

START

0011967⁹⁸

SST WASTE CHARACTERIZATION PROJECT

CORE 1 DATA REPORT

Revision 2

May 14, 1990

Prepared by: T. E. Jones



Battelle, Pacific Northwest Laboratory

91120511770

THIS PAGE
INTENTIONALLY
LEFT BLANK



TABLE OF CONTENTS

Contributors List	1
Introduction	2
Physical Data	7
Core 1, Segment 2-5 Extrusion Photographs	8
Description of Extruded Segments	12
Rheological Characterization	15
Thermal Analysis	18
Particle Size for Unhomogenized Segments	28
Inorganic Data Tables	33
Inorganic Fusion ICP Results	34
Inorganic Acid Leach ICP Results	43
Inorganic Water Leach ICP Results	52
Inorganic Water Leach Anion Analysis Results	54
Other Inorganic Results	60
Radiochemical Data Tables	62
Radiochemical Results	63
Organic Data Tables	71
Core 1 Semivolatile Compound Analysis Report	72
Core 1 Volatile Organic Compound Analysis Report	75

9112091179

TABLE OF CONTENTS

APPENDICES: Primary Analytical Data

Appendix A: Test Instructions

Appendix B: Chain of Custody

B1 - Westinghouse Chain of Custody Forms

B2 - 325A Hot Cell Transfer Forms

B3 - 325B Hot Cell Chain of Custody/Sample Prep Data Sheets

Appendix C: Physical Properties

C1 - Introduction

C2 - Laboratory Analyst Signature List

C3 - 325A Hot Cell Extrusion/Characterization LRB

C4 - 325B Hot Cell SST Characterization Data LRB

C5 - Percent Solids Data

C6 - pH Data

Appendix D: Radiochemical

D1 - Introduction

D2 - Laboratory Analyst Signature List

D3 - Table D1: Radiochemical Serial No. Vs Customer No.

D4 - GEA Analysis

D5 - Alpha Analysis

D6 - Beta Analysis

D7 - Total Uranium by Laser Fluorometry

D8 - Iodine 129 by Gamma Spectrometry

D9 - Tritium

9112051170

TABLE OF CONTENTS

Appendix D (Cont'd):

D10 - Carbon 14 Analysis

D11 - U and Pu Isotopic Analysis by Mass Spectrometry

D12 - Pipet Calibration Data

Appendix E: Inorganic

E1 - Introduction

E2 - Laboratory Analyst Signature List

E3 - ICP Analysis

E4 - IC/TOC/TIC/TC

E5 - Atomic Absorption (AA)

E6 - Chromium (IV)

E7 - Ammonia (NH₃)

E8 - Mercury (Hg)

Appendix F: Organic

F1 - Introduction

F2 - Laboratory Analyst Signature List

F3 - Core 1 Composite Semivolatile Analysis Data

F4 - Core 1 Volatile Analysis Data

F5 - Hexadecane Screens

F6 - Headspace Screens

91120541701

TABLES

Table 1: SST Core 1 Sample Numbers	4
Table 2: SST Core 1 Sub-Sample Numbers	5
Table 3: SST Core 1 Prehomogenization Segment Data	13
Table 4: SST Core 1 Composite Physical Data	14
Table 5: SST Core 1 Composite Thermal Analysis Data	20
Table 6: SST Core 1 Water Leach pH Values	26
Table 7: SST Core 1 Weight Percent Solid Values	27
Table 8.1: SST Core 1, Segments 2 & 3 Fusion ICP Homogenization Check	36
Table 8.2: SST Core 1, Segments 3 & 4 Fusion ICP Homogenization Check	37
Table 8.3: SST Core 1, Segment 5 Fusion ICP Homogenization Check . . .	38
Table 8.4: SST Core 1, Segment 2 Fusion ICP	39
Table 8.5: SST Core 1, Composite 1 Fusion ICP	40
Table 8.6: SST Core 1, Composite 2 Fusion ICP	41
Table 8.7: SST Core 1, Segment 2 Acid Leach ICP	45
Table 8.8: SST Core 1, Segment 3 Acid Leach ICP	47
Table 8.9: SST Core 1, Segments 4 & 5 Acid Leach ICP	49
Table 8.10: SST Core 1, Composite Acid Leach ICP	50
Table 8.11: SST Core 1, Composite Water Leach ICP	53
Table 9.1: SST Core 1, Segments 2, 3 & 4 Anion Analyses by IC/TOC Data	55
Table 9.2: SST Core 1, Seg 5 & Composite Anion Analyses by IC/TOC Data	58
Table 10: SST Core 1, Composite 1, Other Inorganic Analyses	61
Table 11: SST Core 1 Gamma Energy Analysis Data	64
Table 12: SST Core 1 Radiochemistry Data	68

9112511712

FIGURES

Figure 1: Viscosity of the Homogenized 110B Core 1 Composite	17
Figure 2: DSC Plot Core 1 Composite, Run 1	21
Figure 3: DSC Plot Core 1 Composite, Run 2	22
Figure 4: Core 1 Composite Thermogravimetric Curve, Run 1	23
Figure 5: Core 1 Composite Thermogravimetric Curve, Run 2	24
Figure 6: Core 1 Composite Thermogravimetric Curve, Run 3	25

9 1 1 2 7 5 1 1 7 3

THIS PAGE
INTENTIONALLY
LEFT BLANK

CONTRIBUTORS LIST

PROJECT MANAGEMENT OFFICE

TE Jones, Project Manager
SL English, Quality Engineer
BM Gillespie
SG McKinley
TM Longaker
MK Allen
JA Gibson

SAMPLE PREPARATION AND
PHYSICAL TESTING

RD Scheele, Task Leader
RT Steele, Task Leader
TK Andrews
DV Archer
JL Green
FV Hoopes
ME Peterson
GM Richardson
EH Shade
JM Tingey

RADIOCHEMICAL ANALYSIS

JH Kaye, Task Leader
NL Abbey
RE Brinson
VL Dunn
BM Gillespie
MW Goheen
FE Holt
LS Kellog
AE Kozelisky
TC Maiti
PJ Raney
JM Ruggles
JS Schmitt
SW Thompson
NL Wynhoff

