRL STD.	Q
B01SH7	92-05877	17.5		16.6		5%	0.8	U							89%	
B01SB4	92-05953	42.7														
B01SC8	92-05954	16.1				5%			32.6	15.5	31	15.5	106%	96%		

NOTES:

1. 5 ppm NO2 spike in extract OR 5 ppm X dil. factor / frac. solids spike in sample; see run narrative.
2. RPD values have been calculated using sample duplicates OR the two sample plus spike values.
3. 100% extraction efficiency assumed in defining dil. factor as (diluent vol / sample wt) X 1.00 gm/ml.
4. C5: Method Blank ; C4: Spiked Blank; nominal sample wt=2.00gm.
5. Estimated IDL = 0.8 mg/Kg.
6. CRDL= 1.0 mg/Kg.

Quality Flags [Q]

E: Estimated value, interference present
N: Spike recovery not within control limits
*: Duplicate analysis not within control limits

Concentration Flags [C]

B: IDL <= [Analyte] < CRDL
U: [Analyte] < IDL

TABLE 3B: ANION IC ANALYSIS DATA TASK 2 & 4 SDG 17
NITRATE (NO3-N)

CUST ID	ALO #	C1 SAMPLE (mg/Kg)	C	C2 DUP (mg/Kg)	C	RPD	C5 BLANK (mg/Kg)	C	C3 SMPL+ SPIKE (mg/Kg)	C3 SPIKE (mg/Kg)	C6 DUP+ SPIKE (mg/Kg)	C6 SPIKE (mg/Kg)	% RECOVERY C3 SMPL SPIKE	% RECOVERY C6 DUP SPIKE	% RECOVERY C4 CNTRL STD.	Q
B01SH7	92-05877	467		421		10%	0.8	U							100%	
B01SB4	92-05953	1040														
B01SC8	92-05954	782				7%			854	11.5	793	11.5	626%	96%		

NOTES:

1. 5 ppm NO3 spike in extract OR 5 ppm X dil. factor / frac. solids spike in sample; see run narrative.
2. RPD values have been calculated using sample duplicates OR the two sample plus spike values.
3. 100% extraction efficiency assumed in defining dil. factor as (diluent vol / sample wt) X 1.00 gm/ml.
4. C5: Method Blank ; C4: Spiked Blank; nominal sample wt=2.00gm.
5. Estimated IDL = 0.8 mg/Kg.
6. CRDL = 1.0 mg/Kg.

Quality Flags [Q]

E: Estimated value, interference present
N: Spike recovery not within control limits
*: Duplicate analysis not within control limits

Concentration Flags [C]

B: IDL <= [Analyte] < CRDL
U: [Analyte] < IDL

TABLE 3C: ANION IC ANALYSIS DATA TASK 2 & 4 SDG 17
PHOSPHATE (PO4-P)

CUST ID	ALO #	C1 SAMPLE (mg/Kg)	C	C2 DUP (mg/Kg)	C	RPD	C5 BLANK (mg/Kg)	C	C3 SMPL+ SPIKE (mg/Kg)	C3 SPIKE (mg/Kg)	C6 DUP+ SPIKE (mg/Kg)	C6 SPIKE (mg/Kg)	% RECOVERY C3 SMPL SPIKE	% RECOVERY C6 DUP SPIKE	% RECOVERY C4 CNTRL STD.	Q
B01SH7	92-05877	8.3		8.1		2%	1.7	U							97%	
B01SB4	92-05953	112														
B01SC8	92-05954	98				4%			115	16.5	111	16.6	102%	78%		

NOTES:

1. 5 ppm PO4 spike in extract OR 5 ppm X dil. factor / frac. solids spike in sample; see run na see run narrative.
2. RPD values have been calculated using sample duplicates OR the two sample plus spike values.
3. 100% extraction efficiency assumed in defining dil. factor as (diluent vol / sample wt) X 1.00 gm/ml.
4. C5: Method Blank ; C4: Spiked Blank; nominal sample wt=2.00gm.
5. Estimated IDL = 1.7 mg/Kg.
6. CRDL= 5 mg/Kg.

Quality Flags [Q]

E: Estimated value, interference present
N: Spike recovery not within control limits
*: Duplicate analysis not within control limits

Concentration Flags [C]

B: IDL <= [Analyte] < CRDL
U: [Analyte] < IDL

TABLE 3D: ANION IC ANALYSIS DATA TASK 2 & 4 SDG 17
SULFATE (SO4)

CUST ID	ALO #	C1 SAMPLE (mg/Kg)	C	C2 DUP (mg/Kg)	C	RPD	C5 BLANK (mg/Kg)	C	C3 SMPL+ SPIKE (mg/Kg)	C3 SPIKE (mg/Kg)	C6 DUP+ SPIKE (mg/Kg)	C6 SPIKE (mg/Kg)	% RECOVERY C3 SMPL SPIKE	% RECOVERY C6 DUP SPIKE	% RECOVERY C4 CNTRL STD.	Q
B01SH7	92-05877	462		465		1%	4	U							98%	
B01SB4	92-05953	354														
B01SC8	92-05954	271				6%			336	51	315	51	127%	86%		

NOTES:

1. 5 ppm SO4 spike in extract OR 5 ppm X dil. factor / frac. solids spike in sample; see run narrative.
2. RPD values have been calculated using sample duplicates OR the two sample plus spike values.
3. 100% extraction efficiency assumed in defining dil. factor as (diluent vol / sample wt) X 1.00 gm/ml.
4. C5: Method Blank ; C4: Spiked Blank; nominal sample wt=2.00gm.
5. Estimated IDL= 4 mg/Kg.
6. CRDL= 20 mg/Kg.

Quality Flags [Q]

E: Estimated value, interference present
N: Spike recovery not within control limits
*: Duplicate analysis not within control limits

Concentration Flags [C]

B: IDL <= [Analyte] < CRDL
U: [Analyte] < IDL

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CYANIDE ANALYSIS RESULTS

Total cyanide analysis was performed in room 313 of building 325 in the Hanford Site 300 area. This data package includes cyanide results for 9 soil-sediment samples and two water samples. Cyanide results are presented by colorimetric analysis run batch. Data package results are summarized in Table 4a (Soils/Sediments) and Table 4b (Waters).

Total cyanide results for soil-sediment samples and corresponding duplicates (where applicable) were below the instrument detection limit (IDL) of 0.2 mg/kg for samples B01SJ2, B01SJ4, B01SF0, B01SG0, and B01SG8 and below the IDL of 0.4 mg/kg for sample B01SJ0. Samples B01SH7, B01SB4, and B01SC8 were analyzed at concentrations between 35 and 120 mg/Kg and required Free CN analyses. The total cyanide result for the two water samples was below the IDL of 4 μg/L (which was adjusted for the 250 mL sample volume).

The 12 day hold time specified for cyanide analysis under the CLP protocol was met for all samples in this data package. However, sample B01SC8 (92-05954) did experience some difficulties. The initial distillation met the hold time requirement; however, both the RPD and spike were outside reasonable limits and the sample was reanalyzed. The reanalysis distillation was outside the hold time but the value obtained was similar to those obtained for the initial distillation and it is unlikely that the sample result has been compromised. The excessive spike recovery has been attributed to a double addition of the spike solution.

The average spiked soil-sediment sample CN recovery for the nine samples was 102% with a standard deviation of 2%. However, the spike recovery for the initial analysis of B01SC8 has not been included in the average; the 200+% recovery is suspected to be an inadvertent double spiking of the sample. Spiked water sample cyanide recovery was 103%.

Accuracy and precision can be inferred from the recovery data for the distilled CN laboratory control standard; i.e., ICV-6 (LCS-0789), prepared by ICF Corporation. The average CN recovery for the laboratory control sample

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analyzed with the soil-sediment samples was 104% with a standard deviation of 4%. Recovery of cyanide for the laboratory control sample analyzed with the water samples was 101%. Recovery value for ICV-6 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.

Total cyanide found in blanks analyzed for each analysis set within the data package was below the IDL. The IDL for liquids in the colorimetric cyanide analysis procedure is 2 μg/L, based on the EPA CLP approved procedure for determining IDL. For solids the comparable colorimetric cyanide IDL is 0.2 mg/kg, assuming a sample weight of 5 g.

TABLE 4a: TOTAL CYANIDE ANALYSIS DATA FOR TASK 2&4
SDG #17

SOILS-SEDIMENTS

Sample ID	PNL Log#	Sample G1 (mg/kg)	C	Sample Dup G2 (mg/kg)	C	RPD	Blank G5 (µg/L)	C	Spike added (µg)	Sample + Spike G3 (mg/kg)	ICV G4 (mg/L)	%Recoveries Smpl + Spk G3	%Recoveries ICV G4	Q	Footnote #
B01SH6	92-05692+	0.2	U	0.2	U	N/A	2.0	U	48.8	10.2	10.2	100%	108%		1-5
B01SH7	92-05877	104.4													2,3,5
B01SJ2	92-05879	0.2	U	0.2	B	N/A	2.0	U	48.5	10.4	9.8	103%	104%		1-5
B01SJ0	92-05878	0.4	U												2,3,5
B01SJ4	92-05880	0.2	U	0.2	U	N/A	2.0	U	48.3	10.3	9.7	102%	104%		1-5
B01SB4	92-05953	118.7													2,3,5
B01SC8	92-05954	43.0		57.2		28%	2.0	U	48.3	98.4	9.9	232%	105%	N,*	1-5
B01SF0	92-06336	0.2	U	0.2	U	N/A	2.0	U	48.3	10.8	9.1	105%	97%		1-5
B01SC8++	92-05954	35.6													2,3,5
B01SG8	92-06338	0.2	U	0.2	U	N/A	2.0	U	49.0	10.1	10.1	101%	107%		1-5
B01SG0	92-06337	0.2	U												2,3,5

\+ B01SH6 (92-05692) not part of SDG17
++ Reanalysis of B01SC8
+++ Mean spike recovery does not include initial B01SC8.

Mean+++	102%	104%
Std Dev	2%	4%

Footnotes
1. Stock ICV-6=9.4 mg/L; 2 mL (18.8 µg CN) added to distillation flask & recovered in 250 mL NaOH.
2. Contract required detection limit for soil-sediment = 1.0 mg/kg.
3. IDL for solids is 0.2 mg/kg based on 5 g sample; Sample IDL adjusted for weight used.
4. RPD must be within one CRDL when either sample or duplicate are below 5X CRDL.
5. C Flags: U= <IDL, B= <CRDL but =>IDL; Q Flags: N= Spike Recovery Failed, *= RPD failed.

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TABLE 4b: TOTAL CYANIDE ANALYSIS DATA FOR TASK 2&4
SDG #17

WATERS 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)	ICV G4 (mg/L)	%Recoveries Smpl+ Spk G3	%Recoveries ICV G4	Q	Footnote #
B015S2	92-05881	4.0	U	4.0	U	N/A	2.0	U	48.3	101.7	9.5	103%	101%		1-5
B01SB0	92-05882	4.0	U												2,3,5
											Mean	103%	101%		
											Std Dev	N/A	N/A		

Footnotes
1. Stock ICV-6=9.4 mg/L; 2 mL (18.8 µg CN) added to distillation flask & recovered in 250 mL NaOH.
2. Contract required detection limit for water = 10 µg/L.
3. IDL for liquids is 2 µg/L based on 500 mL sample; Sample IDL adjusted for volume distilled.
4. RPD must be within one CRDL when either sample or duplicate are below 5X CRDL.
5. C Flags: U= <IDL, B= <CRDL but =>IDL; Q Flags: N= Spike Recovery Failed, *= RPD failed.

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FREE CYANIDE ANALYSIS RESULTS

The samples in this sample delivery group were extracted using procedure PNL-ALO-107 and were analyzed using procedure PNL-ALO-271, which was derived from Dionex Application Update 107. The sample preparation and analysis were performed in the 325 building in the 300 Area. The samples were extracted and analyzed on two different dates (i.e., May 14, 1992 and May 20, 1992) and were part of large batches which included samples from SDG #17, #18, and #19 for Task 2&4 and SDG #15 for Task 7. Only the results for the three samples from SDG #17 containing measurable Total CN (i.e., 92-05953/B01SB4, 92-05954/B01SC8, and 92-05877/B01SH7) and supporting QC are reported.

Data presentation (See Table 5 and notes below)

Each sample is listed on a separate line with sample, sample duplicate matrix spike, duplicate matrix spike, and control standard information.

RPD 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 method blank.

Concentration flags (C) and quality flags (Q) are also appended, where appropriate.

CRDL and IDL values

CRDL = 10 μg/L, 1 mg/Kg; IDL = 5 μg/L, 0.5 mg/Kg

Values in μg/L are for liquids and those for solids in mg/Kg. The IDL values are estimated at 0.5*CRDL in the liquid and assumes a 100-fold dilution during extraction for solid samples.

Hold Times

The hold time, analysis within 12 days following sample receipt, was not met for the three reportable samples in this SDG. Samples 92-05953/B01SB4 and 92-05954/B01SC8 missed the 12 day hold time by 19 days and sample 92-05877 missed by 32 days. Since the results are similar to those from SDG #16, #18 and #19, it is reasonable to assume that there has been little impact on validity of the results. However, the soil samples were analyzed immediately following extraction.

Accuracy and Precision

The accuracy of reported values between 20-80% of the calibration maximum is estimated to be ±10%. This value may be used over the complete calibration range 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 ±20%, has been met for both analysis runs. The retention time (R.T.) window for the anion of interest, set as ±10%, also has been met.

It should be noted that the analyst opted to spike the samples at one-fourth the normal level for the May 14 analysis run. The spiking level for the May 20 analysis run was at the normal level.

The matrix spike and duplicate matrix spike recoveries for samples 92-05953/B01SB4 (May 14) and 92-05877/B01SH7 (May 20) were within the acceptable criteria.

Duplicate analyses performed on sample 92-05953/B01SB4 (May 14) were quantitated within the acceptable limits. However, the duplicate analysis

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performed on sample 92-05877/B01SH7 were not analyzed within the acceptable RPD criteria. Sample non-homogeneity is the most probable cause of this type of discrepancy.

TABLE 5: FREE CYANIDE ANALYSIS FOR TASKS 2 & 4
SDG #17

Sample ID#	PNL Log#	Sample J1 mg/kg	C	Sample dup. J2 mg/kg	C	RPD	--J5-- Matrix Blank ug/L	C	-------J3 Sample+ Spike mg/kg	----- Spike Added mg/kg	-------J4 Control Std. mg/kg	-------- Standard Added mg/kg	------J6 Dup. + Spike mg/kg	----- Spike Added mg/kg	------ % Recovery ------ --J3-- Spike Rec.	--J6-- Dup. + Spike Rec.	--J4-- Control Std. Rec.	Q Flags
(May 20, 1992)																		
B01SH7	92-05877	1.65		2.08		23	5	U	6.57	5.12	5.62	5.00	6.27	5.02	96	92	112	H,*
(May 14, 1992)																		
B01SB4	92-05953	2.66		2.50		6	5	U	4.03	1.26			3.72	1.23	109	86		H
B01SC8	92-05954	3.54																H

Concentration Flags [C]

U : [ANALYTE] < IDL
B : IDL <= [ANALYTE] < CRDL

CRDL = 1.0 mg/kg (solids)
Estimated IDL = 5 ug/L (liquids), 0.05 mg/kg (solids)

Quality Flags [Q]

H: Hold time not met
E: Estimated Value
N: Spike recovery not within control limits
* : Duplicate Analysis not within control limits

NOTES:

1. 100% spike level is expected to be 50 ug/L in the extract and 5 mg/kg in a 0. 2 gm sample containing no moisture; 5/14/92 samples were run at 1/4 spike level.
2. 100 % extraction efficiency is assumed in defining the extraction dilution as (diluent vol / sample wt) x 1.00 gm/ml .
3. J5 The method blank value has been used in this column. J4 Control Std. is the spiked method blank.
4. The RPD values have been calculated using duplicate results.
5. Sample 92-05877 analyzed 5/20/92; Samples 92-05953,92-05954 on 5/14/92.

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COMPLEX CYANIDE DETERMINATION

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 indeterminant.

Samples are analyzed for free cyanide based on first determining that the total cyanide result is greater than or equal to 2 mg/Kg for soils or 20 μg/L for water. Soil samples <2 mg/Kg and water samples <20 μg/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.

TABLE 6: COMPLEX CYANIDE DETERMINATION
FOR TASKS 2 & 4 SDG #17

Sample ID#	PNL Log#	Total CN mg/Kg	Free CN mg/Kg	Complex CN mg/Kg (1)
B01SH7	92-05877	104.4	1.70	103
B01SB4	92-05953	118.7	2.70	116
B01SC8(2)	92-05954	39.3	3.50	36

(1) Results calculated by subtracting the free cyanide results from the total cyanide results.

(2) Total CN is average from two analysis runs.

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GRAPHITE FURNACE ATOMIC ABSORPTION ANALYSIS RESULTS

Samples for Task 2 & 4 SDG #17 and the accompanying QC samples from SDG #16 were prepared following acid extraction procedure PNL-ALO-101, "Acid Digestion for Metal Analysis." The methodology is consistent with the CLP procedure for acid digestion of waters and sediments. Extracts were analyzed by graphite furnace atomic absorption (GFAA) spectrometry following procedures PNL-ALO-214 (As), PNL-ALO-215 (Se), PNL-ALO-216 (Bi), PNL-ALO-217 (Pb), PNL-ALO-220 (Tl), and PNL-ALO-221 (Ag). The PNL GFAA procedures are comparable to CLP SOW 788 GFAA Methods. Digestion of samples and GFAA analysis were performed in building 325 and analyses were performed on 6/11/92 through 6/25/92. The CLP SOW 788 required hold times of 180 days was met.

The accuracy of the analyses can be estimated from the average of the ICV and CCV percent recoveries, while the analytical precision can be ascertained from the standard deviation of these recoveries.

		Ag	As	Bi	Pb	Se	Tl
ICV/CCV	%Recovery:	100%	98%	99%	108%	99%	104%
	Std. Dev.:	1%	3%	2%	2%	4%	4%

The RPD was omitted in Table 7a for Ag, Bi, Se and Tl since the measured concentrations were below the instrument detection limits. The RPD for As was calculated even though the sample results were below 5x CRDL; the 1 CRDL limit was applied. All samples contained significant quantities of Pb; the RPD was 42% requiring a "*" flag for Pb results in this SDG.

Various problems were encountered during the analysis of this SDG. The problems and issues are addressed on either a "per analyte" or "general" basis.

******** Selenium:
The Se post-digestions spike recoveries were unacceptable on the first set of analyses (6/16/92) and a 10x dilution was performed on the sample and reanalysis was performed (6/17/92 and 6/18/92).

The Se results for all samples were below the CRDL and, since the reanalysis spike recoveries remained very low the Se results were flagged (i.e., "W").

The soil matrix and the presence of Fe may bias Se and As results when deuterium background correction is used (as is the case with the PE 5100) rather than Zeeman background correction. The reportable detection limit for Se has been increased by ten-fold due to the necessity of diluting the samples to reduce the matrix interferences responsible for poor analytical spike recoveries.

The initial runs for Se produced negative analytical spike recoveries due to the requirement that the sample results which are less than the IDL be set to zero to calculate the spike recovery. However, by using measured sample and spiked sample absorbances (negative or positive) to calculate the recovery of the spike, all analytical spike recoveries ranged between 78% to 96%. This provides information to support the contention that the negative absorbances are due to signal suppression caused by a matrix constituent and not due to an analysis or system error.

******** Silver:
The LCS sample continues to analyze on the low side and failure to meet the performance criteria has been a recurrent issue. Other standards containing Ag typically analyze at the certified value, raising concerns that the LCS is suspect or that the analytical solution was diluted by 10x rather than 5x. Additional checks are being performed to ascertain the difficulties with the LCS. However, based on past results of other project samples and the absence of any significant signal for any of the samples in this SDG, reanalysis was not performed. All sample solutions had measured concentrations less than the IDL of 0.69 μg/L.

******** General:
The pre-digestion spikes for all analytes were added in concentrations equivalent to those required by ICP analysis.

9713508.1966

These spike concentrations are 25 to 200 times higher than those prescribed for GFAA analyses. This condition was reported to the Program Manager and authorization was received to proceed with the use of the digestates containing the high spikes. Although the pre-digestion spike concentrations were extremely high, the recoveries for these solutions were quite good as expected.

TABLE 7a: GRAPHITE FURNACE AA ANALYSIS DATA FOR TASKS 2 & 4 -- SDG #17

Analyte	Sample ID#	PNL Log#	B1 Sample mg/Kg	B1 Post Spike Rec	B1 Flags C	B1 Flags Q	B2 Dupl. mg/Kg	B2 Post Spike Rec	B2 C	B2 Q	B1&B2 RPD	B5 Blank mg/Kg	B5 Post Spike Rec	B5 C	B3 Sample+ Spike mg/Kg	B3 Digest Spike Rec	B6 (a) B4 LCS mg/Kg	B6 (a) B4 LCS Rec	B6 (a) B4 Post Spike Rec
Ag	B01SG7 (c)	92-05633	0.14	95%	U		0.12	96%	U		N/A	0.14	95%	U	9.82	101%	8.46	38%	94%
As	B01SG7 (c)	92-05633	2.93	112%			3.55	110%			19%	0.19	102%	U	413.87	106%	890.49	97%	102%
Bi	B01SG7 (c)	92-05633	0.30	89%	U		0.24	92%	U		N/A	0.29	96%	U	2006.0	103%	1943.6	97%	(b)
Pb	B01SG7 (c)	92-05633	3.87	93%		*	6.37	85%		*	42%	0.09	99%	B	107.16	104%	247.88	105%	98%
Se	B01SG7 (c)	92-05633	1.29	80%	U	W	1.07	74%	U	W	N/A	0.13	102%	U	374.91	97%	29.76	76%	102%
Tl	B01SG7 (c)	92-05633	0.07	102%	U		0.06	102%	U		N/A	0.07	102%	U	413.20	107%	43.63	112%	102%
Ag	B01SH7	92-05877	0.11	97%	U														
As	B01SH7	92-05877	2.67	97%															
Bi	B01SH7	92-05877	7.73	93%															
Pb	B01SH7	92-05877	7.95	96%		*													
Se	B01SH7	92-05877	1.04	72%	U	W													
Tl	B01SH7	92-05877	0.07	101%	B														
Ag	B01SB4	92-05953	0.10	95%	U														
As	B01SB4	92-05953	2.79	96%															
Bi	B01SB4	92-05953	9.32	91%															
Pb	B01SB4	92-05953	11.48	103%		*													
Se	B01SB4	92-05953	0.93	78%	U	W													
Tl	B01SB4	92-05953	0.05	104%	U														

The CRDL (IDL) in ug/L: for AA5100 PE (05/18/92) Ag=10 (0.69), As=10 (0.93), Bi=60 (1.44), Pb=3 (0.45), Se=5 (0.63), Tl=10 (0.33)
The CRDL (IDL) in mg/Kg @200X: Ag=2.0 (0.14), As=2.0 (0.18), Bi=12 (0.30), Pb=0.6 (0.09), Se=1.0 (0.13), Tl=2.0 (0.07)
The analytical spike levels in ug/L is 20 for As, Bi, Pb, Tl and 10 for Ag, Se.
LCS standard: Ag, As, Pb, Se, Tl -- ICF 0287; Bi -- Inorganic Ventures G-BI0119
ICV/CCV used during analyses: Ag, Pb, Tl -- ICF ICV4(1290); As, Se -- ICF ICV2(1290); Bi--IV G-BI0119.
Pre-digestion spike (ug/L): Ag=50, As=2000, Bi=10000, Pb=500, Se=2000, Tl=2000. Note that these spike levels are for those required by ICP (not GFAA); no redigestions for GFAA levels were performed.