INORGANIC ANALYSIS

MW Urie, Task Leader
MC Burt
JH Ennen
DE Rinehart
DL Thomas
ML Thomas
TG Walker

ORGANIC ANALYSIS

RW Stromatt, Task Leader
DL Bellofatto
EW Hoppe
MM McBurney

9 1 1 2 0 5 1 1 7 0 4

THIS PAGE
INTENTIONALLY
LEFT BLANK

INTRODUCTION

9 1 1 2 0 5 1 1 7 5

This data package contains results obtained by Pacific Northwest Laboratory (PNL) staff in the characterization and analyses of Core 1 segments taken from the single-shell tank (SST) 110B. The characterization and analysis of Core 1 segments are outlined in the Waste Characterization Plan for Hanford Site Single-Shell Tanks and in the Pacific Northwest Laboratory Single-Shell Tank Waste Characterization Support FY 89/90 Statement of Work (SOW), Rev. 0 dated September, 1989. Specific analyses for each sub-sample taken from a segment are delineated in Test Instructions (TI) prepared by the PNL SST Waste Characterization Project Management Office (SST Project) in accordance with procedures contained in the SST Waste Characterization Procedure Compendium (PNL-MA-599). Analytical procedures used in the characterization activities are also included in PNL-MA-599. This data package contains no technical assessment of the chemical analysis. However, some discussion of physical measurements is included.

Core 1 included five segments. The five samplers were received from Westinghouse Hanford Company (WHC) between 8/9/89 and 8/15/89. Each segment was contained in a sampler which was enclosed in a shipping cask. The shipping cask was butted up to the 325A hot cell and the sampler moved in the cell. The material in the sampler (i.e., the segment) was extruded, photographed, and other physical characteristics assessed. At this point, samples were taken for particle size and volatile organic analyses. Each segment was then homogenized and sub-samples taken for required analyses as delineated in the appropriate TI. Sub-samples were then taken from the homogenized segments and used to make a core composite sample. Required analyses for the core composite are delineated in the appropriate TI.

A complete segment was expected to be ~19 inches in length and to contain ~250 grams of material. The segment 1 sampler contained only ~2 inches of material. As per the guidance outlined in the SOW, no characterization work was completed on segment 1. The other four Core 1 samplers contained the expected amount of material.

Requested analyses for Core 1 homogenized segments included: Weight Percent Solids; inductively-coupled plasma atomic emission spectroscopy (ICP) analysis; gamma energy analysis (GEA); total alpha, total beta, and uranium analysis in duplicate, from samples that have been fused with potassium

hydroxide (KOH); ICP on duplicate samples from an acid leach as per EPA protocols; and anion analysis, total organic carbon, and pH from a water leach procedure. Analyses requested for the core composite included all of the analyses outlined above plus an extensive array of radiochemical, inorganic, and semivolatile organic analyses.

The data within this package are divided into four groups: physical testing, inorganic analysis, radiochemical analysis, and organic analysis. Specific data within a group are separated by individual segment or the core composite. All inorganic chemical analysis data are reported on a per wet-weight basis. This is, no corrections were made for the weight percent water in the samples. Radiochemical results are reported as decays per minute per gram (dpm/g), microgram per gram ($\mu\text{g/g}$), or microcuries per gram ($\mu\text{Ci/g}$), depending on the specific analysis. All sample preparations were completed in duplicate, thus, duplicate analysis data are available for all samples. The quality control (QC) requirements for each sample are defined in specific TIs. To the extent practical, the QC requirements outlined in EPA documents SW-846 and the CLP-SOW were followed. All QC data are included in this data package.

A number of analyses were requested in the SOW for which data are not included in this data package. These include TOX, cyanide, and nickel-63 analyses on Core I composite samples. The missing data will be provided at a later date.

9112051176

TABLE 1: SST Core 1 Sample Numbers

<u>Segment #</u>	<u>Prehomogenized Segment</u>	<u>Homogenization Test</u>	<u>Homogenization Analyses</u>	<u>Composite Test</u>	<u>Composite Analyses</u>
2	89-0322 89-0325	89-0331 89-0332	89-0648		
3	89-0323 89-0326	89-0333 89-0334	89-0649		
4	89-0324 89-0327	89-0335 89-0336	89-0650		
5	89-0329 89-0330	89-0337 89-0338	89-0651		
Composite				89-0622 89-0623 89-1125 89-1126	89-0621 89-0980 89-0981

911205117.07

TABLE 2: SST Core 1 Sub-Sample Numbers

<u>Sample Prep Method</u>	<u>Primary Sample Number</u>	<u>Duplicate Sample Number</u>	<u>Spike Sample Number</u>	<u>Spike Control Sample Number</u>	<u>Blank Sample Number</u>
Volatile	89-0322				
	89-0323				
	89-0324				
	89-0329				
Particle Size	89-0325				
	89-0326				
	89-0327				
	89-0330				
Fusion	89-0331-A-1	89-0331-B-1			
	89-0332-A-1	89-0331-B-1			
	89-0333-A-1	89-0333-B-1			
	89-0334-A-1	89-0334-B-1			
	89-0335-A-1	89-0335-B-1			
	89-0336-A-1	89-0336-B-1			
	89-0337-A-1	89-0337-B-1			
	89-0338-A-1	89-0338-B-1			
	89-0622-A-1	89-0622-A-2			
	89-0622-B-1	89-0622-B-2			
	89-0623-A-1	89-0623-A-2			
	89-0623-B-1	89-0623-B-2			
	89-0648-A-1	89-0648-A-2			89-0648-A-3
	89-0649-A-1	89-0649-A-2			89-0649-A-3
	89-0650-A-1	89-0650-A-2			89-0650-A-3
	89-0651-A-1	89-0651-A-2			89-0651-A-3
	89-0980-A-1	89-0980-B-1			
	89-0980-A-2	89-0980-B-2			
	89-0981-A-1	89-0981-B-1			
	89-0981-A-2	89-0981-B-2			
90-1125-A-1	90-1125-A-2			90-1125-A-3	
90-1126-A-1	90-1126-A-2			90-1126-A-3	
Acid Leach	89-0621-A-1	89-0621-A-2	89-0621-A-3	89-0621-A-4	89-0621-A-5
	89-0621-B-1	89-0621-B-2	89-0621-B-3	89-0621-B-4	89-0621-B-5
	89-0648-B-1	89-0648-B-2	89-0648-B-3	89-0648-B-4	89-0648-B-5
	89-0649-B-1	89-0649-B-2	89-0649-B-3	89-0649-B-4	89-0649-B-5
	89-0650-B-1	89-0650-B-2	89-0650-B-3	89-0650-B-4	89-0650-B-5
	89-0651-B-1	89-0651-B-2	89-0651-B-3	89-0651-B-4	89-0651-B-5

91120511709

TABLE 2: SST Core 1 Sub-Sample Numbers (Cont'd)

<u>Sample Prep Method</u>	<u>Primary Sample Number</u>	<u>Duplicate Sample Number</u>	<u>Spike Sample Number</u>	<u>Spike Control Sample Number</u>	<u>Blank Sample Number</u>
Water Leach	89-0621-C-1	89-0621-C-2	89-0621-C-3	89-0621-C-4	89-0621-C-5
	89-0648-C-1	89-0648-C-2	89-0648-C-3	89-0648-C-4	89-0648-C-5
	89-0649-C-1	89-0649-C-2	89-0649-C-3	89-0649-C-4	89-0649-C-5
	89-0650-C-1	89-0650-C-2	89-0650-C-3	89-0650-C-4	89-0650-C-5
	89-0651-C-1	89-0651-C-2	89-0651-C-3	89-0651-C-4	89-0651-C-5
Mercury	89-0621-D-1	89-0621-D-2	89-0621-D-3	89-0621-D-4	89-0621-D-5
Semi-Volatile	89-0621-E-1	89-0621-E-2	89-0621-E-3	89-0621-E-4	89-0621-E-5
EOX/TOX	89-0621-F-1	89-0621-F-2	89-0621-F-3		89-0621-F-4
Cyanide	89-0621-G-1	89-0621-G-2	89-0621-G-3	89-0621-G-4	89-0621-G-5
Wt % Solids	89-0648-D-1	89-0648-D-2			
	89-0649-D-1	89-0649-D-2			
	89-0650-D-1	89-0650-D-2			
		89-0651-D-1	89-0651-D-2		