(a) The spiked blank (B6) is used for the Bi control standard; spike is at 10,000 ug/L (equivalent to 2000 mg/Kg).
(b) No analytical spike performed on these samples.
(c) Sample not part of SDG17 -- included for QC purposes.
(d) Analysis dates: Ag (6/15/92), As (6/11/92), Bi (6/19 & 6/24/92), Pb (6/22 &6/25/92), Se (6/17 & 6/18/92), Tl (6/12 & 6/18/92).

CLP Flags: U = [Analyte] < IDL B = IDL <= [Analyte] < CRDL
N = Pre-digestion Spike Recovery not within control limits.
W = Post-digestion spike recovery not within control limits; sample absorbance < 50% of spike absorbance.
* = RPD not within control limits.

9713508.1967

TABLE 7a: GRAPHITE FURNACE AA ANALYSIS DATA FOR TASKS 2 & 4 -- SDG #17 (continued)

Analyte	Sample ID#	PNL Log#	B1 (a) Sample mg/Kg	B1 Post Spike Rec	Flags C	Flags Q
Ag	B01SC8	92-05954	0.09	95%	U	
As	B01SC8	92-05954	2.61	96%		
Bi	B01SC8	92-05954	4.61	91%		
Pb	B01SC8	92-05954	8.49	99%		*
Se	B01SC8	92-05954	0.84	73%	U	W
Tl	B01SC8	92-05954	0.06	98%	B	

The CRDL (IDL) in ug/L: for AA5100 PE (05/18/92) Ag=10 (0.69), As=10 (0.93), Bi=60 (1.44), Pb=3 (0.45), Se=5 (0.63), Tl=10 (0.33)
The CRDL (IDL) in mg/Kg @200X: Ag=2.0 (0.14), As=2.0 (0.18), Bi=12 (0.30), Pb=0.6 (0.09), Se=1.0 (0.13), Tl=2.0 (0.07)
The analytical spike levels in ug/L is 20 for As, Bi, Pb, Tl and 10 for Ag, Se.

(a) Supporting QC (e.g., LCS, duplicates, pre-digestion spikes) shown in Table 7a.
(b) Analysis dates: Ag (6/15/92), As (6/11/92), Bi (6/19 & 6/24/92), Pb (6/22 &6/25/92), Se (6/17 & 6/18/92), Tl (6/12 & 6/18/92).

CLP Flags: U = [Analyte] < IDL B = IDL <= [Analyte] < CRDL
W = Post-digestion spike recovery not within control limits; sample absorbance < 50% of spike absorbance.
* = RPD for batch duplicate not within control limits.

9713508.1968

MERCURY COLD VAPOR ATOMIC ABSORPTION ANALYSIS RESULTS

Samples for Task 2 & 4 SDG #17 and their accompanying QC samples were analyzed by cold vapor atomic absorption (CVAA) spectrometry following procedure PNL-ALO-213, Mercury in Water, Solids, and Sludges by Manual Cold Vapor Technique." The PNL manual CVAA procedure is consistent with CLP SOW 788 CVAA Method. Digestion of samples and CVAA Hg analysis were performed in the 325 building, laboratory 313. The CLP SOW 788 and contract required hold time of 26 days was met for the initial analysis of the samples; however, failure of both the spike recovery and the RPD prompted reanalysis. The reanalysis missed hold time by 13 to 20 days. The results of the initial analyses are included in the summary report for comparison with the reruns. It should be noted that there is no significant difference between the results obtained on 4/24/92 and those obtained on 5/21/92. The following summarizes the hold times:

Sample ID	Received	Initial Analysis	Rerun Analysis	Reruns Missed Hold time
B01SH7	04/06/92	04/24/92	05/21/92	20 days
B01SB4, B01SC8	04/13/92	04/24/92	05/21/92	13 days

Due to difficulties in obtaining consistent acceptance of the ICV/CCV analysis and also in meeting the recovery requirements of the LCS, minor modifications of the procedure have been documented (DR-91-99) and implemented. These modifications, which were primarily changes in the reaction chamber design and path length reductions, improve the detection limit by 10x and significantly improve reproducibility at the concentration levels required by the contract. These modifications have been incorporated into a new revision of the Hg procedure which is currently undergoing technical review.

The CVAA Hg analyses were conducted on a Perkin-Elmer 5000 AA instrument. The results for the samples are reported in Table 8, along with the QC spike and duplicate from B06809 (a sample not part of SDG #17) and LCS recoveries.

9713508.1970

For the 5/21/92 Hg analysis batch the average recovery for the three ICV/CCV analyses was 98.6% with a standard deviation of 2.4%. The analysis of the LSC (0287) sample was 9.6 mg/Kg (within the 8.5-17.0 certified range). The pre-digestion spike recovery was 101%. Precision, as indicated by the RPD from two sets of sample and duplicate analyses, was approximately 10%.

TABLE 8: Hg COLD VAPOR AA ANALYSIS DATA FOR TASKS 2 & 4
SDG #17

SOLID SAMPLES

Sample ID#	PNL Log#	B1 Sample mg/Kg	Flags C	Flags Q	B2 Dupl. mg/Kg	C	B1&B2 %RPD	B5 Blank mg/Kg	C	B3 (a) Sample +Spike mg/Kg	B3 (a) Digest Spike %rec	B4 (b) LCS mg/Kg	B4 (b) LCS %rec
B06809+	92-06904	0.03			0.04		11%	0.004	U	0.24	101%	9.56	75%
B01SH7	92-05877	0.26			0.24		6%						
B01SH7$		0.21											
B01SB4	92-05953	0.10											
B01SB4$		0.06											
B01SC8	92-05954	0.13											
B01SC8$		0.11											
	SRM 2704											1.30	89%

+ B06809 not part of SDG 17, included for QC purposes for 5/21/92 analyses.
$ Results are from 4/24/92 analyses, Spike & RPD Failed.

(a) B3 Predigestion Spike = 0.05 ug Hg in 25 mL aliquot. (Analysis aliquot: sample 25mL, spike 10mL)
(b) LCS 0287 Hg certified at 12.7 mg/Kg (Range 8.5 to 17.0 mg/Kg)
(c) If analyzed, NIST SRM2704 certified at 1.47 ug/g Hg
(d) RPD only calculated if both sample and duplicate are >IDL
(e) IDL = 0.04 ug/L [or 0.004 mg/Kg -- (0.04ug/L * 0.1L) / 1g]
(f) CRDL = 0.2 ug/L [or 0.02 mg/Kg -- (0.2ug/L * 0.1L) / 1g]
(g) Calibration standards NIST SRM3133, ICV/CCV standard Johnson-Matthey 14395

INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROPHOTOMETRIC ANALYSIS RESULTS

Samples and their accompanying quality control samples were prepared following acid digestion by procedure PNL-ALO-101, "Acid Digestion for Metal Analysis." The methodology is consistent with the CLP procedure for the acid digestion of waters and sediments. Digestates were then analyzed by Inductively Coupled Plasma Atomic Emission Spectrophotometry (ICP-AES) following procedure PNL-ALO-211. This method is comparable to EPA method 200.7 CLP-M. Calibration standard source and preparation information are located in the PNL-ACL instrument archive file: "ICP-325-405." The analysis was performed in ICP lab 405, building 325 in the 300 area.

The data is presented following U.S. EPA - CLP reporting format according to the SOW 788 protocol. Analyte concentrations are reported as dry weight corrected concentrations on "FORM I - IN, INORGANIC ANALYSES DATA SHEET." Spike sample recoveries are reported on "FORM V (Part 1) - IN, SPIKE SAMPLE RECOVERY." Duplicates and RPD's (Relative Percent Differences) are reported on "FORM VI - IN, DUPLICATES." Laboratory quality control sample results are reported on "FORM VII - IN, LABORATORY CONTROL SAMPLE."

This discussion is relative to the following samples (ALO Log-In #'s) in SDG #17: 92-05877, 92-05953 and 92-05954.

This report contains the results for all of the samples including two pre-digestion and post-digestion spiked samples recovery tests. The pre-digestion spiked sample was within the recovery limits. This was the first time that antimony has ever passed the pre-digestion spike recovery test. Because antimony has routinely failed the pre-digestion spike recovery test in the past, a "post-digestion" spiked sample was prepared and analyzed. This post-digestion spiked sample result for antimony was within acceptance limits.

Precision and accuracy for the quality control samples were within EPA acceptance limits with the following exceptions: low aluminum recovery from the laboratory control sample; low pre-digestion spike recovery for cadmium and manganese; poor duplicate sample precision (21% to 48% relative percent difference) for aluminum, cadmium, calcium, chromium, copper, iron, magnesium,

manganese, vanadium, and zinc; and a slightly elevated cadmium level in the preparation blank. A continuing calibration blank analysis was inadvertently omitted near the beginning of a run. Examination of the raw data indicated that the calibration blanks which were analyzed before and after this period of time in the run were all below contract required detection limit (CRDL) or below the instrument detection limit (IDL). Therefore the omission of this single calibration blank should not have any significant affect on the data results.

The poor duplicate sample precision and pre-digestion spiked sample recovery failure for cadmium and manganese is most likely attributable to sample inhomogeneity. Especially since the "soil" samples were gravelly-coarse sand in texture, with rock chips measuring one centimeter or more and containing hydrous oxide coatings. A pre-digestion spiked blank was measured, even though not required, in an attempt to confirm the proper concentration for antimony in the pre-digestion spiking solution. The pre-digestion spiked matrix blank indicated acceptable recovery (100±6%) for all of the spiked analytes, including antimony. Since the pre-digestion spiked blank recovery was acceptable and the pre-digestion spiked sample recovery was acceptable, with the exception of cadmium and manganese, it appears that the cause of the pre-digestion spiked sample recovery failure for cadmium and manganese was a result of sample inhomogeneity. Post-spiking for cadmium and manganese was not performed.

Cadmium in the preparation blank was reported to be about 4.4 mg/Kg. The reported cadmium concentrations in all of the samples were lower than the preparation blank. The cause of the apparent cadmium contamination in the preparation blank is not known therefore the reported sample cadmium concentrations may be biased high by up to 4.4 mg/Kg.

The cause for the low recovery for aluminum in the laboratory control standard LCS(0287) is not known. Calibration verification standards were well within the 90% to 110% control limits. The aluminum concentration in the LCS(0287) is very low (approximately 300 mg/Kg) compared to the concentration of aluminum found in the samples (5000 to 10,000 mg/Kg).

9713508.1974

U.S. EPA - CLP

1

INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.

B01SB4

Lab Name: BATTELLE_PNL Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: 15--17

Matrix (soil/water): SOIL_ Lab Sample ID: 92-05953-B

Level (low/med): LOW__ Date Received: 04/13/92

% Solids: _97.7

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	6940		*	P
7440-36-0	Antimony	3.3	U		P
7440-39-3	Barium	49.4			P
7440-41-7	Beryllium	0.27	B		P
7440-43-9	Cadmium	0.62	B	N*	P
7440-70-2	Calcium	8310		*	P
7440-47-3	Chromium	12.4		*	P
7440-48-4	Cobalt	5.9	B		P
7440-50-8	Copper	11.7		*	P
7439-89-6	Iron	16900		*	P
7439-95-4	Magnesium	4640		*	P
7439-96-5	Manganese	259		N*	P
7440-02-0	Nickel	94.0			P
7440-09-7	Potassium	945	B		P
7440-23-5	Sodium	3970			P
7440-62-2	Vanadium	34.2		*	P
7440-66-6	Zinc	36.7		*	P

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

ORIGINAL -- SOIL SAMPLE, ACID LEACHED

DATE ANALYZED: 20 MAY 1992

TASK 2 & 4 SDG# 17

FORM I - IN

3/90

U.S. EPA - CLP

1
INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.

B01SC8

Lab Name: BATTELLE_PNL Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: 15--17

Matrix (soil/water): SOIL_ Lab Sample ID: 92-05954-B

Level (low/med): LOW__ Date Received: 04/13/92

% Solids: _97.9

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum_	6970		*	P
7440-36-0	Antimony_	2.9	U		P
7440-39-3	Barium___	56.0			P
7440-41-7	Beryllium	0.25	B		P
7440-43-9	Cadmium__	0.38	U	N*	P
7440-70-2	Calcium__	7610		*	P
7440-47-3	Chromium_	12.8		*	P
7440-48-4	Cobalt___	6.2	B		P
7440-50-8	Copper___	12.8		*	P
7439-89-6	Iron____	17800		*	P
7439-95-4	Magnesium	4920		*	P
7439-96-5	Manganese	273		N*	P
7440-02-0	Nickel___	55.3			P
7440-09-7	Potassium	906	B		P
7440-23-5	Sodium___	3400			P
7440-62-2	Vanadium_	37.2		*	P
7440-66-6	Zinc____	37.5		*	P

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:
ORIGINAL_--_SOIL_SAMPLE,_ACID_LEACHED
DATE_ANALYZED:_20_MAY_1992
TASK_2_&_4_SDG#_17

FORM I - IN

3/90

U.S. EPA - CLP

1
INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.

B01SG7

Lab Name: BATTELLE_PNL Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: 15--17

Matrix (soil/water): SOIL_ Lab Sample ID: 92-05633-B

Level (low/med): LOW__ Date Received: 03/31/92

% Solids: _97.6

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	8010		*	P
7440-36-0	Antimony	2.9	U		P
7440-39-3	Barium	78.9			P
7440-41-7	Beryllium	0.25	B		P
7440-43-9	Cadmium	3.4		N*	P
7440-70-2	Calcium	8370		*	P
7440-47-3	Chromium	10.4		*	P
7440-48-4	Cobalt	9.1	B		P
7440-50-8	Copper	17.8		*	P
7439-89-6	Iron	25900		*	P
7439-95-4	Magnesium	4670		*	P
7439-96-5	Manganese	325		N*	P
7440-02-0	Nickel	8.4			P
7440-09-7	Potassium	767	B		P
7440-23-5	Sodium	1620			P
7440-62-2	Vanadium	68.2		*	P
7440-66-6	Zinc	52.5		*	P

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:
ORIGINAL -- SOIL SAMPLE, ACID LEACHED
DATE ANALYZED: 20 MAY 1992
TASK 2 & 4 SDG# 16

FORM I - IN

3/90

9713508.1977

U.S. EPA - CLP

1

INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.

B01SH7

Lab Name: BATTELLE_PNL Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: 15--17

Matrix (soil/water): SOIL_ Lab Sample ID: 92-05877-B

Level (low/med): LOW__ Date Received: 04/06/92

% Solids: _96.3

Concentration Units (ug/L or mg/kg dry weight): MG/KG

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	7610		*	P
7440-36-0	Antimony	2.7	U		P
7440-39-3	Barium	89.0			P
7440-41-7	Beryllium	0.31	B		P
7440-43-9	Cadmium	0.36	U	N*	P
7440-70-2	Calcium	8820		*	P
7440-47-3	Chromium	14.3		*	P
7440-48-4	Cobalt	8.9	B		P
7440-50-8	Copper	17.1		*	P
7439-89-6	Iron	24700		*	P
7439-95-4	Magnesium	5280		*	P
7439-96-5	Manganese	341		N*	P
7440-02-0	Nickel	88.5			P
7440-09-7	Potassium	831	B		P
7440-23-5	Sodium	1770			P
7440-62-2	Vanadium	61.9		*	P
7440-66-6	Zinc	48.1		*	P

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

ORIGINAL -- SOIL SAMPLE, ACID LEACHED

DATE ANALYZED: 20 MAY 1992

TASK 2 & 4 SDG# 17

FORM I - IN

3/90

ICP/MS ANALYSIS RESULTS

Samples in this SDG were not analyzed for Tc-99 and U concentrations as radioactive dose levels of leachate solutions were above instrument allowable radioactivity limits. Leachate solutions were not diluted to allowable limits as the resulting IDL would, most probably, exceed the concentration of analytes of interest in the samples. Samples were analyzed at a later date by alternate methods and/or instruments and will follow in a separate report.

9713508.1979

TABLE 9: ICP-MS Analysis Data

No data available at this time.

9713508.1980

FROM THE DESK OF:

PAT REICH
SAMPLE MANAGEMENT
(509) 372-2785/H4-14

TO: B01SH7-PNL-068

DATE: March 29, 1995

SUBJECT: **TC-99 AND URANIUM DATA FOR B01SH7-PNL-068**

Due to Lab sample batching the Tc-99 and Uranium analysis with the complete raw data requested for this data package is filed in B015P1-PNL-069.

Attached is a copy of the PNL Summary and Cover letter with the sample results included.

Thank You,

Pat Reich
Sample Management

Tc-99 + U are filed in B015P0-PNL-05: for packages B015N0-PNL-061, B015Q4-PNL-056, B015P0-PNL-055, B015Q2-PNL-063, B015C7-PNL-064, B01SH7-PN 068, B067Y5-PNL-066, B06813-PNL-067.

J.M. Duncan
5/23/96

D. E. Ayres
5/23/96

9713508.1981

COPY

Battelle
Pacific Northwest Laboratories
P.O. Box 999
Richland, Washington U.S.A. 99352
Telephone (509) 376-5802
Telex 15-2874

October 14, 1992

OCT 1992
RECEIVED
OSM DMO

Ms. J. M. Duncan, T6-08
Westinghouse Hanford Company
P.O. Box 1970
Richland, Washington 99352

Dear Ms. Duncan:

TRANSMITTAL OF 200-BP-1 SITE INVESTIGATION ANALYTICAL CHEMISTRY SUPPORT PROJECT, TASKS 2 AND 4, ICP-MS Tc-99 AND URANIUM DATA PACKAGE/REPORT FOR NOS. 12-19, REVISION 0, OCTOBER 14, 1992

(Ref: Letter to J. A. Lerch, "Transmittal of Interim Change Notice for Pacific Northwest Laboratory (PNL) Analytical Procedure for the 200-BP-1 Site Investigation Analytical Chemistry Support Project." dated April 1, 1992.)

Enclosed are two copies of the report and one copy of the appendices of the subject 200-BP-1 Tasks 2 and 4, ICP-MS Tc-99 and Uranium Data Package/Report for SDG Nos. 12-19, Revision 0, October 14, 1992 and a PNL Analytical Chemistry Laboratory Data Report/Package Acknowledgement Form for the data report/package. Please sign the form and return to me as agreed upon in the reference above.

Also, a reminder, PNL is to be notified of validation of all data report/packages as defined in the "Memorandum of Understanding Between the Pacific Northwest Laboratory (PNL) and Westinghouse Hanford Company (WHC) on 325 Laboratory Operations in Support of Hanford Site Tank Waste Characterization Activities, March 10, 1992."

If you have any questions about the above or after reviewing the data, please give me a call on 376-5802.

Sincerely,

B. M. Gillespie

B. M. Gillespie
200-BP-1 PNL Project Manager
Analytical Laboratory Operations

Enclosures

9713508.1982

COPY

200-BP-1 SITE INVESTIGATION
ANALYTICAL CHEMISTRY SUPPORT PROJECT

TASKS 2 & 4

ICP-MS Tc-99 and Uranium

DATA PACKAGE/REPORT for SDG NOs. 12-19

Revision 0

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Pacific Northwest Laboratory

(PNL Project #16772)

9713508.1983

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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 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 to the 300 Area, 325 building 200-BP-1 Sample Custodian.

The analyses requested for the samples in this report are technicium-99 and uranium. The quality control (QC) requirements for the samples are defined in the test instructions for each sample. The QC requirements outlined in the procedures and requested in the WHC SOW were followed. Method blanks, matrix spikes, sample duplicates and/or matrix spike duplicates were analyzed. All QC data that exist are included in this Data Package/Report.

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 ICP-MS analytical data.

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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

10/14/92
Date

9713508.1989

200-BP-1 TASKS 2 AND 4 ICP-MS HOT SAMPLE ANALYSES

DATA PACKAGE TASK 2&4, #12

B015P1	92-02332
B015P5	92-02628
B015P9	92-02629
B015Q1	92-02630

DATA PACKAGE TASK 2&4, #13

B015R5	92-03236
B015S1	92-03300

DATA PACKAGE TASK 2&4, #14

B01S75	92-03738
B01S77	92-03901
B01S89	92-03902

DATA PACKAGE TASK 2&4, #15

B01S97	92-04734
B01S99	92-04735
B01SB1	92-04736
B01SB3	92-04968
B01SB7	92-05102
B01SB2	92-05150

DATA PACKAGE TASK 2&4, #16

B01SC7	92-05270
B01SC9	92-05271
B01SD9	92-05352
B01SD7	92-05353
B01SF7	92-05490
B01SF9	92-05491
B01SG3	92-05492
B01SG7	92-05633
B01SG9	92-05634
B01SH3	92-05690

DATA PACKAGE TASK 2&4, #17

B01SH7	92-05877
B01SB4	92-05953
B01SC8	92-05954

200-BP-1 TASKS 2 AND 4 ICP-MS HOT SAMPLE ANALYSES
(continued)

DATA PACKAGE TASK 2&4, #18

B067Y5	92-06428
B067Y7	92-06713
B067Z3	92-06726
B067Z5	92-06727
B06809	92-06904

DATA PACKAGE TASK 2&4, #19

B06813	92-06963
B06815	92-06964
B06823	92-07040
B06825	92-07141
B06827	92-07142

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RADIOCHEMICAL DATA TABLES

ICP/MS ANALYSIS RESULTS

The project samples and accompanying QC were prepared for ICP/MS analysis using procedure PNL-ALO-101, Acid Digestion for Metal Analysis. This methodology is consistent with CLP Inorganics acid digestion for metals. The resulting digestates were further diluted and analyzed by inductively coupled plasma mass spectrometry (ICP/MS) according to procedure PNL-ALO-280, Inductively Coupled Plasma Mass Spectrometric (ICP/MS) Analysis. Technetium was determined using procedure PNL-ALO-281 (ICP/MS Determination of ^{99}Tc), and uranium was determined using procedure PNL-ALO-282 (Determination of Uranium Concentration / Isotopic Composition Using ICP/MS). The calibration standard concentrations for these analyses are documented in the raw data. Samples were prepared in the 325 building and the ^{99}Tc and U determinations were performed on the shielded ICP/MS unit in room 115 of building 3708 in the 300 area. A total of five analytical runs were necessary to include all regular and QC samples. The fifth analysis was performed for samples requiring a reanalysis. Criteria for a reanalysis included memory effects, concentrations outside calibration range, or general inconsistencies.

Previous ICP/MS analyses for 200-BP-1 samples involved the use of an ultrasonic nebulizer (USN) to maximize instrument sensitivity. Unfortunately, a USN for radioactive use has not yet been acquired, so the samples were aspirated using a conventional pneumatic nebulizer. While the extra sensitivity may have been beneficial for determining the technetium concentration, it would have had a negative impact on the uranium analysis. In fact, it is almost certain that the use of a USN would have resulted in additional dilutions to obtain the uranium concentration in many of the samples. As it was, all samples were analyzed for Tc at an additional dilution of only 2X to mitigate matrix effects.

Results were calculated using PQ software as outlined in PNL-ALO-280 and the operations manuals. Additional calculations can be necessary, however, to correct for interferences with elements of interest. At mass 99, technetium is interfered by an isotope of ruthenium; a correction is therefore necessary to subtract out the isobaric ruthenium contribution. To be accurate, this

correction factor assumes natural ruthenium isotopic abundance. There are indications that the apparent Ru in these samples is not of natural isotopic abundance; this is not surprising considering the likely source of this ruthenium. It appears that this ruthenium does not contain a measurable isotope at mass 99. For this reason, correction factors for ruthenium were obtained but were not applied in calculating the technetium concentrations. (These factors are reported in the data table however for information and for the sake of completeness.) Judging from the spiked sample recovery values, the decision not to factor in the possible Ru contribution appears to be legitimate. Some samples were calculated as containing small but measurable quantities of technetium, however the corresponding spectrum did not indicate a positive identifiable peak; these results are reported but parenthesized to indicate qualitative uncertainty.

As specified in ALO-280, appropriate internal standards were used to correct for instrument drift and general signal suppression. The elements chosen were indium (for technetium), thorium and thallium (for uranium). Indium is commonly used as an internal standard due to its absence in all but very few samples. There is more difficulty in choosing an appropriate internal standard for the high masses; there aren't that many of them above mass 200, and those that exist may be present in soils. For this reason, two elements were chosen to represent the high mass response. Unfortunately, the results indicate that many of the samples contain at least one of these elements in measurable concentrations. This was not a problem in 4 of the 5 runs, because indium served as a legitimate substitute; for one run (2922a) however, only uranium was being analyzed and indium was not scanned. To obtain legitimate results for this run, the uranium concentration for all runs was calculated against each internal standard, and the results calculated against Th and Tl were compared with those calculated against In. A thorough analysis of the data indicates that the results calculated against Tl most closely resembled the In results. It appeared that there was a small but measurable quantity of Th in many of the samples (indicated by 10-20% lower uranium concentrations relative to those obtained against In and Tl), and the few samples that contained Tl contained it in significant quantities (indicated by dramatically lower uranium concentrations relative to those

obtained against In and Th). Simply put, the presence of thallium was obvious when reviewing the raw data but the presence of Th was not. Therefore the results obtained on 2922a were calculated using Tl as the internal standard unless Tl was present in the sample. The three samples that contained Tl were calculated using Th as the internal standard.

Quality control measures included the analysis of check standards, sample duplicates, and spiked samples. Technetium matrix spike recoveries show good agreement with true values, but the uranium concentration in the samples is again far greater than the amount spiked, thereby invalidating the spiked sample recovery. Additionally, the blank spike uranium recovery for sample 2628-B6 was negatively affected by a relatively high blank concentration; this is not believed to be significant, however, considering the significantly higher uranium concentration observed in the samples. The uranium recovery for 5633-B6 is acceptable if the uncertainty of the value is considered. Recovery for Tc and U was calculated using the following formula:

Matrix Spike recovery (%) = 100 * ([Spiked Sample] - [Sample]) / [Spike]

Blank Spike recovery (%) = 100 * ([Spiked Blank] - [Blank]) / [Spike]

Unless otherwise specified, the uncertainty of the values reported is estimated at <±10%, and is based on known versus found check standard results, results between duplicate samples, and deviations between analytical runs. Data in parentheses should be considered suspect.

200-BP-1 HOT SOIL ANALYSIS

Tc-99 and Uranium determinations

Sep. 30, 1992

PNL Sample I.D. # (a)	Digest. Dil. Factor (b)	ICP/MS Analysis No.	Analysis Dilution Factor (c)	[Tc-99] ng/ml leachate (d)	[Tc-99] ng/g soil (e,f)	Ru Correctn Factor (g)	[Tc-99] ng/g spiked (f)	[Tc-99] spike yield, % (h)	[Tc-99] pCi/g, dry soil	ICP/MS Analysis No.	Analysis Dilution Factor (c)	[U] ng/ml leachate (d)	[U] µg/g soil (e,f)	[U] µg/g spiked (f)	[U] spike yield, % (h, i)	[U] pCi/g, dry soil (j)
2332 - B1	180.5	2925a32	2x	<0.02	<4				<70	2925a32	2x	148	26.7			18.7
2628 - B1	175.5	2925a38	2x	0.03	5				(90)	2925a38	2x	288	50.5			35.3
2628 - B2	179.6	2925a37	2x	0.04	6				(100)	2925a37	2x	264	51.0			35.6
2628 - B3	172.7	2925a38	2x	0.12	21	0.160	17	90%	350	2925a38	2x	329	56.6	0.173	3639%	39.7
2628 - B5	200.0	2925a35	2x	<0.02	<4				<70	2925a35	2x	2±8	0.4±2			0.3±2
2628 - B6	200.0	2925a33	2x	0.12	24	0.884	20	120%	410	2925a33	2x	1±4	0.2±0.6	0.200	-88%	0.1±0.5
2629 - B1	193.5	2928a17	2x	0.02	5				(80)	2928a17	2x	228	44.1			30.8
2630 - B1	183.6	2925a24	2x	<0.02	<4				<70	2925a24	2x	34.4	6.32			4.41
3236 - B1	197.8	2925a28	2x	0.03	5				(90)	2925a28	2x	43.0	8.50			5.94
3237 - B1	182.0	2921a31	2x	0.04	6				(100)	2922a30	2x	80.0	13.0			9.05
3300 - B1	176.0	2928a19	2x	0.02	4	0.734			60	2928a19	2x	2.51	0.44			0.31
3738 - B1	184.5	2921a19	2x	0.03	6				(100)	2922a29	2x	221	40.8			28.5
3901 - B1	191.7	2921a17	2x	<0.02	<4				<70	2922a27	5x	407	78.0			54.5
3901 - B2	196.7	2925a20	2x	<0.02	<4				<70	2922a22	5x	402	79.1			55.2
3901 - B3	158.4	2925a21	2x	0.11	17	0.197	16	108%	290	2922a23	5x	401	62.7	0.156	-9816%	43.8
3901 - B4	196.0	2925a4	2x	0.03±0.01	6±2				(100±30)	2922a3	2x	75.5	14.8			10.3
3901 - B5	200.0	2925a6	2x	<0.02	<4				<70	2922a4	2x	0.22	0.04			0.03
3901 - B6	200.0	2925a5	2x	0.12	24	0.955	20	120%	410	2922a5	2x	1.10	0.22	0.200	88%	0.15
3902 - B1	163.2	2921a18	2x	0.03	5				(80)	2922a28	5x	1060	173			121
4734 - Spike		2925a7	2x	0.12		0.949				2925a7	2x	9.13				
4734 - B1	186.7	2925a8	2x	<0.02	<4				<70	2925a8	2x	2.8±0.7	0.52±0.13			0.36±0.09
4735 - B1	207.0	2925a16	2x	0.03	7				(100)	2925a16	2x	103	21.3			14.9
4736 - B1	163.9	2925a12	2x	0.06	10				(200)	2925a12	2x	164	30.2			21.1
4968 - B1	144.8	2925a14	2x	<0.02	<3				<50	2925a14	2x	352	51.0			35.6
5102 - B1	204.5	2925a22	2x	<0.02	<4				<70	2928a4	2x	3.80	0.78			0.54
5102 Dup	200.0	2925a27	2x	<0.02	<4				<70	2928a22	2x	5.14	1.03			0.72
5150 - B1	189.0	2925a13	2x	0.02	5				(80)	2925a13	2x	314	59.4			41.5
5270 - B1	174.5	2923a16	2x	0.07	13				(200)	2928a13	50x	1830	319			223
5271 - B1	192.7	2923a9	2x	<0.02	<4				<70	2923a9	2x	273	52.6			36.7
5352 - B1	168.0	2923a23	2x	<0.02	<3				<60	2923a23	2x	346	58.1			40.6
5353 - B1	141.0	2923a4	2x	<0.02	<3				<50	2923a4	2x	290	40.9			28.6
5490 - B1	185.3	2923a20	2x	<0.02	<4				<70	2923a20	2x	451	83.6			58.4
5491 - B1	173.7	2923a19	2x	<0.02	<3				<60	2923a19	2x	91.0	15.6			11.0
5492 - B1	192.1	2928a18	2x	<0.02	<4				<70	2928a18	2x	3.05	0.59			0.41
5633 - B1	205.5	2928a7	2x	0.03±0.02	6±4	0.509			100±70	2928a7	2x	39.0	8.01			5.60
5633 - B2	170.1	2923a21	2x	<0.02	<3				<70	2923a21	2x	45.0	7.65			5.35
5633 - B3	194.3	2923a7	2x	0.13	25	0.891	19	100%	426	2923a7	2x	38.0	7.38	0.194	-325%	5.16
5633 - B4	195.9	2923a13	2x	0.1±0.2	20±40				(340±680)	2923a13	2x	72.0	14.1			9.85
5633 - B5	200.0	2928a9	2x	<0.02	<4				<70	2928a9	2x	0.1±0.1	0.02			0.01±0.01
5633 - B6	200.0	2928a10	2x	0.11	22	0.945	20	110%	370	2928a10	2x	0.8±0.2	0.16±0.04	0.200	70%	0.11±0.03
5834 - B1	170.0	2923a5	2x	0.04	7				(100)	2923a5	2x	577	98.1			68.5
5690 - B1	199.4	2923a25	2x	<0.02	<4				<70	2928a20	2x	2.88	0.57			0.40
5877 - B1	165.7	2923a26	2x	0.03±0.04	5±7				(90±100)	2928a12	50x	1310	217			152
5953 - B1	152.2	2923a22	2x	0.08±0.01	12±2				(200±30)	2923a22	2x	896	136			95.3

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200-BP-1 HOT SOIL ANALYSIS

Tc-99 and Uranium determinations

Sep. 30, 1992

PNL Sample I.D. # (a)	Digest. Dil. Factor (b)	ICP/MS Analysis No.	Analysis Dilution Factor	[Tc-99] ng/ml leachate (d)	[Tc-99] ng/g soil	Ru Correctn Factor	[Tc-99] ng/g spiked	[Tc-99] spike yield, %	[Tc-99] pCi/g, dry soil
5954 - B1	134.3	2923a27	2x	0.08	10				(200)
6428 - B1	146.9	2921a28	2x	0.02	3				(50)
6713 - B1	169.4	2928a8	2x	<0.02	<4				<70
6726 - B1	187.0	2921a22	2x	0.03	5	0.367			80
6727 - B1	194.2	2921a24	2x	0.03	5				(80)
6904 - B1	184.3	2921a29	2x	<0.02	<4				<70
6963 - B1	175.9	2921a8	2x	<0.02	<4				<60
6964 - B1	191.8	2921a32	2x	<0.02	<4				<70
7040 - B1	193.1	2921a23	2x	<0.02	<4				<70
7141 - B1	176.6	2921a21	2x	<0.02	<4				<70
7142 - B1	145.7	2921a7	2x	<0.02	<3				<50
1%HNO3		2923a30	1.053X	<0.01					
1%HNO3		2921a1	1.053X	<0.01					
1%HNO3		2921a5	1.053X	<0.01					
1%HNO3		2921a25	1.053X	<0.01					
1%HNO3		2921a34	1.053X	<0.01					
HNO3/ H20 Blank		2921a6	2x	<0.01					
50 ppt Tc-99		2925a9	1.053X	0.05					
50 ppt Tc-99		2928a14	1.053X	0.05					
100ppt Tc-99		2921a16	1.053X	0.10					
100ppt Tc-99		2921a20	1.053X	0.09					
500ppt Tc-99		2921a11	1.053X	0.46					
500ppt Tc-99		2921a26	1.053X	0.49					
7.6 pCi/L U									
7.6 pCi/L U									
7.6 pCi/L U									
41.0 pCi/L U									
41.0 pCi/L U									
41.0 pCi/L U									
LCS (287)		2925a10	2x	0.02					
Process Blank		2925a11	2x	<0.02					
"Spike"		2925a17	2x	0.12					

ICP/MS Analysis No.	Analysis Dilution Factor	[U] ng/ml leachate (d)	[U] µg/g soil	[U] µg/g spiked	[U] spike yield, %	[U] pCi/g, dry soil
2923a27	2x	652	87.8			61.2
2922a16	20x	3260	488			341
2922a7	5x	409	77.5			54.1
2922a8	2x	146	27.3			19.1
2922a24	2x	138	26.8			18.7
2922a12	2x	36.6	6.75			4.71
2922a20	5x	449	79.0			55.2
2922a13	2x	211	40.4			28.2
2922a14	2x	2.68	0.58			0.39
2922a19	20x	1730	306			213
2922a6	5x	375	54.8			38.2
2925a1	1.053X	<0.05				
2925a23	1.053X	<0.05				
2925a29	1.053X	<0.05				
2923a1	1.053X	<0.05				
2923a14	1.053X	0.30				
2922a10	2x	0.06				
2922a15	1.053X	7.81				
2923a15	1.053X	7.56				
2928a15	1.053X	6.00				
2922a31	1.053X	41.9				
2923a29	1.053X	40.0				
2925a34	1.053X	42.2				
2925a10	2x	69.3				
2925a11	2x	<0.05				
2925a17	2x	<0.05				

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200-BP-1 HOT SOIL ANALYSIS

Tc-99 and Uranium determinations

Sep. 30, 1992

PNL Sample I.D. # (a)	Digest. Dil. Factor (b)	ICP/MS Analysis No.	Analysis Dilution Factor	[Tc-99] ng/ml leachate (d)	[Tc-99] ng/g soil	Ru Correctn Factor	[Tc-99] ng/g spiked	[Tc-99] spike yield, %	[Tc-99] pCi/g, dry soil	ICP/MS Analysis No.	Analysis Dilution Factor	[U] ng/ml leachate (d)	[U] µg/g soil	[U] µg/g spiked	[U] spike yield, %	[U] pCi/g, dry soil

(a) Sample types:
B1 = sample
B2 = sample, duplicate
B3 = sample + Tc/U spike
B4 = laboratory control sample
B5 = procedural blank
B6 = procedural blank + Tc/U spike

(b) units of mL/g dry soil except for -B5 and -B6 samples (blank/blank spike) units of mL.
(c) additional dilution for analysis
(d) = analyzed concentration X analysis dilution factor
(e) = leachate concentration X digestion dilution factor
(f) -B3 reported in units of ng/g for Tc and µg/g for U; -B6 reported in ng for Tc and µg for U.
(g) estimated fraction of mass 99 actually due to technetium based on natural Ru isotopic abundance.
Because Ru isotopic abundance is suspected to be unnatural, this factor was not used in calculating the final [Tc] result.
(h) = ([Spiked Sample] - [Sample]) / [Spike]
(i) not valid for spiked samples; [U] in soil/leachate >> [U] of added spike
(j) all uranium found to be natural isotopic abundance. Total uranium activity calculated using following formula: pCi/g = [U-238]*0.336/0.481

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GAMMA ENERGY ANALYSIS RESULTS

Nineteen soil samples were received between March 5, 1992 and April 6, 1992 as part of SDG #15, #16 and #17 and were analyzed by Gamma Energy Analysis (GEA) as one batch. Four duplicate samples and a matrix blank were analyzed per the Test Instructions received. A total of 24 samples were analyzed by GEA. The sample preparation and counting were performed in the 329 building in the 300 Area.

The samples were prepared for counting following PNL-ALO-105. This procedure covers the preparation of solid samples for GEA. The samples were either prepared in a glass scintillation vial (which contained 13 to 16 g of soil and were counted on shelf 2 or shelf 5 of the sample holder for diode L) or in a 2/5 dram polyvial (which contained about 1.0 g of soil and counted on shelf 4 of the sample holder for diode L).

The aliquots taken used the "as received" soil; i.e., the soil was not dried. A percent solids determination has been performed as part of Task 2 and 4 and the results for the three samples are found elsewhere in this report. Therefore, one can convert the reported values to a dry weight basis if desired.

The samples were then analyzed by gamma-ray spectroscopy to determine the quantities of Co-60, Ru-106, and Cs-137 present. This portion of the work was performed following PNL-ALO-464 and PNL-ALO-470. The samples were counted on diode L which is a lithium-drifted germanium [Ge(Li)] detector. Table 10 presents the measured quantities for Co-60, Ru-106, and Cs-137 in the soils. The detection limits quoted are calculated as if the isotope was present at a level that is 2.5 times the square root of twice the average background. For the "hotter" samples, at least two counts were taken of each sample. A short count of 100 to 1000 seconds was taken to obtain a peak for Cs-137 that did not overflow the analyzer and a longer count was taken to obtain better sensitivity for Ru-106 and Co-60 but the peak for Cs-137 may have overflowed the analyzer. Therefore, both counts were used to obtain the recommended values for each isotope in each sample.

Two aliquots of the following samples were measured: 92-04736 (SDG #15), 92-05352 (SDG #16), 92-05490 (SDG #16) and 92-05954 (SDG #17). For Cs-137, the counting error is around 1.0% for each sample. The mean and standard deviation for each pair of samples was calculated. The average of the standard deviations for each pair was then determined to be 8.5%. This is well below the required precision for soils of ± 35%.

The detection limits were determined from sample 92-05954-L-5 which is an empty glass scintillation vial counted on shelf 2 of the sample holder for diode L. The average weight of the preceding 18 samples prepared in the glass scintillation vials was determined to be 14.7 g. This weight was assumed to allow the calculation of detection limits in pCi/g for Co-60, Ru-106, and Cs-137. The detection limits at one sigma are 0.08, 0.8 and 0.06 pCi/g, respectively.

Table 10: Gamma Energy Analysis of Soils

(Radionuclide activity in pCi/g[a])

WHC Sample ID	Sample ID	Collection Date	Weight (g)	Co-60	Ru-106	Cs-137
B01S97	92-04734-L-1*	3/2/92 10:12	16.433	1.11 ± 0.09	<34	(8.52 ± 0.09) X 10^4
B01S99	92-04735-L-1*	3/3/92 10:15	1.0717	<70	<4000	(3.34 ± 0.06) X 10^6
B01SB1	92-04736-L-1*	3/3/92 10:15	12.074	<350	<3000	(2.88 ± 0.03) X 10^6
B01SB1	92-04736-L-2*	3/3/92 10:15	1.0962	<40	<3300	(2.75 ± 0.01) X 10^6
B01SB3	92-04968-L-1*	3/5/92 10:40	15.846	<3	<210	(1.66 ± 0.02) X 10^5
B01SB7	92-05102-L-1*	3/10/92 08:33	15.275	<0.14	<0.47	<0.20
B01SB2	92-05150-L-1*	3/11/92 08:50	1.2004	<480	<2500	(4.50 ± 0.05) X 10^6
B01SC7	92-05270-L-1*	3/13/92 08:55	1.0658	<830	<15000	(1.29 ± 0.01) X 10^7
B01SC7	92-05271-L-1*	3/16/92 14:00	14.838	<3	<190	(4.12 ± 0.04) X 10^5
B01SD9	92-05352-L-1*	3/19/92 14:32	15.179	0.70 ± 0.20	<16	(9.12 ± 0.28 X 10^2
B01SD9	92-05352-L-2*	3/19/92 14:32	16.621	0.596 ± 0.006	<2	(9.47 ± 0.09) X 10^2
B01SD7	92-05353-L-1*	3/19/92 10:20	14.404	<2	<140	(5.44 ± 0.03) X 10^4
B01SF7	92-05490-L-1*	3/23/92 14:16	14.144	<210	<14000	(2.35 ± 0.02) X 10^6
B01SF7	92-05490-L-2*	3/23/92/14:16	0.9269	<90	<5300	(1.84 ± 0.02) X 10^6
B01SF9	92-05491-L-1*	3/25/92 09:04	14.794	<0.2	<9	(3.02 ± 0.03) X 10^3
B01SG3	92-05492-L-1*	3/25/92 13:26	14.464	<0.3	<3	2.50 ± 0.36
B01SG9	92-05634-L-1*	3/30/92 10:02	13.680	3.1 ± 0.1	<30	(5.64 ± 0.06) X 10^3
B01SH3	92-05690-L-1*	3/31/92 09:03	13.214	<0.3	<3.5	4.39 ± 0.40
B01SG7	92-05633-L-1*	3/26/92 10:45	16.738	1.81 ± 0.17	<44	(1.25 ± 0.01) X 10^4
B01SH7	92-05877-L-1	4/1/92 10:21	1.0683	62 ± 10	<2700	(4.41 ± 0.04) X 10^6

Table 10: Gamma Energy Analysis of Soils

(Radionuclide activity in pCi/g[a])

WHC Sample ID	Sample ID	Collection Date	Weight (g)	Co-60	Ru-106	Cs-137
B01SB4	92-05953-L-1	4/8/92 08:34	1.2256	<90	<5600	$(2.88 \pm 0.03) \times 10^6$
B01SC8	92-05954-L-1	4/8/92 10:21	15.268	50 ± 2	<690	$(1.37 \pm 0.01) \times 10^6$
B01SC8	92-05954-L-2	4/8/92 10:21	0.8959	64 ± 7	<2200	$(1.60 \pm 0.03) \times 10^6$
B01SC8	92-05954-L-5	4/8/92 10:21	14.7	<0.19	<1.9	<0.14

[a] The one sigma uncertainties are based on counting statistics. All "<" values are detection limits associated with each "not detected" analysis. The detection limits are calculated as if the isotope was present at a level that is 2.5 times the square root of twice the average background.

* Sample not in this SDG but reported for QC and analytical batch purposes.

TOTAL ALPHA ANALYSIS RESULTS

The three samples from SDG #17, along with the corresponding QC samples, were analyzed for gross alpha content in June 1992 in two batches. They were analyzed in conjunction with samples from SDG #18 and #19. Approximately 0.5 to 5 grams were taken for analysis depending on the sample activity level. The samples were leached per procedure PNL-ALO-106, "Acid Digestion for Preparation of Samples for Radiochemical Analysis." Aliquots of the leachate solutions and blanks were taken to perform the alpha analysis. Total alpha analysis proceeded per Procedure PNL-ALO-460, "Source Preparation for Gross Alpha Analysis", and PNL-ALO-461, "Alpha Counting Procedure." All work was performed in building 329 and 329T5 of the 300 area.

Due to the high activity of the QC samples, the matrix spike could not be added to the soil directly. This would have required a very small soil sample be taken and a large alpha spike added. A small sub-sample size would not necessarily be representative of the whole sample due to sample inhomogeneity. Therefore, a matrix spike recovery could not be accurately calculated. To circumvent this problem, an alpha spike was added to an aliquot of a sample leachate; error due to sampling problems is no longer a factor in determining the matrix spike recovery. This spiking protocol tests all analytical parameters except the leaching process.

The minimum QC criteria were met for the analysis of this SDG. In the first batch, the matrix spike resulted in 91% recovery and the blank spike resulted in 102% recovery. In the second batch, the matrix spike resulted in 80% recovery and the blank spike resulted in 81% recovery. These values indicate that an average batch bias of -12% was obtained for this SDG. The precision, estimated by the relative percent difference of the duplicates, are 8% and 34% (first and second batches, respectively). The large RPD obtained for the second batch is within the specification of ±35%. It can be somewhat deceptive in that it actually represents an average result of 18 ± 3 pCi/g; for a screen, this is completely satisfactory. The largest contribution to the imprecision is estimated to come from the sample heterogeneity. Alpha activity in the blank was not detectable at a level of 1 pCi/g.

9713508.2003

A column in the table indicates the normalized blank value. This is a normalization relative to the actual sample size taken for analysis. Due to extremely high activity (in some cases) a very small aliquot of the soil leachate may be analyzed. This would result in a relative blank value quite different from the blank based on a 1 gram equivalent sample size. In the case of a slightly positive blank value, it will help put the sample results in proper perspective, and so it is provided as a courtesy.

Results are reported per received sample weight (not corrected for weight percent solids). No hold times are defined for total alpha analysis.

TABLE 11: TOTAL ALPHA ANALYSIS DATA FOR TASK 2&4
SDG #17

WHC Sample ID	Sample #	Sample Type	Alpha (pCi/g)	+/- 1 sigma*	Normalized Blank**	RPD	Spike Conc. (pCi/g)	+/- 1 sigma*	Spike % Rec.
First Batch									
B01SH7	92-05877-A-1a	Soil	2554	231	<40				
B01SB4	92-05953-A-1a	Soil	1792	153	<20				
	92-06727-A-1a+	Soil	82	14	<8				
	92-06727-A-2a+	Duplicate	75	14	<8	8			
	92-06727-A-3a+	Matrix Spike	2085	152	<20		2206	47	91
	92-06727-A-4a+	Blank Spike	24.4	1.8	<1		24	0.2	102
	92-06727-A-5a+	Method Blank	<1						
Second Batch									
B01SC8	92-05954-A-1a	Soil	969	117	<12				
	92-06726-A-1a+	Soil	15.3	2.9	<1				
	92-06726-A-2a+	Duplicate	21.5	3.3	<1	34			
	92-05352-A-3a+	Matrix Spike	40.5	2.7	<1		50.7	0.6	80
	92-05352-A-4a+	Blank Spike	19.3	1.5	<0.2		23.89	0.15	81
	92-05352-A-5a+	Method Blank	<1						

* One sigma uncertainties are based on propagation of mass, volume, and counting uncertainties.