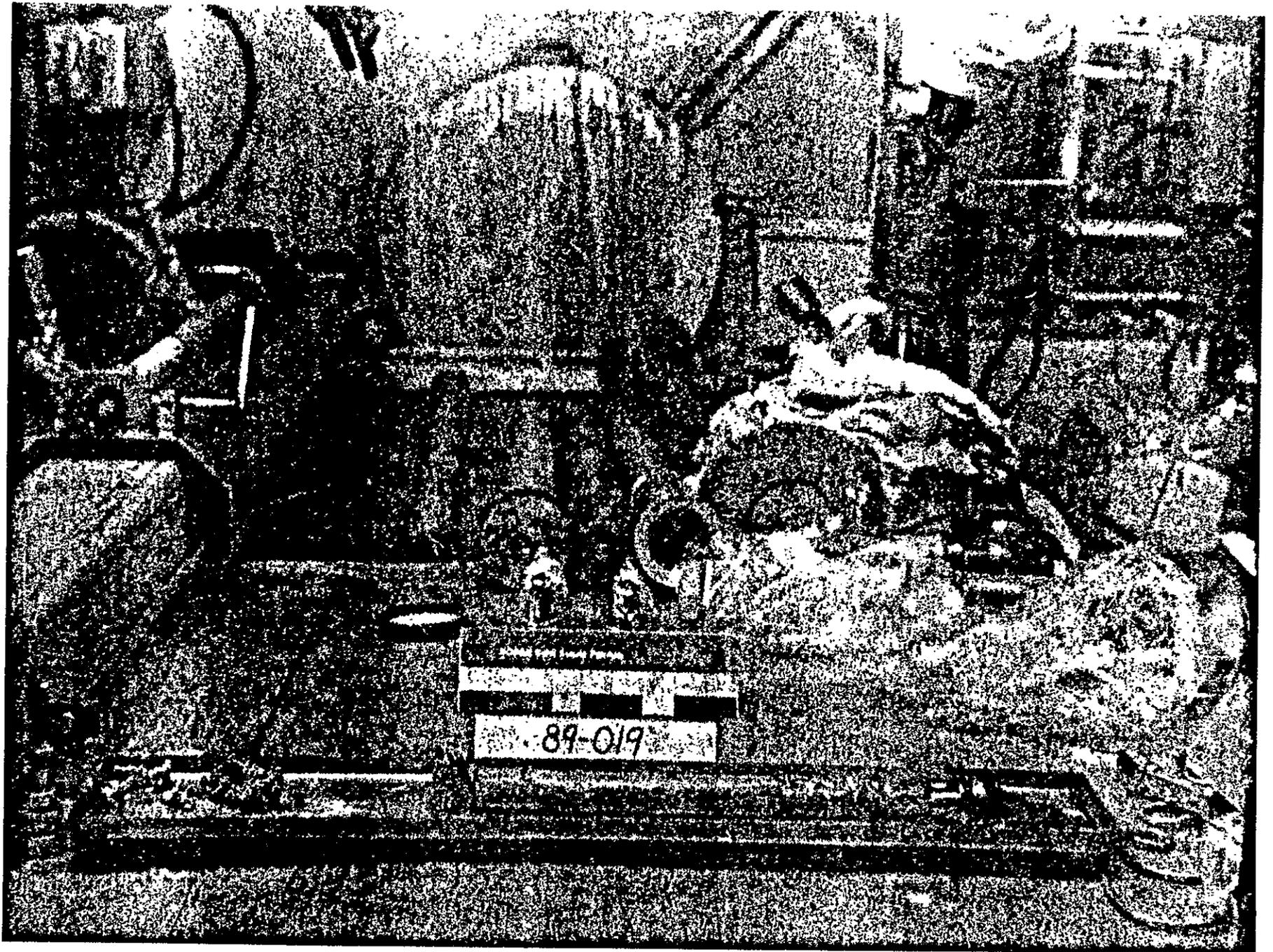
9112051179

THIS PAGE
INTENTIONALLY
LEFT BLANK

PHYSICAL DATA TABLES

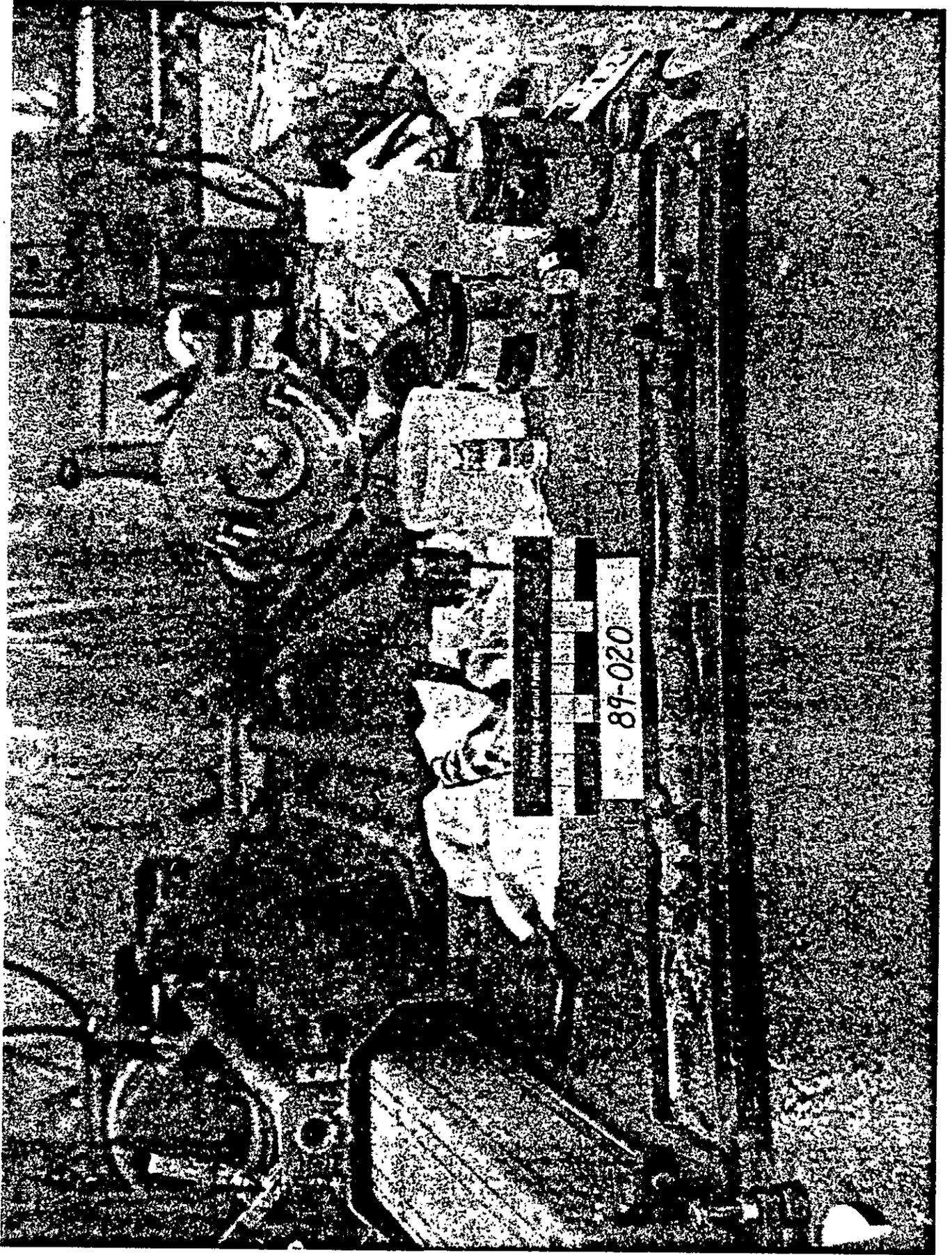
9 1 1 2 7 5 1 1 7 0 0

9 1 1 2 0 5 1 1 7 0 1



Core 1, Segment 2

91120541702



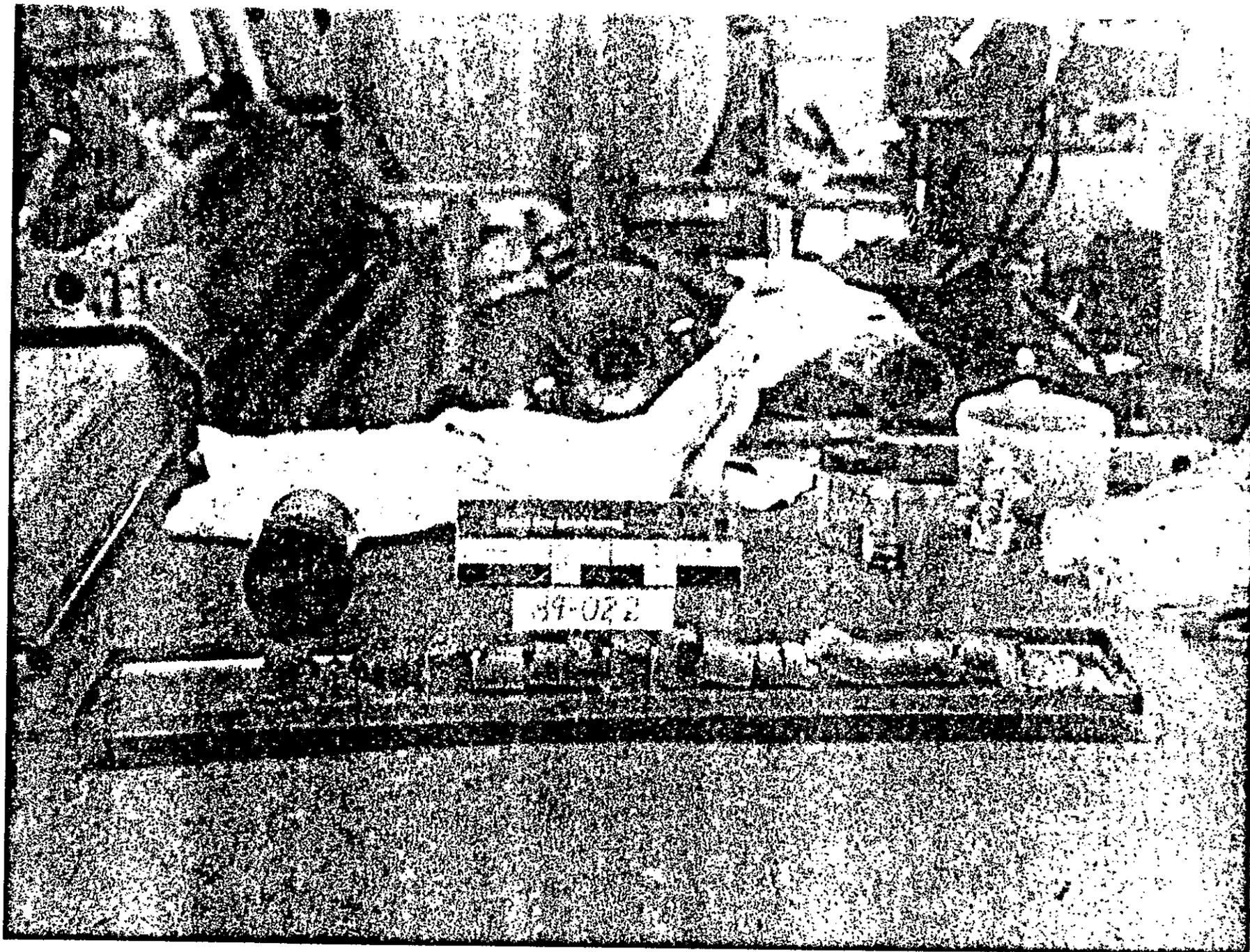
Core 1, Segment 3

9 1 1 2 0 5 1 1 7 0 3



Core 1, Segment 4

9 1 1 2 2 5 1 1 7 2 4



Core 1, Segment 5

THIS PAGE
INTENTIONALLY
LEFT BLANK

DESCRIPTION OF EXTRUDED SEGMENTS

Five Core 1 samplers from tank 110B riser 7 were received and extruded. The mass, volume, density, and penetration resistance for each of the segments extruded is given in Table 3. The first segment was only 2 inches long and contained only 33.1 grams of slurry; therefore, this segment was an invalid segment and only limited data exists for Core 1 segment 1. Also, no drainable liquids nor prehomogenized slurries were obtained from this core; therefore, no data on drainable liquids or prehomogenized slurries for core 3 exists. The penetration resistance of the prehomogenized segments was < 2 psi for all the segments; therefore, the entire core can be considered cohesive. The bulk densities of the segments were all 1.3 g/ml. Photographs of each extruded segment are found on pages 8 to 11.

As can be seen in these photographs, all of the segments were sludges with varying colors and amounts of interstitial liquid. All of the sludges held their shape upon extrusion. Segment 1 was a brown sludge. The outer shell of this segment was much drier than the interior portion of the segment.

The top two to three inches of segment 2 were brown in color and was more fluid than the remainder of the segment. The remainder of this segment was a solid tan sludge.

The top of segment 3 was more fluid than the bottom portion, but still held its shape. The top three inches of segment 3 are brown in color with the remainder of the segment being light tan in color.

Segment 4 was a light tan sludge which upon extrusion broke into approximately 1 inch pieces.

Segment 5 was a tan sludge somewhat darker than segment 4 which gradually turned to a brown sludge 5 inches from the bottom of the core. The consistency of this segment was similar to that of segment 4 and most of the segment broke into 1 inch pieces.

TABLE 3: Core 1 Prehomogenized Segmen. Data

<u>Segment</u>	<u>Mass (g)</u>	<u>Volume (ml)</u>	<u>Density (g/ml)</u>	<u>Penetration Resistance (psi)</u>
1	33	25	1.3	N/A
2	315	245	1.3	< 2
3	310.87	245	1.3	< 2
4	327.70	245	1.3	< 2
5	326.95	245	1.3	< 2

9112511776

TABLE 4: SST Core 1 Composite Physical Data

1) Bulk Slurry Density	7) Wt% Total Solids (Gravimetric)
1.355 g/ml	40.6%
1.365 g/ml	40.9%
AVE = 1.360 ± 0.007 g/ml	AVE = 40.8 ± 0.21%
2) Centrifuged Solids Density	8) Wt% Undissolved Solids
1.53 g/ml	21.1%
1.52 g/ml	Calculated from wt% total and
1.60 g/ml	wt% dissolved solids.
1.56 g/ml	
AVE = 1.55 ± 0.036 g/ml	
3) Centrifuged Supernate Density	
1.24 g/ml	
1.25 g/ml	
AVE = 1.25 ± 0.007 g/ml	
4) Wt% Centrifuged Solids	
49.1%	
45.5%	
45.7%	
47.4%	
AVE = 46.9 ± 1.68%	
5) Vol % Centrifuged Solids	
41.3%	
41.8%	
AVE = 41.6 ± 0.4%	
6) Wt % Dissolved Solid	
18.6%	
20.7%	
AVE = 19.7% ± 1.1%	

91120511707

RHEOLOGICAL CHARACTERIZATION

9 1 1 2 5 1 7 3

The rheological characterizations (shear stress versus shear rate) were measured on the homogenized 110-B Core 1 composite. The data obtained from the rheological characterizations is important for retrieval operation and is used to evaluate the viscosity of a fluid and to assess the ability to transport the waste in pipes. The data are generated in the form of a rheogram or flow curve, which is a plot of shear stress as a function of shear rate. The rheograms were obtained using a Haake RV 100 viscometer equipped with an M5 measuring-drive head and the MVI sensor system. The measurement of viscosity with this instrument requires that the sample be placed in the gap between two coaxial cylinders. About 40 mL of sample is agitated and transferred into the cylinders. When the system is set in motion, a viscosity-related torque, caused by the sample's resistance to shearing, acts on the inner cylinder. This torque deflects a calibrated measuring spring placed between the motor and the inner cylinder. The magnitude of the spring deflection correlates linearly with the torque. The spring deflection is transformed into an electrical signal. The spring deflection and tachometer signals are recorded. This data is used to calculate the shear stress versus shear rate.

A slurry with a yield stress will "clamp" the rotor to the cup until the applied torque exceeds the yield stress. While the rotor is still "clamped" and remains motionless, the motor rotation will cause some spring deflection and consequently a torque signal which is recorded. When sufficient force is transmitted to the material to break the gel or make it yield, the rheogram will angle sharply to the right, and from then on the behavior of the material as a fluid will be recorded. This sharp angle that is recorded as the material becomes fluid is the yield point. The height of this peak measured in Pascals on the ordinate is the value of the yield stress.