** Blank value normalized to sample size analyzed. See narrative for details.

\+ Not part of current SDG but reported for QC purposes as it relates to the duplicate and matrix spike.

9713508.2004

TOTAL BETA ANALYSIS RESULTS

Three samples from SDG #17, along with the corresponding QC samples, were analyzed for gross beta content in June 1992 in two batches. The samples were run in conjunction with samples from SDG #18 and #19. A nominal 0.5 to 5 grams were taken for analysis, in accordance with the sample activity levels. The samples were leached per procedure PNL-ALO-106, "Acid Digestion for Preparation of Samples for Radiochemical Analysis." Aliquots of the leachate solutions and blanks were taken to perform the beta analysis. Total beta analysis proceeded per Procedure PNL-ALO-462, "Source Preparation for Gross Beta Analysis", and PNL-ALO-463, "Beta Counting Procedure." Detectors are calibrated relative to Sr-90/Y-90. All work was performed in building 329 and 329T5 of the 300 area.

Due to the high activity of the samples, the matrix spike could not be added to a soil aliquot directly. This would have required a very small soil sample be taken and a large beta spike added. A small sub-sample size would not necessarily be representative of the whole sample due to sample inhomogeneity. Therefore, a matrix spike recovery could not be accurately calculated. To circumvent this problem, a beta spike was added to an aliquot of a sample leachate; error due to sampling problems is no longer a factor in determining the matrix spike recovery. This spiking protocol tests all analytical parameters except the leaching process.

The analysis for total beta activity proceeded smoothly; all QC criteria were met. In the first batch, the matrix spike resulted in 99% recovery and the blank spike resulted in 77% recovery. The second batch matrix spike resulted in 96% recovery, the blank spike resulted in 107% recovery. The average batch bias, based on the average spike recovery, is -5%. The precision, estimated by the relative percent difference of the duplicates, averages 9%.

Beta activity in the blanks was not detected at 10 pCi/g and 16 pCi/g for the first and second batches, respectively. Lower detection limits, specified in the TPP, could be obtained by counting on the low background beta detectors

9713508.2006

and analysis of a larger sample. However, this was not necessary due to the high level of sample activity.

The normalized blank value is a normalization relative to the actual sample size taken for analysis. Due to extremely high sample activity, a very small aliquot of the soil leachate is analyzed. This results in a relative blank value quite different from the blank based on a 1 gram equivalent sample size. In case of a positive blank value, it shows the worst case estimated relative contamination. The normalized blank is provided as a courtesy.

Results are reported per received sample weight (not corrected for weight percent solids). No hold times are defined for total beta analysis.

TABLE 12: TOTAL BETA ANALYSIS DATA FOR TASK 2&4
SDG #17

WHC Sample ID	Sample #	Sample Type	Beta (pCi/g)	+/- 1 sigma*	Normalized Blank**	RPD	Spike Conc. (pCi/g)	+/- 1 sigma*	Spike % Rec.
First Batch									
B01SH7	92-05877-A-1a	Soil	1.70E+07	6.5E+05	<2E+05				
B01SB4	92-05953-A-1a	Soil	1.45E+07	5.9E+05	<2E+05				
	92-06727-A-1a+	Soil	5.82E+06	2.7E+05	<2E+05				
	92-06727-A-2a+	Duplicate	5.06E+06	2.6E+05	<2E+05	14			
	92-06727-A-3a+	Matrix Spike	5.40E+04	1.7E+03	<5E+02		5.44E+04	2.9E+03	99
	92-06727-A-4a+	Blank Spike	7.37E+01	3.1E+00	<2E+00		9.61E+01	4.8E+00	77
	92-06727-A-5a+	Method Blank	<1E+01						
Second Batch									
B01SC8	92-05954-A-1a	Soil	9.32E+06	3.9E+05	<2E+05				
	92-06726-A-1a+	Soil	1.83E+04	6.0E+02	<3E+02				
	92-06726-A-2a+	Duplicate	1.75E+04	5.9E+02	<3E+02	4			
	92-06726-A-3a+	Matrix Spike	6.37E+02	1.1E+01	<3E+02		6.66E+02	1.9E+01	96
	92-05352-A-4a+	Blank Spike	2.56E+02	5.8E+00	<3E+00		2.39E+02	9.0E+00	107
	92-05352-A-5a+	Method Blank	<2E+01						

*One sigma uncertainties are based on propagation of mass, volume, and counting uncertainties.

**Blank value normalized to sample size analyzed. See narrative for details.

+ Included for QC purposes; sample is one of the duplicates from SDG 18.

9713508.2007

PLUTONIUM ANALYSIS RESULTS

Plutonium analyses were performed on samples from SDG #17 along with samples from SDG #18 and #19 and their corresponding QC samples. The analyses were split into two analytical batches based on activity level. For the SDG #17 samples in the first batch a nominal 1 gram sample was leached. For the second batch a nominal 1 gram sample was leached for B01SC8 and a nominal 5 gram sample for B067Z3 (the QC sample which is from SDG #18). An aliquot of each sample leachate was taken for analysis. This was necessary due to the relatively high level of activity in the samples. The leach procedure used was PNL-ALO-106, "Acid Digestion for Preparation of Samples for Radiochemical Analysis." Analysis was completed according to Procedures PNL-ALO-466, "Plutonium Separation", PNL-ALO-468, "Electroplating", and PNL-ALO-469, "Alpha Energy Analysis." All work was performed in building 329 and 329T5 in the 300 area.

All samples, after leaching, were spiked with Pu-242 tracer which was used to correct for the radiochemical yield of each individual sample. The Pu-239 recoveries for the blank spikes are excellent at 99% and 101% respectively for the two batches. These indicate an average batch bias of 0%. The target accuracy is ±25%. For the first batch the duplicate sample has a relative percent difference (RPD) of 0.8% for Pu-239+240 and 14% for Pu-238. The higher RPD for Pu-238 is consistent with the higher uncertainties of the individual Pu-238 measurements. For the second batch the duplicate sample contains a non-detectable activity of Pu-238 so a RPD could not be calculated for this isotope. For Pu-239 the RPD is 7%. All RPD's are within the project target of ±35% precision. No detectable amount of Pu-239+240 or Pu-238 was found in the blanks for either batch.

The results for the blanks and blank spikes are reported in pCi. In addition, the blank results have been normalized against the samples (based on actual mass analyzed after leaching, dilution and aliquoting) to provide a more meaningful value relative to each sample value.

The results are reported per received sample weight (not corrected for weight percent solids). No hold times are defined for plutonium analyses.

TABLE 13: PLUTONIUM ANALYSIS DATA FOR TASK 2&4
SDG #17

Client Sample ID	Program Sample ID	Sample Type	Pu-239+240 pCi/g	+/- 1 sigma	Pu-239+240 RPD	Pu-239+240 Normalized Blank*** pCi/g	Pu-238 pCi/g	+/- 1 sigma	Pu-238 RPD	Pu-238 Normalized Blank*** pCi/g	Pu-239 Spike pCi/sample	+/- 1 sigma	% Recovery
Batch #1													
B01SH7	92-05877-A-1c	Soil	1.20E+03	4.95E+01		<2.7 E+00	6.51E+01	6.08E+00		<1.8 E+00			
B01SB4	92-05953-A-1c	Soil	6.26E+02	2.24E+01		<1.4 E+00	5.55E+01	3.10E+00		<9.5 E-01			
B067Z5	92-06727-A-1c	Soil*	1.26E+02	4.73E+00		<5.6 E-01	1.70E+00	2.76E-01		<3.7 E-01			
B067Z5	92-06727-A-2c	Duplicate*	1.25E+02	4.66E+00	0.8	<5.4 E-01	1.95E+00	2.85E-01	14	<3.6 E-01			
	92-06727-A-5c	Blank**	<3.4 E-02				<2.4 E-02						
	92-06727-A-4c	Blank Spike**	4.76E+01	1.20E+00			<5.7 E-03				4.81E+01	3.00E-01	99
Batch #2													
B01SC8	92-05954-A-1c	Soil	4.32E+02	1.39E+01		<2.0 E-01	3.42E+01	1.21E+00		<2.0 E-01			
B067Z3	92-06726-A-1c	Soil*	9.60E-02	2.11E-02		<2.4 E-02	<8.9 E-03			<2.4 E-02			
B067Z3	92-06726-A-2c	Duplicate*	1.03E-01	2.20E-02	7	<2.4 E-02	<1.1 E-02		NA	<2.4 E-02			
	92-05352-A-5c	Blank**	<1.3 E-02				<1.2 E-02						
	92-05352-A-4c	Blank Spike**	1.21E+01	3.11E-01			<1.9 E-03				1.20E+01	1.00E-01	101

* Not in this SDG. Reported for QC purposes only.
** Reported in pCi. Not in this SDG. Reported for QC purposes only.
*** Blank value when normalized to actual sample size analyzed.
One sigma uncertainties are the propagated error of individual measurements.
Using a 1 gram sample size results in a detection limit of approximately 3 E-2 pCi/g for Pu-239+240 and 2 E-2 pCi/g for Pu-238.

97135082.2009

STRONTIUM ANALYSIS RESULTS

Sr-90 analyses of SDG #17 were performed in two batches along with samples from SDG #18 and #19. A nominal 1 gram sample size was taken for analysis. The QC samples were from SDG #18. The samples were leached per procedure PNL-ALO-106, "Acid Digestion for Preparation of Samples for Radiochemical Analysis." The sample leachates were diluted serially and aliquots of appropriate activity were taken for Sr-90 analysis. The strontium analysis continued per Procedure PNL-ALO-465, "Strontium-90 Analysis (Oxalate-Nitric Acid Method)", and PNL-ALO-463, "Beta Counting Procedure." All analytical work and calculations were performed in building 329 and 329T5 of the 300 area.

Due to the high Sr-90 activity of the samples, the matrix spike could not be added to a soil aliquot directly. This would have required a very small soil sample be taken and a large Sr-90 spike quantity added. A small sub-sample size would not necessarily be representative of the whole sample due to sample inhomogeneity, and would most probably cause an inaccurate matrix spike recovery calculation. To circumvent this problem, a Sr-90 spike was added to an aliquot of a sample leachate; error due to sampling problems is no longer a factor in determining the matrix spike recovery. This spiking protocol tests all analytical parameters except the leaching process. Previous experience with spiked soils indicate no problems should be anticipated with the dissolution and recovery of Sr with the leaching process.

The analysis for Sr-90 proceeded smoothly. The yield spike of a known blank soil was used to determine the batch yield. The yields of the two batches agreed well: 91% and 95%. All samples of each batch are calculated on the basis of the ratio of the corresponding yield spike recovery normalized to 100% chemical recovery.

The blank spike consisted of 8M HNO_3 and resulted in 108% (first batch) and 122% (second batch) recoveries. A matrix spike was run with the first batch which consisted of an aliquot of B067Z5 (SDG #18) soil leachate spiked with Sr-90 and it resulted in 106% recovery. The second batch matrix spike

sample consisted of an aliquot of B067Z3 (SDG #18) soil leachate and resulted in 91% recovery of Sr-90. Based on the blank and matrix spike recoveries, the overall batch bias is calculated to be +7%. The overall precision, estimated by the relative percent difference of the duplicates, is ± 10%.

Sr-90 activity in the blank was detected in both batches (74 pCi/g and 10 pCi/g). These blank values are generally the result of contamination from the samples themselves into the blank. It is believed to occur during the final plating stage. Because the samples themselves contain high activity, an insignificant fraction of a sample contaminating the blank can cause a large signal in the blank. The blank contamination is insignificant relative to the sample values (see the normalized blank in the accompanying table).

The normalized blank value is a normalization relative to the actual sample size taken for analysis. Due to extremely high sample activity, a very small aliquot of the soil leachate is analyzed. This results in a relative blank value quite different from the blank based on a 1 gram equivalent sample size. In this case of a positive blank value, it shows the worst case estimated relative contamination, that is, if contamination occurred at the beginning, during the sampling or leaching process.

The Sr-90 analysis does not correct for the presence of Sr-89; all activity is assumed to be from Sr-90. Results are reported per received sample weight (not corrected for weight percent solids). No hold times are defined for Sr-90 analysis.

TABLE 14: STRONTIUM-90 ANALYSIS DATA FOR TASK 2 & 4
SDG #17

WHC Sample ID	Sample #	Sample Type	Strontium (pCi/g)	+/- 1 sigma*	Normalized Blank***	RPD	Spike Conc. (pCi/g)	+/- 1 sigma*	Spike % Rec.	Normalized % Yield**
First Batch										
B01SH7	92-05877-A-1b	Soil	5.80E+06	5.2E+05	2.7E+05					
B01SB4	92-05953-A-1b	Soil	6.54E+06	5.7E+05	2.9E+05					
	92-06727-A-1b+	Soil	2.36E+06	2.2E+05	2.2E+05					
	92-06727-A-2b+	Duplicate	2.60E+06	2.3E+05	2.1E+05	10				
	92-06727-A-3b+	Matrix Spike	2.34E+03	2.0E+02	6.7E+01		2.20E+03	1.2E+02	106	
	92-06727-A-3b+	Yield Spike	Used to determine Batch yield							91
	92-06727-A-4b+	Blank Spike	1.29E+02	1.1E+01	7.4E+01		1.19E+02	6.0E+00	108	
	92-06727-A-5b+	Method Blank	7.38E+01	7.7E+00						
Second Batch										
B01SC8	92-05954-A-1b	Soil	3.81E+06	3.3E+05	2.8E+04					
	92-06726-A-1b+	Soil	8.85E+03	7.1E+02	1.0E+02					
	92-06726-A-2b+	Duplicate	8.07E+03	6.6E+02	1.1E+02	9				
	92-06726-A-3b+	Matrix Spike	2.99E+04	2.3E+03	1.0E+02		2.37E+04	1.0E+03	91	
	92-06726-A-3b+	Yield Spike	Used to determine Batch yield							95
	92-06726-A-4b+	Blank Spike	1.45E+02	1.3E+01	2.1E+00		1.19E+02	4.5E+00	122	
	92-06726-A-5b+	Method Blank	1.03E+01	2.5E+00						

*One sigma uncertainties are based on propagation of mass, volume, and counting uncertainties.

**All Sr-90 analyses are calculated on the basis of their ratio to the matrix spike recovery which has been normalized to 100% chemical recovery.

***Blank value when normalized to actual sample mass analyzed.

+ This sample was not part of this SDG but it was one of the duplicates and is included for QC purposes.

9713508.2012

TRITIUM ANALYSIS RESULTS

Three soil samples from SDG #17, and their corresponding QC samples were prepared and analyzed according to procedures PNL-ALO-441, "Radionuclide Separation and Analysis Procedure for Tritium", and PNL-ALO-443, "Liquid Scintillation Counting Procedure for Tritium." Two other SDG groups, #18 and #19, were also analyzed with this set. The leaching, distillation and counting were performed in building 329 in the 300 area.

A nominal 5 grams were initially sub-sampled for leaching. After leaching and a double distillation, the aliquots counted are equivalent to approximately 0.4 g. The QC sample for this set comes from SDG #19, 92-06964. A pre-leached sample (92-06964) is used to determine the batch yield or recovery correction factor (F_s). The recovery correction factor is applied to each analytical result in the batch. The batch yield is 87.5%. This is consistent with past experience.

The duplicate values of the QC sample are 7.9 and <4 pCi/g. Because these values are respectively close to and at the detection limit, the relative percent difference cannot be calculated. At levels of up to about 5 times the detection limit, precision of any analytical method tends to be poor. The blank spike consists of a pre-leached sample (92-06964, duplicate) with a known amount of tritium spike added. It is essentially a duplicate of the batch yield sample described above. The blank spike recovery of 86% is obtained. The matrix spike sample consists of an as received sample (92-06964) to which a known tritium spike quantity is added. The matrix spike shows good recovery at 91%. Results of averaging both blank spike and matrix spike recoveries is 89%, indicating an average -11% batch bias.

The blank result, normalized to a 5 gram sample size, shows tritium is not detected at 4 pCi/g.

This procedure was conducted according to the corrective actions described in the deficiency report, 91-105. It allows us to operate more

9713508.2011

effectively for the soil analysis and allows for better equation application to the procedure. It has been in effect for almost one year.

The sample results are reported per received sample weight (not corrected for weight percent solids). No hold times are defined for tritium analysis.

TABLE 15: TRITIUM ANALYSIS DATA FOR TASKS 2 & 4
TRITIUM SDG #17

WHC Sample ID	Sample #	Sample Type	Tritium (pCi/g)	+/- 1 sigma*	RPD	Conc. (pCi/g)	+/- 1 sigma*	Spike % Rec.	RCF Fs**
B01SH7	92-05877-K-1	Soil	31.8	5.3					
B01SB4	92-05953-K-1	Soil	38.4	1.8					
B01SC8	92-05954-K-1	Soil	35.0	3.0					
	92-06964-K-1+	Soil	7.9	1.3					
	92-06964-K-2+	Duplicate	< 4		na				
	92-06964-K-4+	Matrix Spike	707	15		766	14	91	
	92-06964-K-4+	Blank Spike	3618	75		4193	75	86	
	92-06964-K-5+	Method Blank	< 4						
	92-06964-K-4+	Yield spike to determine Fs							87.5

* One sigma uncertainties are based on propagation of mass, volume, and counting uncertainties.

**Recovery Correction Factor, Fs.

na: not applicable

+ Included for QC purposes.

9713508.2015

TOTAL ORGANIC CARBON ANALYSIS RESULTS

Samples 92-05877 (B01SH7), 92-05953 (B01SB4), and 92-05954 (B01SC8) from SDG #17 were prepared by procedure PNL-ALO-380, Rev. 0, "Determination of Carbon in Solids Using the Coulometrics Carbon Dioxide Coulometer." The procedure methodology is consistent with SW 846 method 9060. PNL-ALO-380 defines the operation of the instrument used as well as the analysis of the sample. SW 846 method 9060 leaves the option for the analyst to follow the manufacturer's instrument instructions for calibration, analysis procedure, and calculations.

Blank Determination

With the Coulometrics TOC system, an average (daily) blank must be determined prior to calibration check of the instrument and analysis of samples. The major carbon source in the blank is CO_2 adsorbed on the sample boat and ladle. The blank is obtained by removing the quartz ladle and sample boat (platinum or porcelain) from the furnace tube and exposing them to air. These parts are then placed back into the furnace and carbon analysis is performed on this blank. This procedure is repeated until consecutive blank values differ by no more than 0.5 μg of total organic carbon for every 10 minutes of analysis time. As there is no sample preparation prior to analysis, this instrument blank is also considered to be the methods blank when determining TOC by this method.

Detection Limits

The blank thus obtained has a direct effect on the quantification limit for each sample as this value must be subtracted from each sample value determined. However, this blank value is not an indicator of instrument sensitivity, and should not be considered as an indication of the true instrument detection limit. If the instrument were operated in a carbon-free atmosphere, a lower blank value could be observed. It is not possible to determine an absolute instrument detection limit (i.e., a measurement of instrument sensitivity) under current laboratory operating conditions. For

purposes of this report, the daily blank value is used as the lower quantification limit for the analyses. Reported results indicate that sample organic carbon levels are above this method quantification limit (instrument background carbon levels).

An average "method detection limit" for this analytical method may be estimated from the absolute difference between consecutive blank values. The "method detection limit," defined as three times this difference, is approximately 3 μg total organic carbon. The method detection limit expressed in concentration terms would be dependent on the sample size analyzed.

Standard Analysis

A Kodak glucose standard was analyzed in duplicate as an initial one point calibration of the instrument. The manufacturer's manual states to use a single point calibration of the instrument as the instrument exhibits a linear response. Each standard determination consisted of a 35 minute analysis and reported results are corrected with the corresponding average 35 minute blank value. Initial recoveries of the same glucose standard on 4/9/92 were 99.7% & 101%, with an average of 100%, and a relative percent difference (RPD) of 1.3%. Standard recoveries on 4/22/92 were 99.2% and 100%, with an average of 99.8%, and a RPD of 1.2%. Based on duplicate analysis results, estimated precision from this set of data is $\pm$ 1.3% relative, with a bias (accuracy) of 0% on the average for 4/9/92 analyses, and $\pm$ 1.2% relative with a bias of - 0.2% on the average for 4/22/92 analyses. Reported sample values are corrected for the corresponding average initial standard recoveries.

Two additional glucose standard analyses were performed after sample analysis on 4/9/92. Recoveries were 91.5% (standard 2) and 100% (standard 3). Recovery for standard 2 (91.5%) was low in comparison to the initial standard recoveries (100% average) and recovery of standard 3. It is believed this deviation is most likely due to a weighing or sample handling error, and not instrument error. It is believed as such for the following three reasons: 1) a very small weighing error (0.4 mg) would be required to show the associated recovery decrease, 2) sample handling techniques, or laboratory/hood air flow

could result in a small loss of standard, and 3) recovery of the 2nd standard (standard 3) was at par with initial recoveries (~100%).

Sample Analysis

All samples were first screened, and then analyzed in duplicate for 35 minutes at ~500 °C. The RPD between duplicate analyses ranged from 2.3% to 18.2%. Replicate analysis deviation is mostly attributed to the inherent heterogeneity of the soil samples received, and the small sample sizes analyzed. Due to the small sample size required (2 - 100+ mg), a selective sub-sample of smaller soil particles is necessary. The very nature of this sample selection process may bias reported carbon results and increase deviation between replicate analyses.

Samples analyzed on 4/9/92 exhibited a trend of decreasing TOC content followed by a low initial post-sample glucose standard recovery. This could possibly suggest a decreasing instrument sensitivity for CO_2 evolved from the sample. This is not believed to be the case, though. Screened sample behavior exhibited very similar responses for the individual samples while being analyzed in a slightly different order. Furthermore, it is believed the low standard recovery (standard 2) was probably due to a weighing or sample handling error, as explained in the "standard analysis" section.

Reported sample organic carbon levels are not adjusted for moisture content of the soil matrix.

Hold Times

The Environmental Protection Agency (EPA) hold time for Total Organic Carbon Analysis in soils is defined at 14 days from the date of sampling. The hold times were met for these samples.

TABLE 16: TOTAL ORGANIC CARBON ANALYSIS DATA FOR TASKS 2 & 4
SDG #17

Soil Samples

WHC Sample	PNL ALO #	Sample Type	Sample Wt. g	ug C Results	ug C in Sample	mgC/Kg Sample	RPD Dups	% Rec.* Standard	Date Received	Date Analyzed
B01SH7	92-05877-1	Sample	0.05550	33.86	24.16	435	2.3		4-06-92	4-09-92
B01SH7	92-05877-2	Duplicate	0.04924	30.61	20.91	425			4-06-92	4-09-92
B01SH7	92-05877-3	Standard	0.00516	2068.14	2058.44		1.3	99.7		4-09-92
B01SH7	92-05877-3	Std Dup	0.00498	2029.49	2019.79			101		4-09-92
B01SH7	92-05877-4	Blank		9.70						4-09-92
		+Standard 2	0.00521	1917.12	1907.42			91.5		4-09-92
		++Standard 3	0.00664	2680.29	2670.59			100		4-09-92
B01SB4	92-05953-1	Sample	0.06321	32.43	24.30	385	5.1		4-13-92	4-22-92
B01SB4	92-05953-2	Duplicate	0.05549	28.41	20.28	366			4-13-92	4-22-92
B01SC8	92-05954-1	Sample	0.06628	41.99	33.86	532	18.2		4-13-92	4-22-92
B01SC8	92-05954-2	Duplicate	0.06376	36.38	28.25	444			4-13-92	4-22-92
B01SC8	92-05954-3	Standard	0.00582	2317.09	2308.96		1.2	99.2		4-22-92
B01SC8	92-05954-3	Std Dup	0.00412	1662.41	1654.28			100		4-22-92
B01SC8	92-05954-4	Blank		8.13						4-22-92

* Based on standard carbon content of 40.0% by weight.