Two rheograms were obtained for the sample of the homogenized Core 1 composite. The measurements were obtained at the cell temperature of 31°C. The viscosity changed with shear rate and therefore, the waste is classified as a non-Newtonian fluid. The homogenized Core 1 composite had a density of 1.36 g/mL, contained 21.1 wt% total undissolved solids and 41.6 vol% centrifuged solids. The sample of waste was thoroughly agitated prior to

obtaining the first rheogram. The sample was not agitated prior to the second measurement. Settling of the solids in the cylinders may occur between measurements. The homogenized Core 1 composite settled very slowly and therefore, the effect of not agitating in between measurements may not be significant for this particular waste. However, in future characterizations, the sample will be agitated prior to each measurement to assure that the second rheogram is obtained under the same conditions as the first rheogram.

The homogenized 110-B Core 1 composite exhibited a yield stress, and therefore, the data from the rheogram were "fit" to a yield-pseudoplastic model. The rheological models obtained from the two rheograms of this waste were:

$$\begin{aligned}\tau &= 4.83 + 0.0448(\dot{\gamma})^{0.8817} \\ \tau &= 3.80 + 0.0258(\dot{\gamma})^{0.9851}\end{aligned}\tag{3}$$

where τ = shear stress, Pa
 $\dot{\gamma}$ = shear rate, sec^{-1} .

Figure 1 shows the viscosity versus shear rate for the homogenized core #1 composite. A shear rate of 250 sec^{-1} corresponds to a velocity of 8 ft/sec in a 3-in.-diameter pipe. The viscosity of this waste decreases with increasing velocity (ie. shear rate).

The rheological parameters, together with the density, were input into the Hanks' computer model (Hanks 1978) to obtain the critical Reynolds number, critical velocity, friction factor and pressure drop for transporting the slurries in 4.0-in., 3.0-in. and 2.0-in.-diameter pipes. For a 4.0-in.-diameter pipe, the critical Reynolds number for both rheological models was 6600 and the critical velocity ranged from 3.8 ft/sec (150 gpm) to 4.2 ft/sec (166 gpm). For a 3.0-in.-diameter pipe, the critical Reynolds number ranged from 5500 to 5700 and the critical velocity ranged from 4.1 ft/s (95 gpm) to 4.5 ft/sec (105 gpm). For a 2.0-in.-diameter pipe, the critical Reynolds number ranged from 4300 to 4600, and the critical velocity ranged from 4.7 ft/sec (50 gpm) to 5.1 ft/sec (54 gpm).

The rheological characterizations were performed under PNL Quality Assurance Manual MA-70, Impact Level II requirements. The Hanks' computer

9
1
1
7
9

model is a purchased computer program but has not been verified internally by PNL. Therefore, the computer model is being used in accordance with MA-70, Impact Level III requirements.