+ Standard 2: 1st post-sample standard analyzed

++ Standard 3: 2nd post-sample standard analyzed

Total Organic Carbon by Procedure PNL-ALO-380, on Instrument WA92040,
325 Bldg., rm 701. Data reported from LRB 52821, pp 97-102 & 104-105.

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VOLATILE ORGANIC ANALYSIS RESULTS

SAMPLE ANALYSIS REPORTED

Analysis of three soil samples for volatile organic compounds by gas chromatography/mass spectrometry (GC/MS) is the subject of this report.

SAMPLE DESCRIPTION

Sample ID	ACL Lab Number	Date received
B01SH7	92-05877	04/06/92
B01SB4	92-05953	04/13/92
B01SC8	92-05954	04/13/92

The samples, B01SH7, B01SB4, and B01SC8 were received on the dates specified above in the 325 building. These samples are referred to as SDG #17.

SAMPLE PREPARATION

The preparation and analysis followed EPA-CLP SOW 2/88 procedures for the analysis of volatile compounds in soils.

ANALYSIS METHOD

- GC/MS procedure: PNL-ALO-335.
- GC/MS instrumentation: HP-5890/5970 GC/MS (WB46864).
- GC/MS location: Lab 327-A, 325 building

QUALITY CONTROL

Quality control procedures specified for this method were followed. The quality assurance performance requirements are summarized as follows:

Form	Information	Comments
2B	Surrogate Recovery	Meets all requirements.
3B	MS/MSD Recovery	Meets all requirements.

4A	Method Blank Summary	Meets all requirements.
5A	Tune/Mass Calibration	Meets all requirements.
6A	Initial Calibration	5 point calibration. Meets all requirements.
7A	Daily Calibration	Meets all requirements.
8A	Internal Standards	Meets all requirements.

RESULTS

CLP Target Compounds: The attached 1A Forms show that volatile target compounds, **Acetone and 2-Butanone**, were observed in the BLANK. However, they were below the Contract Required Quantitation Limits, (CRQLs). **Acetone** was observed, below the CRQL, in 92-05877DUPE, 92-05953DUPE, 92-05954, and 92-05954DUPE.

No target compounds were observed in 92-05953 or 92-05877.

In summary, no CLP Target Compounds were observed in the samples 92-05953, 92-05954, and 92-05877 above the quantitation limits.

The following defines the qualifiers, Q-flags, in the Form 1's:

"Q" Flag	Definition
U	Indicates the compound was analyzed for but not detected, the U-flagged concentration number is the CRQL.
J	Indicates an estimated value for the target or tentatively identified compounds, spectra meet criteria but response is below the CRQL for the target compounds.
B	Compound was found in the blank.
X	Indicates compound was manually deleted because all requirements were not met.

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D	Analysis was performed on a diluted sample.
E	Indicates that Quantitation was outside the calibration range.

Tentatively Identified Compounds: As shown in the attached 1E forms, there were no non-CLP target Compounds in the Blank.

In sample 92-05953 and 92-05953DUPE, a series of late eluting Unknown Alkanes were observed at the ppm level. Sample 92-05954 and 92-05954DUPE also contained Unknown Alkanes, but at a much lower level. These late eluting and coeluting alkanes are likely to include dodecane, tridecane, tetradecane, and branched alkanes.

The sample 92-05877 and 92-05877DUPE contained an Unknown. This Unknown is likely to be either a cyclic alkane or an alkene.

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1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

92-05954

Lab Name: BATTELLE-PNL Contract: ------

Lab Code: ------ Case No.: ----- SAS No.: ----- SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: B01SC8

Sample wt/vol: 5.390 (g/mL) G Lab File ID: >PB504

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec. 2.1 Date Analyzed: 4/15/92

Column: (pack/cap) CAP Dilution Factor: 1.00000

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
74-87-3	Chloromethane	9.	U
74-83-9	Bromomethane	9.	U
75-01-4	Vinyl Chloride	9.	U
75-00-3	Chloroethane	9.	U
75-09-2	Methylene Chloride	5.	U
67-64-1	Acetone	4.	JB
75-15-0	Carbon Disulfide	5.	U
75-35-4	1,1-Dichloroethene	5.	U
75-34-3	1,1-Dichloroethane	5.	U
540-59-0	1,2-Dichloroethene (total)	5.	U
67-66-3	Chloroform	5.	U
107-02-2	1,2-Dichloroethane	5.	U
78-93-3	2-Butanone	9.	U
71-55-6	1,1,1-Trichloroethane	5.	U
56-23-5	Carbon Tetrachloride	5.	U
108-05-4	Vinyl Acetate	9.	U
75-27-4	Bromodichloromethane	5.	U
78-87-5	1,2-Dichloropropane	5.	U
10061-01-5	cis-1,3-Dichloropropene	5.	U
79-01-6	Trichloroethene	5.	U
124-48-1	Dibromochloromethane	5.	U
79-00-5	1,1,2-Trichloroethane	5.	U
71-43-2	Benzene	5.	U
10061-02-6	trans-1,3-Dichloropropene	5.	U
75-25-2	Bromoform	5.	U
108-10-1	4-Methyl-2-pentanone	9.	U
591-78-6	2-Hexanone	9.	U
127-18-4	Tetrachloroethene	5.	U
79-34-5	1,1,2,2-Tetrachloroethane	5.	U
108-88-3	Toluene	5.	U
108-90-7	Chlorobenzene	5.	U
100-41-4	Ethylbenzene	5.	U
100-42-5	Styrene	5.	U
133-02-7	Xylene (total)	5.	U

9713508.2024

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

92-05954

Lab Name: BATTELLE-PNL Contract: ------

Lab Code: ------ Case No.: ----- SAS No.: ----- SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: B01SC8

Sample wt/vol: 5.390 (g/mL) G Lab File ID: >PB504

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec. 2.1 Date Analyzed: 4/15/92

Column: CAP Dilution Factor: 1.00000

Number TICs found: 1

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.	Unknown Alkanes	29.5-35.	260.	J
2.				
3.				
4.				
5.				
6.				
7.				
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30.				

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1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

92-05877

Lab Name: BATTELLE-PNL Contract: ------

Lab Code: ------ Case No.: ----- SAS No.: ----- SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: B01SH7

Sample wt/vol: 5.188 (g/mL) G Lab File ID: >PB508

Level: (low/med) LOW Date Received: 04/06/92

% Moisture: not dec. 3.7 Date Analyzed: 4/15/92

Column: (pack/cap) CAP Dilution Factor: 1.00000

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
74-87-3	Chloromethane	10.	U
74-83-9	Bromomethane	10.	U
75-01-4	Vinyl Chloride	10.	U
75-00-3	Chloroethane	10.	U
75-09-2	Methylene Chloride	5.	U
67-64-1	Acetone	10.	U
75-15-0	Carbon Disulfide	5.	U
75-35-4	1,1-Dichloroethene	5.	U
75-34-3	1,1-Dichloroethane	5.	U
540-59-0	1,2-Dichloroethene (total)	5.	U
67-66-3	Chloroform	5.	U
107-02-2	1,2-Dichloroethane	5.	U
78-93-3	2-Butanone	10.	U
71-55-6	1,1,1-Trichloroethane	5.	U
56-23-5	Carbon Tetrachloride	5.	U
108-05-4	Vinyl Acetate	10.	U
75-27-4	Bromodichloromethane	5.	U
78-87-5	1,2-Dichloropropane	5.	U
10061-01-5	cis-1,3-Dichloropropene	5.	U
79-01-6	Trichloroethene	5.	U
124-48-1	Dibromochloromethane	5.	U
79-00-5	1,1,2-Trichloroethane	5.	U
71-43-2	Benzene	5.	U
10061-02-6	trans-1,3-Dichloropropene	5.	U
75-25-2	Bromoform	5.	U
108-10-1	4-Methyl-2-pentanone	10.	U
591-78-6	2-Hexanone	10.	U
127-18-4	Tetrachloroethene	5.	U
79-34-5	1,1,2,2-Tetrachloroethane	5.	U
108-88-3	Toluene	5.	U
108-90-7	Chlorobenzene	5.	U
100-41-4	Ethylbenzene	5.	U
100-42-5	Styrene	5.	U
133-02-7	Xylene (total)	5.	U

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1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

92-05877

Lab Name: BATTELLE-PNL Contract: ------

Lab Code: ------ Case No.: ----- SAS No.: ----- SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: B01SH7

Sample wt/vol: 5.188 (g/mL) G Lab File ID: >PB508

Level: (low/med) LOW Date Received: 04/06/92

% Moisture: not dec. 3.7 Date Analyzed: 4/15/92

Column: CAP Dilution Factor: 1.00000

Number TICs found: 1

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.	Unknown	30.05	8.	J
2.				
3.				
4.				
5.				
6.				
7.				
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29.				
30.				

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1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

92-05953

Lab Name: BATTELLE-PNL Contract: ------

Lab Code: ------ Case No.: ----- SAS No.: ----- SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: B01SB4

Sample wt/vol: 5.201 (g/mL) G Lab File ID: >PB510

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec.2.3 Date Analyzed: 4/15/92

Column: (pack/cap) CAP Dilution Factor: 1.00000

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
74-87-3	Chloromethane	10.	U
74-83-9	Bromomethane	10.	U
75-01-4	Vinyl Chloride	10.	U
75-00-3	Chloroethane	10.	U
75-09-2	Methylene Chloride	5.	U
67-64-1	Acetone	10.	U
75-15-0	Carbon Disulfide	5.	U
75-35-4	1,1-Dichloroethene	5.	U
75-34-3	1,1-Dichloroethane	5.	U
540-59-0	1,2-Dichloroethene (total)	5.	U
67-66-3	Chloroform	5.	U
107-02-2	1,2-Dichloroethane	5.	U
78-93-3	2-Butanone	10.	U
71-55-6	1,1,1-Trichloroethane	5.	U
56-23-5	Carbon Tetrachloride	5.	U
108-05-4	Vinyl Acetate	10.	U
75-27-4	Bromodichloromethane	5.	U
78-87-5	1,2-Dichloropropane	5.	U
10061-01-5	cis-1,3-Dichloropropene	5.	U
79-01-6	Trichloroethene	5.	U
124-48-1	Dibromochloromethane	5.	U
79-00-5	1,1,2-Trichloroethane	5.	U
71-43-2	Benzene	5.	U
10061-02-6	trans-1,3-Dichloropropene	5.	U
75-25-2	Bromoform	5.	U
108-10-1	4-Methyl-2-pentanone	10.	U
591-78-6	2-Hexanone	10.	U
127-18-4	Tetrachloroethene	5.	U
79-34-5	1,1,2,2-Tetrachloroethane	5.	U
108-88-3	Toluene	5.	U
108-90-7	Chlorobenzene	5.	U
100-41-4	Ethylbenzene	5.	U
100-42-5	Styrene	5.	U
133-02-7	Xylene (total)	5.	U

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1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

92-05953

Lab Name: BATTELLE-PNL Contract: ------

Lab Code: ------ Case No.: ----- SAS No.: ----- SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: B01SB4

Sample wt/vol: 5.201 (g/mL) G Lab File ID: >PB510

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec.2.3 Date Analyzed: 4/15/92

Column: CAP Dilution Factor: 1.00000

Number TICs found: 1

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.	Unkown Alkanes	31.1-35	4500	J
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
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SEMI-VOLATILE ORGANIC ANALYSIS RESULTS

SAMPLE ANALYSIS REPORTED

Analysis of three samples from SDG #17 for semivolatile organic compounds by gas chromatography/mass spectrometry (GC/MS) is the subject of this report.

SAMPLE DESCRIPTION AND PREPARATION

Sample ID	ACL Lab Number
B01SH7	92-05877
B01SB4	92-05953
B01SC8	92-05954

The samples were received in good condition: sample B01SH7 on 04/06/92 and samples B01SB4 and B01SC8 on 04/13/92. Extractions of both samples and spiked sample were performed.

- Extraction procedure — PNL-ALO-344.
- Extraction location — Lab 302, 325 building.
- Extraction type — Sonication, low level, soil.
- Sample/Extract storage temperature — 4°C(+/-2°).

ANALYSIS METHOD

- GC/MS procedure: PNL-ALO-345.
- GC/MS instrumentation: HP-5890/5970 GC/MS (WB38473)
- GC/MS location: Lab 325, 325 building.

QUALITY CONTROL

The QC features in the analytical procedure were followed, the following summarizes the QC results.

Form	Information	Comments
2D	Surrogate Recovery	Requirements were met. Sample B01SH7 had one acid surrogate below limits, which is acceptable.

3D	MS/MSD Recovery	Requirements were met.
4B	Method Blank Summary	Requirements were met.
5B	Tune/Mass Calibration	Requirements were met.
6B,C	Initial Calibration	Requirements were met.
7B,C	Daily Calibration	Requirements were met.
8B,C	Internal Standards	Requirements were met.

The following comments provide additional information on QC and related features for analysis of these samples.

- Holding time. All requirements were met.
- 12-hour calibration. The last sample analyzed, B01SC8-MSD was injected after the 12-hour period, and was re-analyzed with another batch of 200-BP-1 samples two days later. Consequently, there are two sets of Forms 5 (mass calibration and tune check report), 7 (continuing calibration report), and 8 (internal standard report).
- Surrogate recoveries. There was one surrogate recovery out of limits low for sample B01SH7. The 2,4,6-tribromophenol recovery was 2%. The other two acid surrogate recoveries were lower than usual, but within specified limits. The nitration product of 2-fluorophenol was observed, but nitration products of the other two acid surrogates, phenol-d5 and 2,4,6-tribromophenol, were not found.

RESULTS

CLP Target Compounds: As seen in the attached 1B,C Forms, target compounds found were di-n-butylphthalate in all three samples and the blank.

Bis(2-ethylhexyl)phthalate was found in samples B01SH7 and B01SB4, and di-n-octylphthalate in sample B01SC8. In all cases, the concentrations were well below quantitation limits.

Tentatively Identified Compounds (TIC): The attached 1F Forms show results for the TIC's. Concentration estimates for the TIC's are made assuming that the response factor for each TIC is one. The peak area for each TIC is then compared to the area of the nearest internal standard (for which concentrations are known) to estimate the TIC concentrations. Identification of the TIC is made by a computer search of the NIST mass spectral library to attempt a match with the spectrum of each of the TIC's. The TIC's reported as "Unknown" did not have satisfactory matches with library spectra. The TIC concentration estimate and identification are reported only if the TIC peak area is 10% or greater than the nearest internal standard peak area.

- The TIC's in the blank are products of the aldol condensation reaction occurring during sample preparation. These products are designated by an "A" in the "Q" column of the 1F forms.

- The 2-fluoro-4-nitrophenol in sample B01SH7 is the nitration reaction product of the acid surrogate spike 2-fluorophenol. The "Unknown" compounds all appeared to be oxidized organics. Although tributyl phosphate was present, there was no indication of solvent.

- Sample B01SB4 contained a large number of unknown alkanes plus four unknown cycloalkanes, and two unknown ketones in addition to tetradecane, hexadecane, and tributyl phosphate. The unusual nature of this sample is that all of these compounds eluted by 22 minutes (~180° C). In previous samples with this many TIC's, a number of compounds eluted at higher temperatures.

- Sample B01SC8 had the tributyl phosphate as well as the higher boiling normal paraffin hydrocarbon constituents. Several unknown alkanes were also present.

The following defines the Q-flags in the Form 1's

"Q" Flag	Definition
U	Indicates the compound was analyzed for, but not detected, the U-flagged concentration is the Contract Required Quantitation Limit.
J	Indicates an estimated value for target and tentatively identified compounds, spectra meet criteria but response is below Contract Required Quantitation Limit for the target compounds.
B	Indicates compound was found in the blank.
X	Indicates compound was manually deleted because all requirements were not met.
D	Indicates analysis was performed on a diluted sample.
E	Indicates that quantitation was outside of the calibration range.
A	Aldol condensation product.

1B
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B01SH7

Lab Name: BATTELLE-PNL Contract: -------

Lab Code: ------- Case No.: ------ SAS No.: ------ SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05877-E1

Sample wt/vol: 30.232 (g/mL) G Lab File ID: >E1306

Level: (low/med) LOW Date Received: 04/06/92

% Moisture: not dec.3.72 dec. -- Date Extracted: 04/16/92

Extraction: (Sepf/Cont/Sonc) SONC Date Analyzed: 5/13/92

GPC Cleanup: (Y/N) N pH:6.4 Dilution Factor: 1.00000

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
108-95-2	Phenol	340.	U
111-44-4	bis(2-Chloroethyl)Ether	340.	U
95-57-8	2-Chlorophenol	340.	U
541-73-1	1,3-Dichlorobenzene	340.	U
106-46-7	1,4-Dichlorobenzene	340.	U
100-51-6	Benzyl alcohol	340.	U
95-50-1	1,2-Dichlorobenzene	340.	U
95-48-7	2-Methylphenol	340.	U
39638-32-9	bis(2-chloroisopropyl)ether	340.	U
106-44-5	4-Methylphenol	340.	U
621-64-7	N-Nitroso-Di-n-propylamine	340.	U
67-72-1	Hexachloroethane	340.	U
98-95-3	Nitrobenzene	340.	U
78-59-1	Isophorone	340.	U
88-75-5	2-Nitrophenol	340.	U
105-67-9	2,4-Dimethylphenol	340.	U
65-85-0	Benzoic acid	1700.	U
111-91-1	bis(2-Chloroethoxy)methane	340.	U
120-83-2	2,4-Dichlorophenol	340.	U
120-82-1	1,2,4-Trichlorobenzene	340.	U
91-20-3	Naphthalene	340.	U
106-47-8	4-Chloroaniline	340.	U
87-68-3	Hexachlorobutadiene	340.	U
59-50-7	4-Chloro-3-methylphenol	340.	U
91-57-6	2-Methylnaphthalene	340.	U
77-47-4	Hexachlorocyclopentadiene	340.	U
88-06-2	2,4,6-Trichlorophenol	340.	U
95-95-4	2,4,5-Trichlorophenol	1700.	U
91-58-7	2-Chloronaphthalene	340.	U
88-74-4	2-Nitroaniline	1700.	U
131-11-3	Dimethylphthalate	340.	U
208-96-8	Acenaphthylene	340.	U
606-20-2	2,6-Dinitrotoluene	340.	U

9713508.2034

1C
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B01SH7

Lab Name: BATTELLE-PNL Contract: -------

Lab Code: ------- Case No.: ------ SAS No.: ------ SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05877-E1

Sample wt/vol: 30.232 (g/mL) G Lab File ID: >E1306

Level: (low/med) LOW Date Received: 04/06/92

% Moisture: not dec.3.72 dec. -- Date Extracted: 04/16/92

Extraction: (Sepf/Cont/Sonc) SONC Date Analyzed: 5/13/92

GPC Cleanup: (Y/N) N pH: 6.4 Dilution Factor: 1.00000

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
99-09-2	3-Nitroaniline	1700.	U
83-32-9	Acenaphthene	340.	U
51-28-5	2,4-Dinitrophenol	1700.	U
100-02-7	4-Nitrophenol	1700.	U
132-64-9	Dibenzofuran	340.	U
121-14-2	2,4-Dinitrotoluene	340.	U
84-66-2	Diethylphthalate	340.	U
7005-72-3	4-Chlorophenyl-phenylether	340.	U
86-73-7	Fluorene	340.	U
100-01-6	4-Nitroaniline	1700.	U
534-52-1	4,6-Dinitro-2-methylphenol	1700.	U
86-30-6	N-Nitrosodiphenylamine (1)	340.	U
101-55-3	4-Bromophenyl-phenylether	340.	U
118-74-1	Hexachlorobenzene	340.	U
87-86-5	Pentachlorophenol	1700.	U
85-01-8	Phenanthrene	340.	U
120-12-7	Anthracene	340.	U
84-74-2	Di-n-butylphthalate	86.	JB
206-44-0	Fluoranthene	340.	U
129-00-0	Pyrene	340.	U
85-68-7	Butylbenzylphthalate	340.	U
91-94-1	3,3'-Dichlorobenzidine	690.	U
56-55-3	Benzo(a)anthracene	340.	U
218-01-9	Chrysene	340.	U
117-81-7	bis(2-Ethylhexyl)phthalate	19.	J
117-84-0	Di-n-octylphthalate	340.	U
205-99-2	Benzo(b)fluoranthene	340.	U
207-08-9	Benzo(k)fluoranthene	340.	U
50-32-8	Benzo(a)pyrene	340.	U
193-39-5	Indeno(1,2,3-cd)pyrene	340.	U
53-70-3	Dibenz(a,h)anthracene	340.	U
191-24-2	Benzo(g,h,i)perylene	340.	U

(1) - Cannot be separated from Diphenylamine

1F
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

B01SH7

Lab Name: BATTELLE-PNL Contract: -------

Lab Code: ------- Case No.: ------ SAS No.: ------ SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05877-E1

Sample wt/vol: 30.232 (g/mL) G Lab File ID: >E1306

Level: (low/med) LOW Date Received: 04/06/92

% Moisture: not dec. 3.72 dec. -- Date Extracted: 04/16/92

Extraction: (Sepf/Cont/Sonc) SONC Date Analyzed: 5/13/92

GPC Cleanup: (Y/N) N pH: 6.4 Dilution Factor: 1.00000

Number TICs found: 10

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.	Unknown	4.13	24000.	JAB
2.	Unknown	4.79	610.	JAB
3.	Unknown	5.12	2300.	JAB
4.	Unknown	5.22	630.	J
5.	Unknown	6.43	140.	J
6.	Unknown	7.89	200.	J
7. 403190	2-FLUORO-4-NITROPHENOL	8.23	220.	J
8.	Unknown	12.23	200.	J
9.	Unknown	20.20	370.	J
10. 126738	Phosphoric acid tributyl est	20.48	1900.	J
11.				
12.				
13.				
14.				
15.				
16.				
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1B
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B01SB4

Lab Name: BATTELLE-PNL Contract: -------

Lab Code: ------- Case No.: ------ SAS No.: ------ SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05953-E1

Sample wt/vol: 30.040 (g/mL) G Lab File ID: >E1307

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec.2.30 dec. -- Date Extracted: 04/16/92

Extraction: (Sepf/Cont/Sonc) SONC Date Analyzed: 5/13/92

GPC Cleanup: (Y/N) N pH:9.0 Dilution Factor: 1.00000

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
108-95-2	Phenol	340.	U
111-44-4	bis(2-Chloroethyl)Ether	340.	U
95-57-8	2-Chlorophenol	340.	U
541-73-1	1,3-Dichlorobenzene	340.	U
106-46-7	1,4-Dichlorobenzene	340.	U
100-51-6	Benzyl alcohol	340.	U
95-50-1	1,2-Dichlorobenzene	340.	U
95-48-7	2-Methylphenol	340.	U
39638-32-9	bis(2-chloroisopropyl)ether	340.	U
106-44-5	4-Methylphenol	340.	U
621-64-7	N-Nitroso-Di-n-propylamine	340.	U
67-72-1	Hexachloroethane	340.	U
98-95-3	Nitrobenzene	340.	U
78-59-1	Isophorone	340.	U
88-75-5	2-Nitrophenol	340.	U
105-67-9	2,4-Dimethylphenol	340.	U
65-85-0	Benzoic acid	1700.	U
111-91-1	bis(2-Chloroethoxy)methane	340.	U
120-83-2	2,4-Dichlorophenol	340.	U
120-82-1	1,2,4-Trichlorobenzene	340.	U
91-20-3	Naphthalene	340.	U
106-47-8	4-Chloroaniline	340.	U
87-68-3	Hexachlorobutadiene	340.	U
59-50-7	4-Chloro-3-methylphenol	340.	U
91-57-6	2-Methylnaphthalene	340.	U
77-47-4	Hexachlorocyclopentadiene	340.	U
88-06-2	2,4,6-Trichlorophenol	340.	U
95-95-4	2,4,5-Trichlorophenol	1700.	U
91-58-7	2-Chloronaphthalene	340.	U
88-74-4	2-Nitroaniline	1700.	U
131-11-3	Dimethylphthalate	340.	U
208-96-8	Acenaphthylene	340.	U
606-20-2	2,6-Dinitrotoluene	340.	U

1C
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B01SB4

Lab Name: BATTELLE-PNL Contract: -------

Lab Code: ------- Case No.: ------ SAS No.: ------ SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05953-E1

Sample wt/vol: 30.040 (g/mL) G Lab File ID: >E1307

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec.2.30 dec. -- Date Extracted: 04/16/92

Extraction: (Sepf/Cont/Sonc) SONC Date Analyzed: 5/13/92

GPC Cleanup: (Y/N) N pH:9.0 Dilution Factor: 1.00000

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
99-09-2	3-Nitroaniline	1700.	U
83-32-9	Acenaphthene	340.	U
51-28-5	2,4-Dinitrophenol	1700.	U
100-02-7	4-Nitrophenol	1700.	U
132-64-9	Dibenzofuran	340.	U
121-14-2	2,4-Dinitrotoluene	340.	U
84-66-2	Diethylphthalate	240.	J
7005-72-3	4-Chlorophenyl-phenylether	340.	U
86-73-7	Fluorene	340.	U
100-01-6	4-Nitroaniline	1700.	U
534-52-1	4,6-Dinitro-2-methylphenol	1700.	U
86-30-6	N-Nitrosodiphenylamine (1)	340.	U
101-55-3	4-Bromophenyl-phenylether	340.	U
118-74-1	Hexachlorobenzene	340.	U
87-86-5	Pentachlorophenol	1700.	U
85-01-8	Phenanthrene	340.	U
120-12-7	Anthracene	340.	U
84-74-2	Di-n-butylphthalate	140.	JB
206-44-0	Fluoranthene	340.	U
129-00-0	Pyrene	340.	U
85-68-7	Butylbenzylphthalate	340.	U
91-94-1	3,3'-Dichlorobenzidine	680.	U
56-55-3	Benzo(a)anthracene	340.	U
218-01-9	Chrysene	340.	U
117-81-7	bis(2-Ethylhexyl)phthalate	23.	J
117-84-0	Di-n-octylphthalate	340.	U
205-99-2	Benzo(b)fluoranthene	340.	U
207-08-9	Benzo(k)fluoranthene	340.	U
50-32-8	Benzo(a)pyrene	340.	U
193-39-5	Indeno(1,2,3-cd)pyrene	340.	U
53-70-3	Dibenz(a,h)anthracene	340.	U
191-24-2	Benzo(g,h,i)perylene	340.	U

(1) - Cannot be separated from Diphenylamine

1F
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

B01SB4

Lab Name: BATTELLE-PNL Contract: -------

Lab Code: ------- Case No.: ------ SAS No.: ------ SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05953-E1

Sample wt/vol: 30.040 (g/mL) G Lab File ID: >E1307

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec.2.30 dec. -- Date Extracted: 04/16/92

Extraction: (Sepf/Cont/Sonc) SONC Date Analyzed: 5/13/92

GPC Cleanup: (Y/N) N pH:9.0 Dilution Factor: 1.00000

Number TICs found: 30

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.	Unknown	4.13	22000.	JAB
2.	Unknown	4.78	640.	JAB
3.	Unknown alkane	10.84	1600.	J
4. 629505	Tridecane	11.51	4600.	J
5.	Unknown alkane	11.93	820.	J
6.	Unknown cycloalkane	12.61	680.	J
7.	Unknown alkane	12.75	510.	J
8.	Unknown alkane	12.96	1200.	J
9.	Unknown alkane	13.08	2300.	J
10.	Unknown alkane	13.47	9700.	J
11.	Unknown cycloalkane	13.80	470.	J
12. 629594	Tetradecane	14.12	21000.	J
13.	Unknown alkane	14.18	1700.	J
14.	Unknown alkane	14.28	640.	J
15.	Unknown alkane	14.44	520.	J
16.	Unknown cycloalkane	15.31	3200.	J
17.	Unknown alkane	15.39	800.	J
18.	Unknown alkane	15.55	1600.	J
19.	Unknown alkane	15.71	13000.	J
20.	Unknown alkane	15.88	2500.	J
21.	Unknown alkene	16.49	630.	J
22.	Unknown alkane	16.86	470.	J
23.	Unknown cycloalkane	17.99	950.	J
24.	Unknown alkane	18.25	720.	J
25.	Unknown alkane	18.46	1100.	J
26.	Unknown ketone	18.53	770.	J
27. 544763	Hexadecane	19.22	4100.	J
28. 126738	Phosphoric acid tributyl est	20.51	2600.	J
29.	Unknown ketone	21.02	390.	J
30.	Unknown alkane	21.70	460.	J

1B
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B01SC8

Lab Name: BATTELLE-PNL Contract: -------

Lab Code: ------- Case No.: ------ SAS No.: ------ SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05954-E1

Sample wt/vol: 30.240 (g/mL) G Lab File ID: >E1308

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec.2.10 dec. -- Date Extracted: 04/16/92

Extraction: (Sepf/Cont/Sonc) SONC Date Analyzed: 5/13/92

GPC Cleanup: (Y/N) N pH:9.0 Dilution Factor: 1.00000

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
108-95-2	Phenol	340.	U
111-44-4	bis(2-Chloroethyl)Ether	340.	U
95-57-8	2-Chlorophenol	340.	U
541-73-1	1,3-Dichlorobenzene	340.	U
106-46-7	1,4-Dichlorobenzene	340.	U
100-51-6	Benzyl alcohol	340.	U
95-50-1	1,2-Dichlorobenzene	340.	U
95-48-7	2-Methylphenol	340.	U
39638-32-9	bis(2-chloroisopropyl)ether	340.	U
106-44-5	4-Methylphenol	340.	U
621-64-7	N-Nitroso-Di-n-propylamine	340.	U
67-72-1	Hexachloroethane	340.	U
98-95-3	Nitrobenzene	340.	U
78-59-1	Isophorone	340.	U
88-75-5	2-Nitrophenol	340.	U
105-67-9	2,4-Dimethylphenol	340.	U
65-85-0	Benzoic acid	1700.	U
111-91-1	bis(2-Chloroethoxy)methane	340.	U
120-83-2	2,4-Dichlorophenol	340.	U
120-82-1	1,2,4-Trichlorobenzene	340.	U
91-20-3	Naphthalene	340.	U
106-47-8	4-Chloroaniline	340.	U
87-68-3	Hexachlorobutadiene	340.	U
59-50-7	4-Chloro-3-methylphenol	340.	U
91-57-6	2-Methylnaphthalene	340.	U
77-47-4	Hexachlorocyclopentadiene	340.	U
88-06-2	2,4,6-Trichlorophenol	340.	U
95-95-4	2,4,5-Trichlorophenol	1700.	U
91-58-7	2-Chloronaphthalene	340.	U
88-74-4	2-Nitroaniline	1700.	U
131-11-3	Dimethylphthalate	340.	U
208-96-8	Acenaphthylene	340.	U
606-20-2	2,6-Dinitrotoluene	340.	U

1C
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B01SC8

Lab Name: BATTELLE-PNL Contract: -------

Lab Code: ------- Case No.: ------ SAS No.: ------ SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05954-E1

Sample wt/vol: 30.240 (g/mL) G Lab File ID: >E1308

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec.2.10 dec. -- Date Extracted: 04/16/92

Extraction: (Sepf/Cont/Sonc) SONC Date Analyzed: 5/13/92

GPC Cleanup: (Y/N) N pH:9.0 Dilution Factor: 1.00000

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
99-09-2	3-Nitroaniline	1700.	U
83-32-9	Acenaphthene	340.	U
51-28-5	2,4-Dinitrophenol	1700.	U
100-02-7	4-Nitrophenol	1700.	U
132-64-9	Dibenzofuran	340.	U
121-14-2	2,4-Dinitrotoluene	340.	U
84-66-2	Diethylphthalate	340.	U
7005-72-3	4-Chlorophenyl-phenylether	340.	U
86-73-7	Fluorene	340.	U
100-01-6	4-Nitroaniline	1700.	U
534-52-1	4,6-Dinitro-2-methylphenol	1700.	U
86-30-6	N-Nitrosodiphenylamine (1)	340.	U
101-55-3	4-Bromophenyl-phenylether	340.	U
118-74-1	Hexachlorobenzene	340.	U
87-86-5	Pentachlorophenol	1700.	U
85-01-8	Phenanthrene	340.	U
120-12-7	Anthracene	340.	U
84-74-2	Di-n-butylphthalate	39.	JB
206-44-0	Fluoranthene	340.	U
129-00-0	Pyrene	340.	U
85-68-7	Butylbenzylphthalate	340.	U
91-94-1	3,3'-Dichlorobenzidine	680.	U
56-55-3	Benzo(a)anthracene	340.	U
218-01-9	Chrysene	340.	U
117-81-7	bis(2-Ethylhexyl)phthalate	340.	U
117-84-0	Di-n-octylphthalate	15.	J
205-99-2	Benzo(b)fluoranthene	340.	U
207-08-9	Benzo(k)fluoranthene	340.	U
50-32-8	Benzo(a)pyrene	340.	U
193-39-5	Indeno(1,2,3-cd)pyrene	340.	U
53-70-3	Dibenz(a,h)anthracene	340.	U
191-24-2	Benzo(g,h,i)perylene	340.	U

(1) - Cannot be separated from Diphenylamine

9713508.2041

1F

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

B01SC8

Lab Name: BATTELLE-PNL Contract: -------

Lab Code: ------- Case No.: ------ SAS No.: ------ SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05954-E1

Sample wt/vol: 30.240 (g/mL) G Lab File ID: >E1308

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec.2.10 dec. -- Date Extracted: 04/16/92

Extraction: (Sepf/Cont/Sonc) SONC Date Analyzed: 5/13/92

GPC Cleanup: (Y/N) N pH:9.0 Dilution Factor: 1.00000

Number TICs found: 11

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/Kg

	CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.		Unknown	4.11	21000.	JAB
2.		Unknown	4.77	660.	JAB
3.		Unknown	5.08	170.	JAB
4.		Unknown alkane	10.82	170.	J
5,	629505	Tridecane	11.46	230.	J
6.		Unknown alkane	13.39	650.	J
7.	629594	Tetradecane	13.99	950.	J
8.		Unknown alkane	15.61	950.	J
9.	629629	Pentadecane	16.59	1100.	J
10.	544763	Hexadecane	19.17	360.	J
11.	126738	Phosphoric acid tributyl est	20.43	1300.	J
12.					
13.					
14.					
15.					
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PCB/PESTICIDE ANALYSIS RESULTS

SAMPLE ANALYSIS REPORTED

Analysis of three, 200-BP-1 samples consisting of sample delivery group sixteen (SDG #17) for pesticides and PCBs (Aroclors) by gas chromatography/electron capture detection (GC/ECD) is the subject of this report.

SAMPLE DESCRIPTION AND PREPARATION

Sample ID	ACL Lab Number
B01SH7	92-05877
B01SB4	92-05953
B01SC8	92-05954
B01SC8 MS	92-05954-P2
B01SC8 MSD	92-05954-P3
PBLK07 (Method Blank)	92-05954-P4

The samples were received in good condition on 04/06/92 (B01SH7) and on 04/01/92 (B01SB4, B01SC8). Samples were extracted on 04/16/92.

- Extraction procedure — PNL-ALO-347.
- Extraction location — Lab 302, 325 building
- Extraction type — Sonication, low level, soil
- Sample/Extract storage temperature — 4°C(+/-2°)

ANALYSIS METHOD

- GC/ECD procedure: — PNL-ALO-346.
- GC/ECD instrumentation: — HP-5890 (WB60701)
- GC/ECD location: — Lab 325, 325 building.

QUALITY CONTROL

In addition to the result forms (1D) the QC features in the analytical procedure were followed as described; the following lists the forms that relate to QC and summarizes the QC results.

Form	Information	Comments
2F	Surrogate Recovery	Meets all requirements.(1)
3F	MS/MSD Recovery	DDT recovered high in the matrix spike sample, otherwise meets all recovery and RPD requirements.
4C	Method Blank Summary	Meets all method blank requirements.
8D	Evaluation Standards	Meets all requirements.
8E	Retention Time Summary	Meets all requirements.
9	Standards Summary	%D exceeded limits for Methoxychlor.
1D	Extraction Holding Time	Requirements were met.
1D	Analysis Holding Time	Requirements were met.

(1) Surrogate and matrix spike recoveries are advisory: although corrective action is not required.

RESULTS

As indicated on the attached 1D Forms the blank and the samples were not found to contain target compounds at levels greater than the contract required quantitation limit (CRQL) and are "Q" flagged accordingly.

Matrix Spike recovery results are slightly high for DDT in sample B01SC8MS. Matrix Spike recovery criteria are advisory and do not require further action.

The following defines the Q-flags in the Form 1's

"Q" Flag	Definition
U	Indicates the compound was analyzed for but not detected, the U-flagged concentration is the Contract Required Quantitation Limit.
B	Indicates compound was found in the blank.
D	Indicates analysis was performed on a diluted sample.
M	Indicates a matrix spike compound.

9713508.2045

1D
PESTICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B01SH7

Lab Name: Battelle PNL Contract:

Lab Code: PNL Case No.: SAS No.: SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05877-P-1

Sample wt/vol: 30.23 (g/mL)G Lab File ID: >01526

Level: (low/med) LOW Date Received: 04/06/92

% Moisture: not dec. 3.72 dec. Date Extracted: 04/16/92

Extraction: (SepF/Cont/Sonc) SONC Date Analyzed: 05/13/92

GPC Cleanup: (Y/N) N pH: 6.4 Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
319-84-6	Alpha-BHC	8.20	U
319-85-7	Beta-BHC	8.20	U
319-86-8	Delta-BHC	8.20	U
58-89-9	Gamma-BHC (Lindane)	8.20	U
76-44-8	Heptachlor	8.20	U
309-00-2	Aldrin	8.20	U
1024-57-3	Heptachlor Epoxide	8.20	U
959-98-8	Endosulfan I	8.20	U
60-57-1	Dieldrin	16.00	U
72-55-9	4,4'-DDE	16.00	U
72-20-8	Endrin	16.00	U
33213-65-9	Endosulfan II	16.00	U
72-54-8	4,4'-DDD	16.00	U
1013-07-8	Endosulfan Sulfate	16.00	U
50-29-3	4,4'-DDT	16.00	U
72-43-5	Methoxychlor	82.00	U
53494-70-5	Endrin Ketone	16.00	U
5103-71-9	alpha-Chlordane	82.00	U
5103-74-2	gamma-Chlordane	82.00	U
8001-35-2	Toxaphene	160.00	U
12674-11-2	Aroclor-1016	82.00	U
11104-28-2	Aroclor-1221	82.00	U
11141-16-5	Aroclor-1232	82.00	U
53469-21-9	Aroclor-1242	82.00	U
12672-29-6	Aroclor-1248	82.00	U
11097-69-1	Aroclor-1254	160.00	U
11096-82-5	Aroclor-1260	160.00	U

9713508.2046

1D
PESTICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B01SB4

Lab Name: Battelle PNL Contract:

Lab Code: PNL Case No.: SAS No.: SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05953-P-1

Sample wt/vol: 30.04 (g/mL)G Lab File ID: >01527

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec. 2.3 dec. Date Extracted: 04/16/92

Extraction: (SepF/Cont/Sonc) SONC Date Analyzed: 05/13/92

GPC Cleanup: (Y/N) N pH: 9 Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
319-84-6	Alpha-BHC	8.20	U
319-85-7	Beta-BHC	8.20	U
319-86-8	Delta-BHC	8.20	U
58-89-9	Gamma-BHC (Lindane)	8.20	U
76-44-8	Heptachlor	8.20	U
309-00-2	Aldrin	8.20	U
1024-57-3	Heptachlor Epoxide	8.20	U
959-98-8	Endosulfan I	8.20	U
60-57-1	Dieldrin	16.00	U
72-55-9	4,4'-DDE	16.00	U
72-20-8	Endrin	16.00	U
33213-65-9	Endosulfan II	16.00	U
72-54-8	4,4'-DDD	16.00	U
1013-07-8	Endosulfan Sulfate	16.00	U
50-29-3	4,4'-DDT	16.00	U
72-43-5	Methoxychlor	82.00	U
53494-70-5	Endrin Ketone	16.00	U
5103-71-9	alpha-Chlordane	82.00	U
5103-74-2	gamma-Chlordane	82.00	U
8001-35-2	Toxaphene	160.00	U
12674-11-2	Aroclor-1016	82.00	U
11104-28-2	Aroclor-1221	82.00	U
11141-16-5	Aroclor-1232	82.00	U
53469-21-9	Aroclor-1242	82.00	U
12672-29-6	Aroclor-1248	82.00	U
11097-69-1	Aroclor-1254	160.00	U
11096-82-5	Aroclor-1260	160.00	U

1D
PESTICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

B01SC8

Lab Name: Battelle PNL Contract:

Lab Code: PNL Case No.: SAS No.: SDG No.: 17

Matrix: (soil/water) SOIL Lab Sample ID: 92-05954-P-1

Sample wt/vol: 30.24 (g/mL)G Lab File ID: >01528

Level: (low/med) LOW Date Received: 04/13/92

% Moisture: not dec. 2.1 dec. Date Extracted: 04/16/92

Extraction: (SepF/Cont/Sonc) SONC Date Analyzed: 05/13/92

GPC Cleanup: (Y/N) N pH: 9 Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
319-84-6	Alpha-BHC	8.10	U
319-85-7	Beta-BHC	8.10	U
319-86-8	Delta-BHC	8.10	U
58-89-9	Gamma-BHC (Lindane)	8.10	U
76-44-8	Heptachlor	8.10	U
309-00-2	Aldrin	8.10	U
1024-57-3	Heptachlor Epoxide	8.10	U
959-98-8	Endosulfan I	8.10	U
60-57-1	Dieldrin	16.00	U
72-55-9	4,4'-DDE	16.00	U
72-20-8	Endrin	16.00	U
33213-65-9	Endosulfan II	16.00	U
72-54-8	4,4'-DDD	16.00	U
1013-07-8	Endosulfan Sulfate	16.00	U
50-29-3	4,4'-DDT	16.00	U
72-43-5	Methoxychlor	81.00	U
53494-70-5	Endrin Ketone	16.00	U
5103-71-9	alpha-Chlordane	81.00	U
5103-74-2	gamma-Chlordane	81.00	U
8001-35-2	Toxaphene	160.00	U
12674-11-2	Aroclor-1016	81.00	U
11104-28-2	Aroclor-1221	81.00	U
11141-16-5	Aroclor-1232	81.00	U
53469-21-9	Aroclor-1242	81.00	U
12672-29-6	Aroclor-1248	81.00	U
11097-69-1	Aroclor-1254	160.00	U
11096-82-5	Aroclor-1260	160.00	U

9713508.2048

SAMPLE RECEIPT FORM

Delivered by: Wendy Thompson Date/Time: 4-6-92 1135

Received by: Nancy Wynhoff

Customer Name or Project: 200-BP-1

Customer Sample Number(s): B01SH7

ALO Sample Number(s): 92-05877

1. Customer Chain-of-Custody Form: Present ✓ Absent ____

2. Additional Shipping Forms (list):
Sample Analysis Request, off-site RSR, Rad Shipment Checklist, Container Inspection Checklist

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

   Present X Absent ____

   If Present, Condition: ____

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

   Notes:

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

6. Condition of Sample Vials.
OK

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

8. Resolution of Problems or Discrepancies.
none

RETURN COMPLETED FORM TO PROJECT MANAGER

9713508.2049

B01-003

Battelle Pacific Northwest Laboratories	Radiation Protection Record: RADIATION SURVEY REPORT	Facility: 329	Location: 5-A	Time: 1130 To 1200	Date: 6APRIL92	Survey Number: 110139

Description of Job: Survey of 200 BP-1 in pure 200 area Sample # 92-05877

See attached sheet

Item No.	LOCATION OR ITEM	MEASUREMENT DATA: Meter Deflection W O	Meter Deflection W C	Source Size	Source a) Dist.	Measurement Beta mrad/h	Measurement Gamma mR/h	Measurement Neutron mrem/h	CONTAMINATION DATA: Direct b) Alpha dpm	Direct b) Alpha <DL	Direct c) Beta-Gamma dpm	Direct c) Beta-Gamma <DL	Removable Alpha dpm	Removable Alpha <DL	Removable Beta-Gamma dpm	Removable Beta-Gamma <DL	Removable Area cm²	d) Un-Cond. Relsd Yes
1	reading of jar	150	150	3"x6"	C	NA	150	NA	NA		NA			✓		✓		NA
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		

PERSONNEL DOSE RATE DATA

Item No.	Phase of Work	Whole Body Penetrating mrem/h	Whole Body Nonpenetrating mrem/h	Extremities mrem/h
1	to handle jar	150	150	150

INSTRUMENT DATA

Instrument Type	Serial Number	Response Checked	Instrument Type	Serial Number	Response Checked
[X] CP	6564	[X] Yes	[X] GM with Pancake Probe	1358 / 380	[X] Yes
[ ] HRCP		[ ] Yes	[ ] PAM		[ ] Yes
[ ] BBCP		[ ] Yes	[ ] PAC 6		[ ] Yes
[ ] BWCP		[ ] Yes	[ ]		[ ] Yes
[ ] Snoopy					

RESPIRATORY PROTECTION DATA

Type of respirator(s) worn: ☐ ~~Supplied Air~~ ☐ ~~Filtered~~ ☐ ~~Other~~ N.A ☒ None

SURVEY PERFORMED BY

RPT Signature: Jeffrey R Abel

a) = Distance = B = Beam, C = Contact, F = Field

b) = per probe area, or about 50 cm².

c) = per probe area, or about 20 cm².

<DL = Less than detection limits, or Alpha = <220 dpm per probe area, or Beta-Gamma = <1,000 dpm per probe area.

d) = Un-Cond. Relsd = Unconditionally Released

NT = None taken

SEE REVERSE SIDE

9713508.2050

Westinghouse Hanford Company	CHAIN OF CUSTODY

Custody Form Initiator M. C. Douglas — Project #: 91-019

Company Contact H. S. Thompson — Telephone (509) 376-2153

Project Designation/Sampling Locations 200-BP-1 Operable Unit — Collection Date 4-1-92

Boring: 216-B-48C — Time 0930

Ice Chest No. #1DA39 — Field Logbook No. WHC-385-12

Bill of Lading/Airbill No. N/A — Offsite Property No. N/A

Method of Shipment Hand Carry

Shipped to 329 PNL Laboratory Attn: Nancy Wynhoff

Possible Sample Hazards/Remarks Sample is radioactive. No chemical hazards detected. 100 mr/hr. Keep sample chilled. All analyses CLP. See lab statement of work.

Sample Identification

① B01SH7

1, 1000 ml, glass, soil, CLP - Total alpha/beta, GEA, Sr90, Cs137, Co60, Pu238, Pu239/240, Ru106.

☐ Field Transfer of Custody	CHAIN OF POSSESSION	(Sign and Print Names)
Relinquished by: M. C. Douglas Matthew C. Douglas	Received by: W.S. Thompson Kendy S Thompson	Date/Time: ~~3~~ 4-6-92 0900
Relinquished by: W.S. Thompson Kendy S Thompson	Received by: N Wynhoff	Date/Time: 4-6-92 1135
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:

Comments:

A-6000-407 (12/90)

B01-004

9713508.2051

Westinghouse Hanford Company

SAMPLE ANALYSIS REQUEST

PART I: FIELD SECTION

Collector W.S. Thompson Date Sampled 4-1-92 Time 0930 hours

Company Contact W.S. Thompson Telephone (509) 376-2153

Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
① B01SH7	1, 1000 ml, glass	soil	CLP-Total alpha/beta, GEA, Sr90, Cs137, Co60, Pu238, Pu239/240, Ru106.