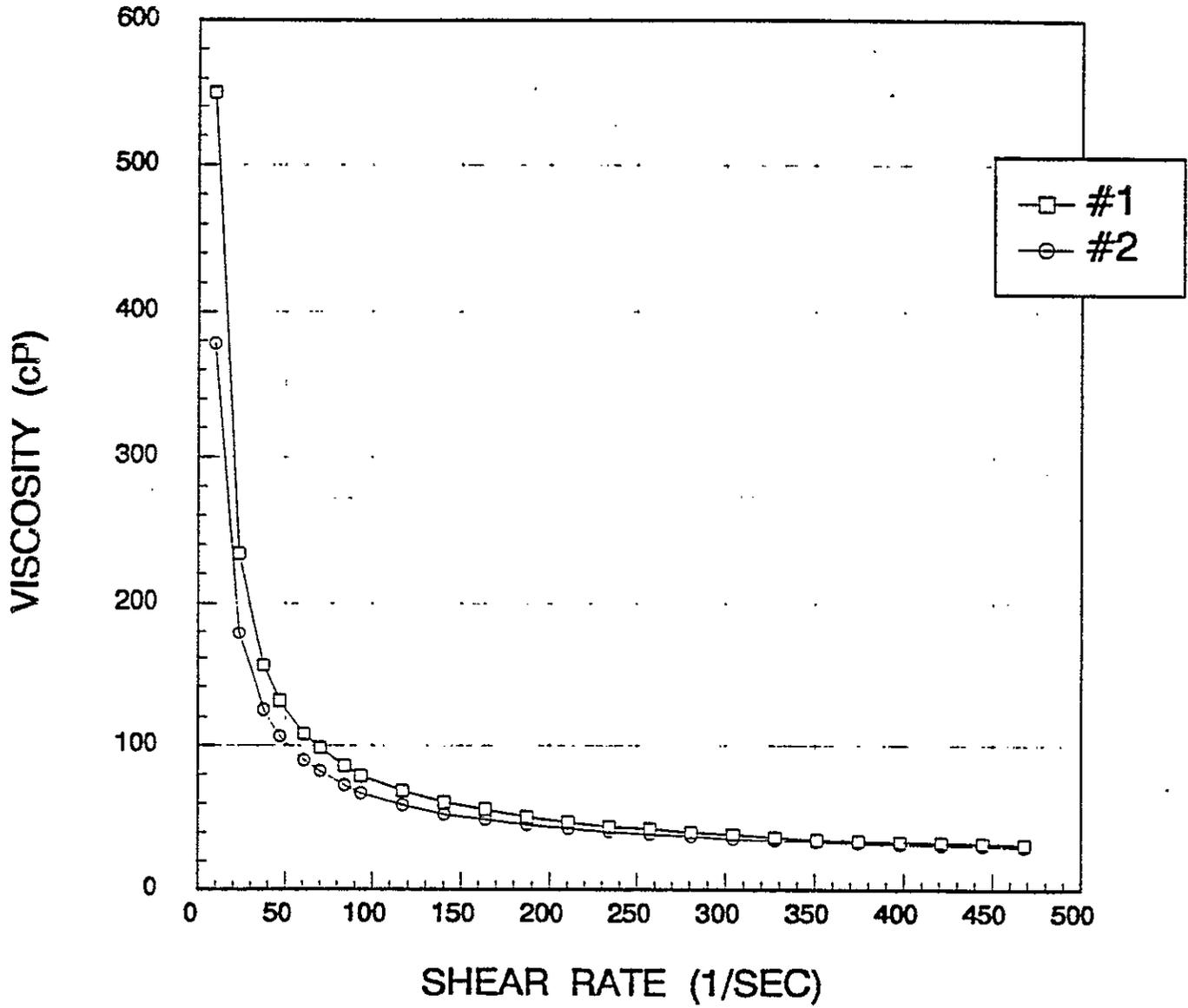


FIGURE 1. Viscosity of the Homogenized 110-B Core 1 Composite

THERMAL ANALYSIS

Scanning thermogravimetry and differential scanning calorimetry were performed in duplicate on the homogenized Core 1 composite. Differential Scanning Calorimetry (DSC) and Scanning Thermogravimetry (STG) are two thermal analysis techniques that are useful for determining the thermal stability or reactivity of a material. DSC measures heat released or absorbed while the temperature of the sample is increased at a constant rate. STG measures the mass of a sample while the temperature of the sample is increased at a constant rate. Both methods can be modified to measure isothermal changes in the material. DSC is often used to measure thermal decomposition temperatures, heats of reaction, reaction temperatures, melting points, and solid-solid transition temperatures. STG is used to measure thermal decomposition temperatures, water contents, and reaction temperatures. The two methods often provide complimentary information. The calibration of the differential scanning calorimeter and the thermogravimetric analyzer were checked before running these samples. A Indium standard was run on the calorimeter to check the temperature and enthalpy calibrations. The balance calibration of the thermogravimetric analyzer was checked with a 100 mg standard weight, and the temperature calibration of the analyzer was checked with an alumel/nico seal curie point magnetic transition standard.

Figures 2 and 3 are the DSC plots for the Core 1 composite material runs 1 and 2, respectively. As is shown in Table 5, both runs of the core composite material exhibited three major transitions with maximums around 80, 120, and 290°C. The temperature range, onset temperature, and enthalpy change for each transition are given in Table 5.

Transitions 1 and 2 are indicative of at least two types of water. These two transitions have broad peaks which cannot be resolved; therefore, the temperature range, onset temperature, and enthalpy changes for these transitions are only approximations. The temperature range and enthalpy change for the combination of these water transitions are also given in Table 5. The variance in the temperature range of the combined transitions between runs 1 and 2 may be due to differences in their heating rates. Run 1 was made at a heating rate of 5°C/min while the heating rate for run 2 was 10°C/min.

Increasing the heating rate in run 2 may have caused the transitions to be smeared over a larger temperature range. Variances in the enthalpy changes between runs 1 and 2 may be due to the difficulty in determining the actual baseline for this portion of the thermogram; since the water appears to begin to evaporate even before the temperature is increased. This premature evaporation makes it difficult to determine the baseline of these transitions. There may also be other smaller transitions in this temperature region which are obscured by these broad peaks.

Thermogravimetric measurements of this core composite confirm the fact that at least two types of water exist. Figures 4, 5 and 6 are thermogravimetric curves for the core composite material. At the heating rates at which these curves were recorded, the two water loss steps cannot be separated; but it is evident that the water is lost in at least two distinct steps. Transition 1 appears to be a loss of free water from the core material. This transition has a temperature range from below 50°C to approximately 100°C. Most of the water in the sample is lost in this transition step. The second transition occurs over a temperature range from approximately 110 to 150°C. This second transition may be the loss of waters of hydration. Further measurements are being performed to determine the salts to which the water molecules are coordinated.

It is also evident that all the water cannot be driven from the sample at 105°C, the temperature used in the total solids measurements. Two isothermal runs at 105°C also indicate that all the water will not be lost at 105°C. Some consideration should be taken in deciding which temperature should be used to determine the wt% total solids in these types of material.

As is evidenced by the immediate loss of water at the starting temperature of 50°C, it is essential that the thermogravimetric curves be started at a lower temperature. A comparison of the thermogravimetric data with the total solids measurements made in the drying oven indicate that less water is being driven off by the thermogravimetric method. This discrepancy may be due to water loss from the sample before it can be weighed, because the sample is weighed at an elevated temperature (50°C). We are in the process of developing a method to begin these curves at ambient or below ambient temperatures in order to accurately measure the amount of water in the sample.

TABLE 5: SST Core 1 Composite Thermal Analysis Data

Differential Scanning Calorimetry

	<u>Run 1</u>	<u>Run 2</u>
Transition 1		
Temp. Range (°C)	25 to 83	25 to 120
Max. Temp. (°C)	74	66
Onset Temp. (°C)	48	27
Enthalpy Change (cal/g)	200	92
Transition 2		
Temp. Range (°C)	108 to 165	95 to 220
Max. Temp. (°C)	121	123
Onset Temp. (°C)	114	117
Enthalpy Change (cal/g)	90	130
Transitions 1 and 2 Combined		
Temp. Range (°C)	25 to 165	25 to 220
Enthalpy Change (cal/g)	290	227
Transition 3		
Temp. Range (°C)	268 to 316	275 to 310
Max. Temp. (°C)	290	290
Onset Temp. (°C)	273	279
Enthalpy Change (cal/g)	11	6

Thermogravimetry

	<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>
Weight % Solids	47.98%	47.88%	48.36%