Field Information** Sample collected in support of the 200-BP-1, Task 2 & 4, RI/FS project.
Borehole: 216-B-48C

Special Handling and/or Storage Attn: Nancy Wynhoff Keep sample chilled.
All analyses CLP. See lab statement of work.

Possible Sample Hazards Sample is radioactive. 100 mr/hr.
No chemical hazards detected.

PART II: LABORATORY SECTION

Received by N.J. Wynhoff Title Sr Tech Spec I Date 4-6-92

Analysis Required

*Indicate whether sample is soil, sludge, water, etc.
**Use back of page for additional information relative to sample location

A 6000 406 (05/90)

B01-005

9713508.2052

SAMPLE RECEIPT FORM

Delivered by: Wendy Thompson Date/Time: 4/6/92 12:15 pm

Received by: Jim Robbins / Diana Bellofatto

Customer Name or Project: _____

Customer Sample Number(s): B015H7, B015J0, B015J2, B015J4, B015J2, B015BC

ALO Sample Number(s): 92-05877, 92-05878, 92-05879, 92-05880, 92-05881, 92-05882

1. Customer Chain-of-Custody Form: Present X Absent _____

2. Additional Shipping Forms (list): SAR

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

   Present good Absent _____

   If Present, Condition: intact

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

   Notes:

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.) good 1°C

6. Condition of Sample Vials. good

7. Verification of Agreement or Nonagreement of Information on Receiving Documents. N/A

8. Resolution of Problems or Discrepancies. N/A

RETURN COMPLETED FORM TO PROJECT MANAGER

9713508.2053

Westinghouse Hanford Company	CHAIN OF CUSTODY

Custody Form Initiator M.C. Douglas — Project #: 91-019

Company Contact W. S. Thompson — Telephone (509) 376-2153

Project Designation/Sampling Locations 200-BP-1 Operable Unit — Collection Date 4-1-92

Boring: 216-B-48C — Time: 0930

ice Chest No. # EFSG 18 — Field Logbook No. WHC-N-305-12

Bill of Lading/Airbill No. N/A — Offsite Property No. N/A

Method of Shipment Hand Carry

Shipped to 325 PNL Laboratory (300 Area) Attn: Diana Bellofatto.

Possible Sample Hazards/Remarks Sample is radioactive: 120 ml - 40 mr/hr, 950 ml - 70 mr/hr. No chemical hazards detected. Keep sample chilled. All analyses CLP. See lab statement of work.

Sample Identification

① B01SH7

1, 120 ml, amber glass, soil, CLP - VOA

1, 950 ml, amber glass, soil, CLP - Semi-VOA, Pest/PCB, TOC

] Field Transfer of Custody	CHAIN OF POSSESSION	(Sign and Print Names)
Relinquished by: M.C. Douglas Matthew C. Douglas	Received by: W.S. Thompson Wendy S Thompson	Date/Time: 4-6-92 0900
Relinquished by: W.S. Thompson Wendy S Thompson	Received by: Diana Bellofatto Diana Bellofatto	Date/Time: 4/6/92 12:15 pm
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:

Comments:

A-6000-407 (12-90)

B01-007

9713508.2054

Westinghouse Hanford Company

SAMPLE ANALYSIS REQUEST

PART I: FIELD SECTION

Collector W. S. Thompson Date Sampled 4-1-92 Time 0930 hours

Company Contact W. S. Thompson Telephone (509) 376-2153

Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
① B01SH7	1, 120 ml, amber glass	Soil	CLP- VOA
	1, 950 ml, amber glass	Soil	CLP- Semi-VOA, Pest/PCB, TOC

Field Information** Sample collected in support of the 200-BP-1, Task 2 & 4, RI/FS project.
Borehole: 216-B-48C

Special Handling and/or Storage Attn: Diana Bellofatto. Keep sample chilled.
All analyses CLP. See lab statement of work.

Possible Sample Hazards Sample is radioactive: 120 ml - 40 mr/hr; 950 ml - 70 mr/hr.
No chemical hazards detected.

PART II: LABORATORY SECTION

Received by [illegible] 4-6-92 Title ~~Sr Tech Spec I~~ Sr. Technician 4-6-92 Date 4-6-92

Analysis Required Diana Bellofatto

*Indicate whether sample is soil, sludge, water, etc.

**Use back of page for additional information relative to sample location

A 6000 406 (05/90)

B01-008

9713508.2055

Westinghouse Hanford Company	CHAIN OF CUSTODY

Custody Form Initiator M.C. Douglas — Project #: 91-019

Company Contact W. S. Thompson — Telephone (509) 376-2153

Project Designation/Sampling Locations 200-BP-1 Operable Unit — Collection Date 4-1-92, 4-2-92, 4-3-92

Boring: 216-B-48C and 216-B-44C — Time: 0930, 1421, 0844, 0925, 0930, 1010

Ice Chest No. (#1) = ETS6#18, SMC-111(#2-6) — Field Logbook No. WHC-N-385-12

Bill of Lading/Airbill No. N/A — Offsite Property No. N/A

Method of Shipment Hand Carry

Shipped to 325 PNL Laboratory (300 Area) Attn: Jim Robbins

Possible Sample Hazards/Remarks Sample ① is radioactive: 25 mr/hr. All other samples are <D. No chemical hazards detected. Keep samples chilled All analyses CLP. See lab statement of work.

Sample Identification

① B01SH7 (0930 on 4-1-92)
1, 125 ml, glass, soil, CLP-Total Cn, Free Cn, Ferro Cn, ICP/AA Metals, ICP/ms, Bi, Tc99, NO3, SO4, PO4, Total Uranium

② B01SJ0 (1421 on 4-2-92)
1, 125 ml, glass, soil, CLP - Total Cn, Free Cn, Ferro Cn.

③ B01SJ2 (0844 on 4-3-92)
1, 125 ml, glass, soil, CLP - Total Cn, Free Cn, Ferro Cn.

④ B01SJ4 (0925 on 4-3-92)
1, 125 ml, glass, soil, CLP - Total Cn, Free Cn, Ferro Cn.

⑤ B01SS2 (0930 on 4-3-92)
3, 1000 ml, plastic, water (NaOH), CLP - Total Cn, Free Cn, Ferro Cn

⑥ B01SB0 (1010 on 4-3-92)
3, 1000 ml, plastic, water (NaOH), CLP - Total Cn, Free Cn, Ferro Cn.

☐ Field Transfer of Custody	CHAIN OF POSSESSION	(Sign and Print Names)
Relinquished by: M. C. Douglas Matthew C. Douglas	Received by: W. S. Thompson Hendy S Thompson	Date/Time: 4-6-92 0900
Relinquished by: W. S. Thompson Hendy S Thompson	Received by: J. ROBBINS James Robbins	Date/Time: 4-6-92 1230
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:

Comments:

A-6000-407 (12/91)

B01-009

9713508.2056

Westinghouse Hanford Company

SAMPLE ANALYSIS REQUEST

PART I: FIELD SECTION

Collector W. S. Thompson, M. C. Douglas

Company Contact W. S. Thompson

Date Sampled 4-1-92 4-2-92 4-3-92

Time 0930 ① 1421 ② 0844 ③ hours 0925 ④ 0930 ⑤ 1010 ⑥

Telephone (509) 376-2153

Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
① B01SH7	1, 125ml, glass	Soil	CLP - Total Cn, Free Cn, Ferro Cn, ICP/AA metals, ICP/ms, Bi, Tc99, NO3, SO4, PO4, Total Uranium.
② B01SJ0	1, 125ml, glass	Soil	CLP - Total Cn, Free Cn, Ferro Cn
③ B01SJ2	1, 125ml, glass	Soil	CLP - Total Cn, Free Cn, Ferro Cn.
④ B01SJ4	1, 125ml, glass	Soil	CLP - Total Cn, Free Cn, Ferro Cn.
⑤ B01SS2	3, 1000ml, plastic	Water (NaOH)	CLP - Total Cn, Free Cn, Ferro Cn.
⑥ B01SB0	3, 1000ml, plastic	Water (NaOH)	CLP - Total Cn, Free Cn, Ferro Cn.

Field Information** Sample collected in support of the 200-BP-1, Task 2 & 4, RI/FS project.
Borehole: 216-B-48C & 216-B-44C.

Special Handling and/or Storage Attn: Jim Robbins. Keep samples chilled.
All analyses CLP. See lab statement of work.

Possible Sample Hazards Sample ① is radioactive: 25mr/hr. All other samples are <D.
No chemical hazards detected.

PART II: LABORATORY SECTION

Received by [signature] Title Sr. RES SCIENTIST Date 4-6-92

Analysis Required TOTAL CN (6) PARAMETERS OF INTEREST (1)

*Indicate whether sample is soil, sludge, water, etc.
**Use back of page for additional information relative to sample location

A 6000 406 (05/90)

B01-010

9713508.2057

SAMPLE RECEIPT FORM

Delivered by: W. Thompson Date/Time: 4/13/92, 11:20

Received by: S. K. Fadeff

Customer Name or Project: 200BP1

Customer Sample Number(s): B013B4, B01SC8

ALO Sample Number(s): 92-05953, 92-05954

1. Customer Chain-of-Custody Form: Present ✓ Absent ____

2. Additional Shipping Forms (list):
RSR, Radioactive Shipment checklist, CoC, analysis request, total activity report

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

   Present ✓ Absent ____

   If Present, Condition: in tact

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

   Notes: NA

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.) in tact

6. Condition of Sample Vials. Ok

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

8. Resolution of Problems or Discrepancies.
B01SC8 - smearable contamination on exterior jar
B01SB4 - " " " "

S.K. Fadeff
4/13/92

RETURN COMPLETED FORM TO PROJECT MANAGER

9713508.2058

Westinghouse Hanford Company	CHAIN OF CUSTODY

Custody Form Initiator M. C. Douglas — Project #: 91-019

Company Contact W. S. Thompson — Telephone (509) 376-2153

Project Designation/Sampling Locations 200-BP-1 Operable Unit — Collection Date 4-8-92

Boring: 216-B-44C — Time 0834, 1021

Ice Chest No. #391DA — Field Logbook No. WHC-385-12

Bill of Lading/Airbill No. N/A — Offsite Property No. N/A

Method of Shipment Hand Carry

Shipped to 329 PNL Laboratory, Attn: Nancy Wynhoff.

Possible Sample Hazards/Remarks Samples are radioactive: ① 19 mr/hr, 243 mrad/hr; ② 10 mr/hr, 180 mrad/hr. No chemical hazards detected. Keep samples chilled. All analyses CLP.

Sample Identification — See lab statement of work.

① B01SB4

1, 1000 ml, glass, soil, CLP - Total alpha/beta, GEA, Sr90, Cs137, Co60, Pu238, Pu239/240, Ru106.

② B01SC8

1, 1000 ml, glass, soil, CLP - Total alpha/beta, GEA, Sr90, Cs137, Co60, Pu238, Pu239/240, Ru106.

☐ Field Transfer of Custody — CHAIN OF POSSESSION — (Sign and Print Names)

Relinquished by:	Received by:	Date/Time:
M. C. Douglas Matthew C. Douglas	W. S. Thompson Wendy S. Thompson	0830 4-13-92
Relinquished by: W. S. Thompson Wendy S. Thompson	Received by: S. L. Fedoff	Date/Time: 4/13/92 11:20 a.m.
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:

Comments:

A-6000-407 (12/90)

B01-012

9713508.2059

Westinghouse Hanford Company

SAMPLE ANALYSIS REQUEST

PART I: FIELD SECTION

Collector M. C. Douglas Date Sampled 4-8-92 Time 0834 1021 hours

Company Contact W. S. Thompson Telephone (509) 376-2153

	Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
①	B01SB4	1, 1000 ml, glass	Soil	CLP - Total alpha/beta, GEA, Sr90, Cs137, Co60, Pu238, Pu239/240, Ru106.
②	B01SC8	1, 1000 ml, glass	Soil	CLP - Total alpha/beta, GEA, Sr90, Cs137, Co60, Pu238, Pu239/240, Ru106.

Field Information** Samples collected in support of the 200-BP-1, Task 2 & 4, RI/FS project.
Borehole: 216-B-44C.

Special Handling and/or Storage Attn: Nancy Wynhoff. Keep samples chilled.
All analyses CLP. See lab statement of work.

Possible Sample Hazards Samples are radioactive: ① 19 mr/hr, 243 mrad/hr; ② 10 mr/hr, 180 mrad/hr. No chemical hazards detected.

PART II: LABORATORY SECTION

Received by _____ Title _____ Date _____

Analysis Required _____

*Indicate whether sample is soil, sludge, water, etc.
**Use back of page for additional information relative to sample location

A 6000 406 (05/90)

B01-013

9713508.2060

SAMPLE RECEIPT FORM

Delivered by: Wendy Thompson Date/Time: 4-13-92 - 12:15 pm

Received by: Sim Robbins/Diana Bellofatto

Customer Name or Project: 200-BP-1

Customer Sample Number(s): B01SB4, B01SC8

ALO Sample Number(s): 92-05953, 92-05954

1. Customer Chain-of-Custody Form: Present X Absent ____

2. Additional Shipping Forms (list): SAR

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

   Present X 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: good

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.) 4°C

6. Condition of Sample Vials. good

7. Verification of Agreement or Nonagreement of Information on Receiving Documents. N/A

8. Resolution of Problems or Discrepancies.

   N/A

RETURN COMPLETED FORM TO PROJECT MANAGER

9713508.2061

Westinghouse Hanford Company	CHAIN OF CUSTODY

Custody Form Initiator M.C. Douglas — Project #: 91-019

Company Contact W. S. Thompson — Telephone (509) 376-2153

Project Designation/Sampling Locations 200-BP-1 Operable Unit — Collection Date 4-8-92

Boring: 216-B-44C — Time: 0834, 1021

Ice Chest No. SML 159 — Field Logbook No. WHC-N-385-12

Bill of Lading/Airbill No. N/A — Offsite Property No. N/A

Method of Shipment Hand Carry

Shipped to 325 PNL Laboratory (300 Area) Attn: Jim Robbins

Possible Sample Hazards/Remarks Samples are radioactive: ① 9 mr/hr, 111 mrad/hr; ② 6 mr/hr, 90 mrad/hr
No chemical hazards detected. Keep samples chilled. All analyses CLP. See lab statement of work.

Sample Identification

① B01SB4
1, 125 ml, glass, soil, CLP - Total Cn, Free Cn, Ferro Cn, ICP/AA metals, ICP/ms, Bi, Tc99, NO3, SO4, PO4, Total Uranium.

② B01SC8
1, 125 ml, glass, soil, CLP - Total Cn, Free Cn, Ferro Cn, ICP/AA metals, ICP/ms, Bi, Tc99, NO3, SO4, PO4, Total Uranium.

] Field Transfer of Custody	CHAIN OF POSSESSION	(Sign and Print Names)
Relinquished by: M.C. Douglas Matthew C. Douglas	Received by: W.S. Thompson Wesley S Thompson	Date/Time: 4-13-92 0830
Relinquished by: W.S. Thompson Wesley S Thompson	Received by: J. ROBBINS James Robbins	Date/Time: 4-13-92 12:15
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:

Comments:

A-6000-407 (12/90)

B01-015

9713508.2062

Westinghouse Hanford Company

SAMPLE ANALYSIS REQUEST

PART I: FIELD SECTION

Collector M. C. Douglas Date Sampled 4-8-92 Time 0834 1021 hours

Company Contact W. S. Thompson Telephone (509) 376-2153

Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
① B01SB4	1, 125 ml, glass, ~~soil~~ mco 4-13-92	Soil	CLP- Total Cn, Free Cn, Ferro Cn, ICP/AA metals, ICP/ms, Bi, Tc99, NO3, SO4, PO4, Total Uranium.
② B01SC8	1, 125 ml, glass	Soil	CLP- Total Cn, Free Cn, Ferro Cn, ICP/AA metals, ICP/ms, Bi, Tc99, NO3, SO4, PO4, Total Uranium.

Field Information** Samples collected in support of the 200-BP-1, Task 2 & 4, RI/FS project.
Borehole: 216-B-44C

Special Handling and/or Storage Attn: Jim Robbins. Keep Samples chilled
All analyses CLP. See lab statement of work.

Possible Sample Hazards Samples are radioactive. ① 9 mr/hr, 111 mrad/hr; ② 6 mr/hr, 90 mrad/hr. No chemical hazards detected.

PART II: LABORATORY SECTION

Received by James Robbins Title SR. RES. SCIENTIST Date 4/13/92

Analysis Required TOTAL CN

*Indicate whether sample is soil, sludge, water, etc.
**Use back of page for additional information relative to sample location

A 6000 406 (05/90)

B01-016

9713508.2063

Westinghouse Hanford Company	CHAIN OF CUSTODY

Custody Form Initiator M.C. Douglas — Project #: 91-019

Company Contact W. S. Thompson — Telephone (509) 376-2153

Project Designation/Sampling Locations 200-BP-1 Operable Unit — Collection Date 4-8-92

Boring: 216-B-44C — Time: 0834, 1021

Ice Chest No. SML-159 — Field Logbook No. WHC-N-385-12

Bill of Lading/Airbill No. N/A — Offsite Property No. N/A

Method of Shipment Hand Carry

Shipped to 325 PNL Laboratory (300 Area) Attn: Diana Bellofatto.

Possible Sample Hazards/Remarks Samples are radioactive: ① 950 ml - 40 mr/hr, 60 mrad/hr. 120 ml - 25 mr/hr, 30 mrad/hr. ; ② 950 ml - 28 mr/hr, 96 mrad/hr. 120 ml - 12 mr/hr, 18 mrad/hr. No chemical hazards detected. Keep samples chilled. All analyses CLP. See Lab statement of work.

Sample Identification

① B01SB4
1, 120 ml, amber glass, soil, CLP - VOA
1, 950 ml, amber glass, soil, CLP - Semi-VOA, Pest/PCB, TOC

② B01SC8
1, 120 ml, amber glass, soil, CLP - VOA.
1, 950 ml, amber glass, soil, CLP - Semi-VOA, Pest/PCB, TOC.

☐ Field Transfer of Custody — CHAIN OF POSSESSION — (Sign and Print Names)

Relinquished by:	Received by:	Date/Time:
M.C. Douglas Matthew C. Douglas	W. S. Thompson Sandy J Thompson	0830 4-13-92
W.S. Thompson Sandy J Thompson	Diana Bellofatto Diana Bellofatto	12:15 pm 4-13-92

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:

Comments:

A-6000-407 (12/90)

B01-017

9713508.2064

Westinghouse Hanford Company

SAMPLE ANALYSIS REQUEST

PART I: FIELD SECTION

Collector M. C. Douglas Date Sampled 4-8-92 Time 0834 1021 hours

Company Contact W. S. Thompson Telephone (509) 376-2153

	Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
①	B01SB4	1, 120 ml, amberglass	Soil	CLP- VOA
		1, 950 ml, amberglass	Soil	CLP- Semi-VOA, Pest/PCB, TOC
②	B01SC8	1, 120 ml, amberglass	Soil	CLP- VOA
		1, 950 ml, amberglass	Soil	CLP- Semi-VOA, Pest/PCB, TOC

Field Information** Samples collected in support of the 200-BP-1, Task 2 & 4, RI/FS project.
Borehole: 216-B-44C

Special Handling and/or Storage Attn: Diana Bellofatto. Keep samples chilled.
All analyses CLP. See lab statement of work.

Possible Sample Hazards Samples are radioactive: ① 120 ml - 25 mr/hr, 30 mrad/hr. 950 ml - 40 mr/hr, 60 mrad/hr.
② 120 ml - 12 mr/hr, 18 mrad/hr, 950 ml - 28 mr/hr, 96 mrad/hr. No chemical hazards detected.

PART II: LABORATORY SECTION

Received by Diana Bellofatto Title Senior Tech Date 4-13-92

Analysis Required

*Indicate whether sample is soil, sludge, water, etc.
**Use back of page for additional information relative to sample location

A 6000 406 (05/90)

B01-018

9713508.2065

SAMPLE RECEIPT FORM

Delivered by: MATT DOUGLAS Date/Time: 4-16-92 11:25

Received by: JIM ROBBINS

Customer Name or Project: 200 BP-1 TASK 2&4

Customer Sample Number(s): BØ1SF0, BØ1SG0, BØ1SG8

ALO Sample Number(s): 92-06336, 92-06337, 92-06338

1. 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 10°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

9713508.2066

Westinghouse Hanford Company	CHAIN OF CUSTODY

Custody Form Initiator M. C. Douglas — Project #: 91-019

Company Contact W. S. Thompson — Telephone (509) 376-2153

Project Designation/Sampling Locations 200-BP-1 Operable Unit — Collection Date 4-14-92 (1,2), 4-15-92 (3)

Boring: 216-B-47A — Time: 1244 (1), 1244 (2), 0915 (3)

Ice Chest No. SML-10 — Field Logbook No. WHC-N-385-12

Bill of Lading/Airbill No. N/A — Offsite Property No. N/A

Method of Shipment Hand Carry

Shipped to 325 PNL Laboratory (300 Area) Attn: Jim Robbins

Possible Sample Hazards/Remarks No radiological or chemical hazards detected. Keep samples chilled. All analyses CLP. See lab statement of work.

Sample Identification

(1) B01SF0 (1244)
1, 125ml, glass, soil, CLP - Total Cn, Free Cn, Ferro Cn.

(2) B01SG0 (1244)
1, 125ml, glass, soil, CLP - Total Cn, Free Cn, Ferro Cn.

(3) B01SG8 (0915)
1, 125ml, glass, soil, CLP - Total Cn, Free Cn, Ferro Cn.

☐ Field Transfer of Custody	CHAIN OF POSSESSION	(Sign and Print Names)
Relinquished by: M. C. Douglas Matthew C. Douglas	Received by: J. ROBBINS J. Robbins	Date/Time: 4-16-92 11:25
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:

Comments:

A-6000-407 (12/90)

B01-020

9713508.2067

Westinghouse Hanford Company

SAMPLE ANALYSIS REQUEST

PART I: FIELD SECTION

Collector M. C. Douglas Date Sampled 4-14-92 / 4-15-92 Time 1244 / 1244 / 0915 hours

Company Contact W. S. Thompson Telephone (509) 376-2153

	Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
1	B01SF0	1, 125 ml, glass	Soil	CLP - Total Cn, Free Cn, Ferro Cn
2	B01SG0	1, 125 ml, glass	Soil	CLP - Total Cn, Free Cn, Ferro Cn
3	B01SG8	1, 125 ml, glass	Soil	CLP - Total Cn, Free Cn, Ferro Cn

Field Information** Samples collected in support of the 200-BP-1, RI/FS Project, Task 2 & 4.
Borehole 21b-B-47A

Special Handling and/or Storage Keep Samples chilled. All analyses CLP. See lab statement of work.

Possible Sample Hazards No radiological or chemical hazards detected.

PART II: LABORATORY SECTION

Received by J. ROBBINS Title Sr. RES SCIENTIST Date 4-16-92

Analysis Required TOTAL CN

*Indicate whether sample is soil, sludge, water, etc.
**Use back of page for additional information relative to sample location

A 6000 406 (05/9

B01-021

9713508.2068

MCO 4-15-92

SAMPLE STATUS REPORT FOR E 1977. E-BLANK ~~B01SDS~~ B01SFO TIME: 4/15/92 8:18
DISPATCHED: 3/30/92 13:45 SAMPLE HAS NOT BEEN SLURPED
RECEIVED: 4/14/92 14:53

EXT.	DETER.	RESULTS OR STATUS	OUT OF RANGE?	GOOD ANS?	CHARGE CODE
****	********	**********	***	***	******
4271	TOT-ACT	< 5.00000E 01 PICI/G	N	Y	FE3DP

END OF REPORT

B01-022

9713508.2069

B01SGO mco 4-15-92

SAMPLE STATUS REPORT FOR E 1978. E-BLANK ~~B01CF9~~ TIME: 4/15/92 8:18
DISPATCHED: 3/30/92 13:45 SAMPLE HAS NOT BEEN SLURPED
RECEIVED: 4/14/92 14:53

EXT.	DETER.	RESULTS OR STATUS	OUT OF RANGE?	GOOD ANS?	CHARGE CODE
****	********	**********	***	***	******
4271	TOT-ACT	< 5.00000E 01 PICI/G	N	Y	PE3DP

END OF REPORT

B01-023

9713508.2070

B01SG8 mcd 4-16-92

SAMPLE STATUS REPORT FOR E-1979. E-BLANK ~~B01SG4~~ TIME: 4/16/92 8:45
DISPATCHED: 3/30/92 13:46 SAMPLE HAS NOT BEEN SLURPED
RECEIVED: 4/15/92 15:7

EXT.	DETER.	RESULTS OR STATUS	OUT OF RANGE?	GOOD ANS?	CHARGE CODE
****	********	**********	***	***	******
4271	TOT-ACT	< 5.00000E 01 PICI/G	N	Y	PE3BP

END OF REPORT

B01-024

9713508.2071

Samples were delivered to analysts directly, therefore no PNL Chain of Custody was required.

9713508.2072

FROM THE DESK OF: **PAT REICH**
SAMPLE MANAGEMENT
(509) 372-2785/H4-14

TO: B01SH7-PNL-068 **DATE:** March 29, 1995

SUBJECT: **TC-99 AND URANIUM DATA FOR B01SH7-PNL-068**

Due to Lab sample batching the Tc-99 and Uranium analysis with the complete raw data requested for this data package is filed in B015P1-PNL-069.

Attached is a copy of the PNL Summary and Cover letter with the sample results included.

Thank You,

Pat Reich
Sample Management

9713508.2073

COPY

Battelle
Pacific Northwest Laboratories
P.O. Box 999
Richland, Washington U.S.A. 99352
Telephone (509) 376-5802
Telex 15-2874

October 14, 1992

OCT 1992
RECEIVED
OSM DMO

Ms. J. M. Duncan, T6-08
Westinghouse Hanford Company
P.O. Box 1970
Richland, Washington 99352

Dear Ms. Duncan:

TRANSMITTAL OF 200-BP-1 SITE INVESTIGATION ANALYTICAL CHEMISTRY SUPPORT PROJECT, TASKS 2 AND 4, ICP-MS Tc-99 AND URANIUM DATA PACKAGE/REPORT FOR NOS. 12-19, REVISION 0, OCTOBER 14, 1992

(Ref: Letter to J. A. Lerch, "Transmittal of Interim Change Notice for Pacific Northwest Laboratory (PNL) Analytical Procedure for the 200-BP-1 Site Investigation Analytical Chemistry Support Project." dated April 1, 1992.)

Enclosed are two copies of the report and one copy of the appendices of the subject 200-BP-1 Tasks 2 and 4, ICP-MS Tc-99 and Uranium Data Package/Report for SDG Nos. 12-19, Revision 0, October 14, 1992 and a PNL Analytical Chemistry Laboratory Data Report/Package Acknowledgement Form for the data report/package. Please sign the form and return to me as agreed upon in the reference above.

Also, a reminder, PNL is to be notified of validation of all data report/packages as defined in the "Memorandum of Understanding Between the Pacific Northwest Laboratory (PNL) and Westinghouse Hanford Company (WHC) on 325 Laboratory Operations in Support of Hanford Site Tank Waste Characterization Activities, March 10, 1992."

If you have any questions about the above or after reviewing the data, please give me a call on 376-5802.

Sincerely,

B. M. Gillespie

B. M. Gillespie
200-BP-1 PNL Project Manager
Analytical Laboratory Operations

Enclosures

9713508.2079

COPY

200-BP-1 SITE INVESTIGATION
ANALYTICAL CHEMISTRY SUPPORT PROJECT

TASKS 2 & 4

ICP-MS Tc-99 and Uranium

DATA PACKAGE/REPORT for SDG NOs. 12-19

Revision 0

RECORD COPY

October 14, 1992

OCT 1992
RECEIVED
OSM DMO

Prepared by: B.M. Gillespie

Pacific Northwest Laboratory

(PNL Project #16772)

9713508.2075

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9713588.2076

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APPENDICES: Primary Analytical Data

Appendix A: Test Instructions

Appendix B: Chain of Custody

B1 - Westinghouse Chain of Custody and Sample Analysis Request Forms

B2 - PNL Chain of Custody Forms

Appendix C: Radiochemistry Analysis Primary Data

C1 - Introduction

C2 - Laboratory Analyst Signature List

C3 - ICP-MS, Tc-99 and Uranium Activity Analysis

9715508.2077

TABLES

Table 1: 200-BP-1 Sample Numbers 4

Table 2: ICP-MS, Tc-99 and Uranium Activity Analysis 10

9713508.2078

CONTRIBUTORS LIST

PROJECT MANAGEMENT OFFICE

BM Gillespie, Project Manager
TL Ehlert, Quality Engineer
RM Nipper, Project Assistant
JL Reilly, Senior Clerk
SL Humphreys, Senior Clerk

QUALITY CONTROL OFFICERS

KJ Kuhl-Klinger

RADIOCHEMISTRY ANALYSIS

DW Koppenaal, Sub Task Leader
JP Bramson
MW Goheen
EJ Wyse

HOT CELL ANALYSIS

RT Steele
FV Hoopes
JK Rau

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 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 to the 300 Area, 325 building 200-BP-1 Sample Custodian.

The analyses requested for the samples in this report are technicium-99 and uranium. The quality control (QC) requirements for the samples are defined in the test instructions for each sample. The QC requirements outlined in the procedures and requested in the WHC SOW were followed. Method blanks, matrix spikes, sample duplicates and/or matrix spike duplicates were analyzed. All QC data that exist are included in this Data Package/Report.

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 ICP-MS analytical data.

9715508.2080

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

10/14/92
Date

200-BP-1 TASKS 2 AND 4 ICP-MS HOT SAMPLE ANALYSES

DATA PACKAGE TASK 2&4, #12

B015P1	92-02332
B015P5	92-02628
B015P9	92-02629
B015Q1	92-02630

DATA PACKAGE TASK 2&4, #13

B015R5	92-03236
B015S1	92-03300

DATA PACKAGE TASK 2&4, #14

B01S75	92-03738
B01S77	92-03901
B01S89	92-03902

DATA PACKAGE TASK 2&4, #15

B01S97	92-04734
B01S99	92-04735
B01SB1	92-04736
B01SB3	92-04968
B01SB7	92-05102
B01SB2	92-05150

DATA PACKAGE TASK 2&4, #16

B01SC7	92-05270
B01SC9	92-05271
B01SD9	92-05352
B01SD7	92-05353
B01SF7	92-05490
B01SF9	92-05491
B01SG3	92-05492
B01SG7	92-05633
B01SG9	92-05634
B01SH3	92-05690

DATA PACKAGE TASK 2&4, #17

B01SH7	92-05877
B01SB4	92-05953
B01SC8	92-05954

200-BP-1 TASKS 2 AND 4 ICP-MS HOT SAMPLE ANALYSES
(continued)

DATA PACKAGE TASK 2&4, #18

B067Y5	92-06428
B067Y7	92-06713
B067Z3	92-06726
B067Z5	92-06727
B06809	92-06904

DATA PACKAGE TASK 2&4, #19

B06813	92-06963
B06815	92-06964
B06823	92-07040
B06825	92-07141
B06827	92-07142

9713508.2083

RADIOCHEMICAL DATA TABLES

ICP/MS ANALYSIS RESULTS

The project samples and accompanying QC were prepared for ICP/MS analysis using procedure PNL-ALO-101, Acid Digestion for Metal Analysis. This methodology is consistent with CLP Inorganics acid digestion for metals. The resulting digestates were further diluted and analyzed by inductively coupled plasma mass spectrometry (ICP/MS) according to procedure PNL-ALO-280, Inductively Coupled Plasma Mass Spectrometric (ICP/MS) Analysis. Technetium was determined using procedure PNL-ALO-281 (ICP/MS Determination of ^{99}Tc), and uranium was determined using procedure PNL-ALO-282 (Determination of Uranium Concentration / Isotopic Composition Using ICP/MS). The calibration standard concentrations for these analyses are documented in the raw data. Samples were prepared in the 325 building and the ^{99}Tc and U determinations were performed on the shielded ICP/MS unit in room 115 of building 3708 in the 300 area. A total of five analytical runs were necessary to include all regular and QC samples. The fifth analysis was performed for samples requiring a reanalysis. Criteria for a reanalysis included memory effects, concentrations outside calibration range, or general inconsistencies.

Previous ICP/MS analyses for 200-BP-1 samples involved the use of an ultrasonic nebulizer (USN) to maximize instrument sensitivity. Unfortunately, a USN for radioactive use has not yet been acquired, so the samples were aspirated using a conventional pneumatic nebulizer. While the extra sensitivity may have been beneficial for determining the technetium concentration, it would have had a negative impact on the uranium analysis. In fact, it is almost certain that the use of a USN would have resulted in additional dilutions to obtain the uranium concentration in many of the samples. As it was, all samples were analyzed for Tc at an additional dilution of only 2X to mitigate matrix effects.

Results were calculated using PQ software as outlined in PNL-ALO-280 and the operations manuals. Additional calculations can be necessary, however, to correct for interferences with elements of interest. At mass 99, technetium is interfered by an isotope of ruthenium; a correction is therefore necessary to subtract out the isobaric ruthenium contribution. To be accurate, this

correction factor assumes natural ruthenium isotopic abundance. There are indications that the apparent Ru in these samples is not of natural isotopic abundance; this is not surprising considering the likely source of this ruthenium. It appears that this ruthenium does not contain a measurable isotope at mass 99. For this reason, correction factors for ruthenium were obtained but were not applied in calculating the technetium concentrations. (These factors are reported in the data table however for information and for the sake of completeness.) Judging from the spiked sample recovery values, the decision not to factor in the possible Ru contribution appears to be legitimate. Some samples were calculated as containing small but measurable quantities of technetium, however the corresponding spectrum did not indicate a positive identifiable peak; these results are reported but parenthesized to indicate qualitative uncertainty.

As specified in ALO-280, appropriate internal standards were used to correct for instrument drift and general signal suppression. The elements chosen were indium (for technetium), thorium and thallium (for uranium). Indium is commonly used as an internal standard due to its absence in all but very few samples. There is more difficulty in choosing an appropriate internal standard for the high masses; there aren't that many of them above mass 200, and those that exist may be present in soils. For this reason, two elements were chosen to represent the high mass response. Unfortunately, the results indicate that many of the samples contain at least one of these elements in measurable concentrations. This was not a problem in 4 of the 5 runs, because indium served as a legitimate substitute; for one run (2922a) however, only uranium was being analyzed and indium was not scanned. To obtain legitimate results for this run, the uranium concentration for all runs was calculated against each internal standard, and the results calculated against Th and Tl were compared with those calculated against In. A thorough analysis of the data indicates that the results calculated against Tl most closely resembled the In results. It appeared that there was a small but measurable quantity of Th in many of the samples (indicated by 10-20% lower uranium concentrations relative to those obtained against In and Tl), and the few samples that contained Tl contained it in significant quantities (indicated by dramatically lower uranium concentrations relative to those

obtained against In and Th). Simply put, the presence of thallium was obvious when reviewing the raw data but the presence of Th was not. Therefore the results obtained on 2922a were calculated using Tl as the internal standard unless Tl was present in the sample. The three samples that contained Tl were calculated using Th as the internal standard.

Quality control measures included the analysis of check standards, sample duplicates, and spiked samples. Technetium matrix spike recoveries show good agreement with true values, but the uranium concentration in the samples is again far greater than the amount spiked, thereby invalidating the spiked sample recovery. Additionally, the blank spike uranium recovery for sample 2628-B6 was negatively affected by a relatively high blank concentration; this is not believed to be significant, however, considering the significantly higher uranium concentration observed in the samples. The uranium recovery for 5633-B6 is acceptable if the uncertainty of the value is considered. Recovery for Tc and U was calculated using the following formula:

Matrix Spike recovery (%) = 100 * ([Spiked Sample] - [Sample]) / [Spike]

Blank Spike recovery (%) = 100 * ([Spiked Blank] - [Blank]) / [Spike]

Unless otherwise specified, the uncertainty of the values reported is estimated at <±10%, and is based on known versus found check standard results, results between duplicate samples, and deviations between analytical runs. Data in parentheses should be considered suspect.

200-BP-1 HOT SOIL ANALYSIS

Tc-99 and Uranium determinations

Sep. 30, 1992

PNL Sample I.D. # (a)	Digest. Dil. Factor (b)	ICP/MS Analysis No.	Analysis Dilution Factor (c)	[Tc-99] ng/ml leachate (d)	[Tc-99] ng/g soil (e,f)	Ru Correctn Factor (g)	[Tc-99] ng/g spiked (f)	[Tc-99] spike yield, % (h)	[Tc-99] pCi/g, dry soil	ICP/MS Analysis No.	Analysis Dilution Factor (c)	[U] ng/ml leachate (d)	[U] µg/g soil (e,f)	[U] µg/g spiked (f)	[U] spike yield, % (h, i)	[U] pCi/g, dry soil (j)
2332 - B1	180.5	2925a32	2x	<0.02	<4				<70	2925a32	2x	148	26.7			18.7
2628 - B1	175.5	2925a38	2x	0.03	5				(90)	2925a38	2x	288	50.5			35.3
2628 - B2	179.6	2925a37	2x	0.04	8				(100)	2925a37	2x	284	51.0			35.6
2628 - B3	172.7	2925a36	2x	0.12	21	0.180	17	90%	350	2925a36	2x	329	56.8	0.173	3639%	39.7
2628 - B5	200.0	2925a35	2x	<0.02	<4				<70	2925a35	2x	2±8	0.4±2			0.3±2
2628 - B6	200.0	2925a33	2x	0.12	24	0.864	20	120%	410	2925a33	2x	1±4	0.2±0.8	0.200	-86%	0.1±0.5
2629 - B1	193.5	2928a17	2x	0.02	5				(80)	2928a17	2x	228	44.1			30.8
2630 - B1	183.6	2925a24	2x	<0.02	<4				<70	2925a24	2x	34.4	8.32			4.41
3236 - B1	197.8	2925a28	2x	0.03	5				(90)	2925a28	2x	43.0	8.50			5.94
3237 - B1	162.0	2921a31	2x	0.04	6				(100)	2922a30	2x	80.0	13.0			9.05
3300 - B1	176.0	2928a19	2x	0.02	4	0.734			60	2928a19	2x	2.51	0.44			0.31
3738 - B1	184.5	2921a19	2x	0.03	6				(100)	2922a29	2x	221	40.8			28.5
3901 - B1	191.7	2921a17	2x	<0.02	<4				<70	2922a27	5x	407	78.0			54.5
3901 - B2	196.7	2925a20	2x	<0.02	<4				<70	2922a22	5x	402	79.1			55.2
3901 - B3	158.4	2925a21	2x	0.11	17	0.197	16	108%	290	2922a23	5x	401	62.7	0.156	-9816%	43.8
3901 - B4	196.0	2925a4	2x	0.03±0.01	6±2				(100±30)	2922a3	2x	75.5	14.8			10.3
3901 - B5	200.0	2925a6	2x	<0.02	<4				<70	2922a4	2x	0.22	0.04			0.03
3901 - B6	200.0	2925a5	2x	0.12	24	0.955	20	120%	410	2922a5	2x	1.10	0.22	0.200	86%	0.15
3902 - B1	163.2	2921a18	2x	0.03	5				(80)	2922a28	5x	1060	173			121
4734 - Spike		2925a7	2x	0.12		0.949				2925a7	2x	9.13				
4734 - B1	186.7	2925a8	2x	<0.02	<4				<70	2925a8	2x	2.8±0.7	0.52±0.13			0.36±0.09
4735 - B1	207.0	2925a16	2x	0.03	7				(100)	2925a16	2x	103	21.3			14.9
4736 - B1	163.9	2925a12	2x	0.08	10				(200)	2925a12	2x	184	30.2			21.1
4968 - B1	144.8	2925a14	2x	<0.02	<3				<50	2925a14	2x	352	51.0			35.6
5102 - B1	204.5	2925a22	2x	<0.02	<4				<70	2928a4	2x	3.80	0.78			0.54
5102 Dup	200.0	2925a27	2x	<0.02	<4				<70	2928a22	2x	5.14	1.03			0.72
5150 - B1	189.0	2925a13	2x	0.02	5				(80)	2925a13	2x	314	59.4			41.5
5270 - B1	174.5	2923a16	2x	0.07	13				(200)	2928a13	50x	1830	319			223
5271 - B1	192.7	2923a9	2x	<0.02	<4				<70	2923a9	2x	273	52.6			36.7
5352 - B1	168.0	2923a23	2x	<0.02	<3				<60	2923a23	2x	346	58.1			40.6
5353 - B1	141.0	2923a4	2x	<0.02	<3				<50	2923a4	2x	290	40.9			28.6
5490 - B1	185.3	2923a20	2x	<0.02	<4				<70	2923a20	2x	451	83.6			58.4
5491 - B1	173.7	2923a19	2x	<0.02	<3				<60	2923a19	2x	91.0	15.8			11.0
5492 - B1	192.1	2928a18	2x	<0.02	<4				<70	2928a18	2x	3.05	0.59			0.41
5633 - B1	205.5	2928a7	2x	0.03±0.02	6±4	0.509			100±70	2928a7	2x	39.0	8.01			5.60
5633 - B2	170.1	2923a21	2x	<0.02	<3				<70	2923a21	2x	45.0	7.65			5.35
5633 - B3	194.3	2923a7	2x	0.13	25	0.691	19	100%	426	2923a7	2x	38.0	7.38	0.194	-325%	5.16
5633 - B4	195.9	2923a13	2x	0.1±0.2	20±40				(340±680)	2923a13	2x	72.0	14.1			9.85
5633 - B5	200.0	2928a9	2x	<0.02	<4				<70	2928a9	2x	0.1±0.1	0.02			0.01±0.01
5633 - B6	200.0	2928a10	2x	0.11	22	0.945	20	110%	370	2928a10	2x	0.8±0.2	0.16±0.04	0.200	70%	0.11±0.03
5634 - B1	170.0	2923a5	2x	0.04	7				(100)	2923a5	2x	577	98.1			68.5
5690 - B1	199.4	2923a25	2x	<0.02	<4				<70	2928a20	2x	2.88	0.57			0.40
5877 - B1	185.7	2923a28	2x	0.03±0.04	5±7				(90±100)	2928a12	50x	1310	217			152
5953 - B1	152.2	2923a22	2x	0.08±0.01	12±2				(200±30)	2923a22	2x	896	136			95.3

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200-BP-1 HOT SOIL ANALYSIS

Tc-99 and Uranium determinations

Sep. 30, 1992

PNL Sample I.D. # (a)	Digest. Dil. Factor (b)	ICP/MS Analysis No.	Analysis Dilution Factor	[Tc-99] ng/ml leachate (d)	[Tc-99] ng/g soil	Ru Correctn Factor	[Tc-99] ng/g spiked	[Tc-99] spike yield, %	[Tc-99] pCi/g, dry soil
5954 - B1	134.3	2923a27	2x	0.08	10				(200)
6428 - B1	148.9	2921a28	2x	0.02	3				(50)
6713 - B1	189.4	2928a6	2x	<0.02	<4				<70
6726 - B1	187.0	2921a22	2x	0.03	5	0.367			80
6727 - B1	194.2	2921a24	2x	0.03	5				(80)
6904 - B1	184.3	2921a29	2x	<0.02	<4				<70
6963 - B1	175.9	2921a6	2x	<0.02	<4				<60
6964 - B1	191.6	2921a32	2x	<0.02	<4				<70
7040 - B1	193.1	2921a23	2x	<0.02	<4				<70
7141 - B1	176.6	2921a21	2x	<0.02	<4				<70
7142 - B1	145.7	2921a7	2x	<0.02	<3				<50
1%HNO3		2923a30	1.053X	<0.01					
1%HNO3		2921a1	1.053X	<0.01					
1%HNO3		2921a5	1.053X	<0.01					
1%HNO3		2921a25	1.053X	<0.01					
1%HNO3		2921a34	1.053X	<0.01					
HNO3/ H20 Blank		2921a8	2x	<0.01					
50 ppt Tc-99		2925a9	1.053X	0.05					
50 ppt Tc-99		2928a14	1.053X	0.05					
100ppt Tc-99		2921a16	1.053X	0.10					
100ppt Tc-99		2921a20	1.053X	0.09					
500ppt Tc-99		2921a11	1.053X	0.46					
500ppt Tc-99		2921a26	1.053X	0.49					
7.6 pCi/L U									
7.6 pCi/L U									
7.6 pCi/L U									
41.0 pCi/L U									
41.0 pCi/L U									
41.0 pCi/L U									
LCS (267)		2925a10	2x	0.02					
Process Blank		2925a11	2x	<0.02					
"Spike"		2925a17	2x	0.12					

ICP/MS Analysis No.	Analysis Dilution Factor	[U] ng/ml leachate (d)	[U] µg/g soil	[U] µg/g spiked	[U] spike yield, %	[U] pCi/g, dry soil
2923a27	2x	652	67.6			61.2
2922a16	20x	3260	488			341
2922a7	5x	409	77.5			54.1
2922a8	2x	146	27.3			19.1
2922a24	2x	138	26.8			18.7
2922a12	2x	36.6	6.75			4.71
2922a20	5x	449	79.0			55.2
2922a13	2x	211	40.4			28.2
2922a14	2x	2.88	0.56			0.39
2922a19	20x	1730	306			213
2922a6	5x	375	54.6			38.2
2925a1	1.053X	<0.05				
2925a23	1.053X	<0.05				
2925a29	1.053X	<0.05				
2923a1	1.053X	<0.05				
2923a14	1.053X	0.30				
2922a10	2x	0.06				
2922a15	1.053X	7.81				
2923a15	1.053X	7.56				
2928a15	1.053X	8.00				
2922a31	1.053X	41.9				
2923a29	1.053X	40.0				
2925a34	1.053X	42.2				
2925a10	2x	69.3				
2925a11	2x	<0.05				
2925a17	2x	<0.05				

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200-BP-1 HOT SOIL ANALYSIS

Tc-99 and Uranium determinations

Sep. 30, 1992

PNL Sample I.D. # (a)	Digest. Dil. Factor (b)	ICP/MS Analysis No.	Analysis Dilution Factor	[Tc-99] ng/ml leachate (d)	[Tc-99] ng/g soil	Ru Correctn Factor	[Tc-99] ng/g spiked	[Tc-99] spike yield, %	[Tc-99] pCi/g, dry soil	ICP/MS Analysis No.	Analysis Dilution Factor	[U] ng/ml leachate (d)	[U] µg/g soil	[U] µg/g spiked	[U] spike yield, %	[U] pCi/g, dry soil

(a) Sample types:
B1 = sample
B2 = sample, duplicate
B3 = sample + Tc/U spike
B4 = laboratory control sample
B5 = procedural blank
B6 = procedural blank + Tc/U spike

(b) units of mL/g dry soil except for -B5 and -B6 samples (blank/blank spike) units of mL.
(c) additional dilution for analysis
(d) = analyzed concentration X analysis dilution factor
(e) = leachate concentration X digestion dilution factor
(f) -B3 reported in units of ng/g for Tc and µg/g for U; -B6 reported in ng for Tc and µg for U.
(g) estimated fraction of mass 99 actually due to technetium based on natural Ru isotopic abundance.
Because Ru isotopic abundance is suspected to be unnatural, this factor was not used in calculating the final [Tc] result.
(h) = ([Spiked Sample] - [Sample]) / [Spike]
(i) not valid for spiked samples; [U] in soil/leachate >> [U] of added spike
(j) all uranium found to be natural isotopic abundance. Total uranium activity calculated using following formula: pCi/g = [U-238]*0.336/0.481

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