91120541303

9 1 1 2 5 1 1 9 0 4

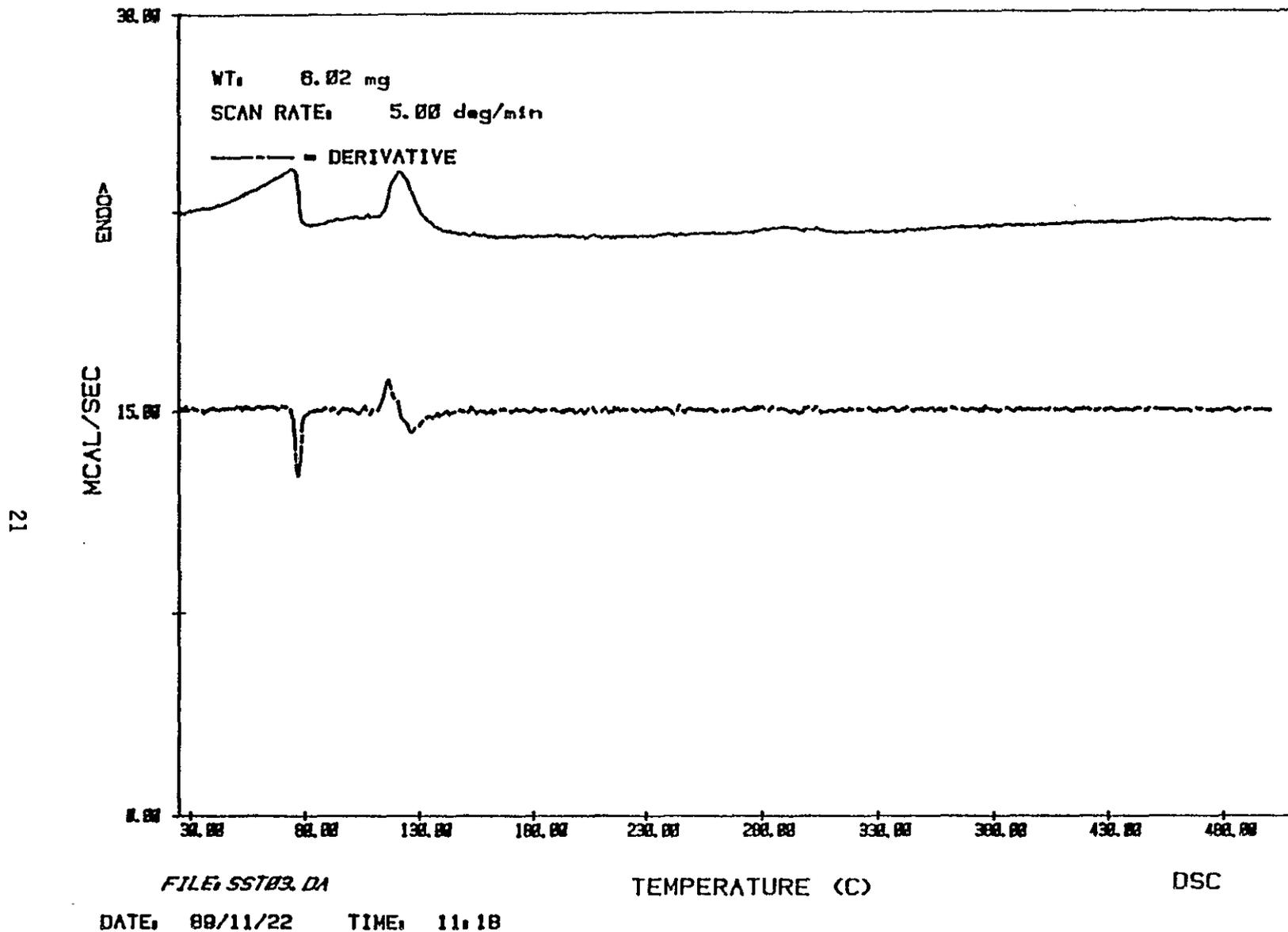


FIGURE 2. DSC Plot Core 1 Composite, Run 1

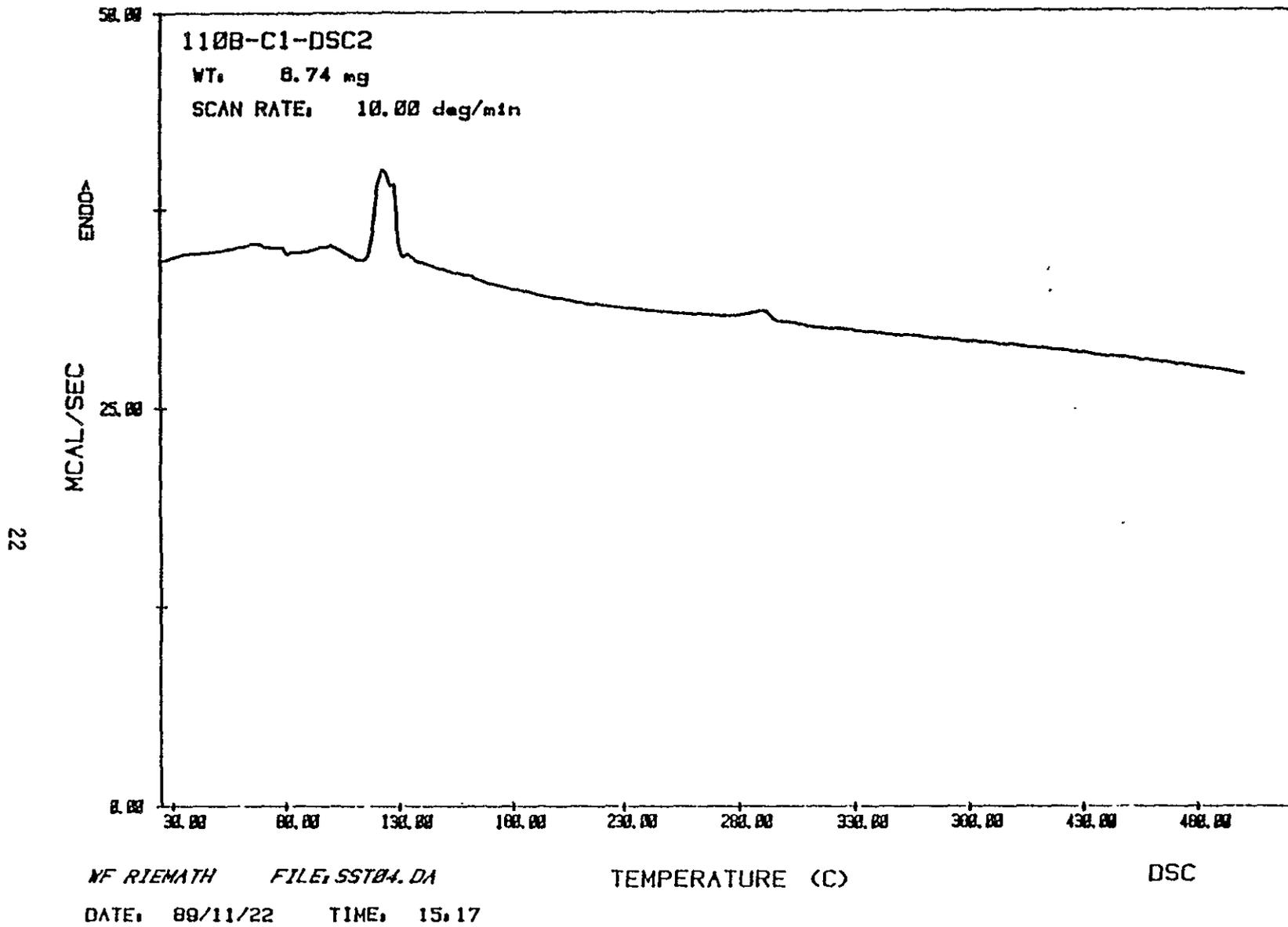


FIGURE 3. DSC Plot Core 1 Composite, Run 2

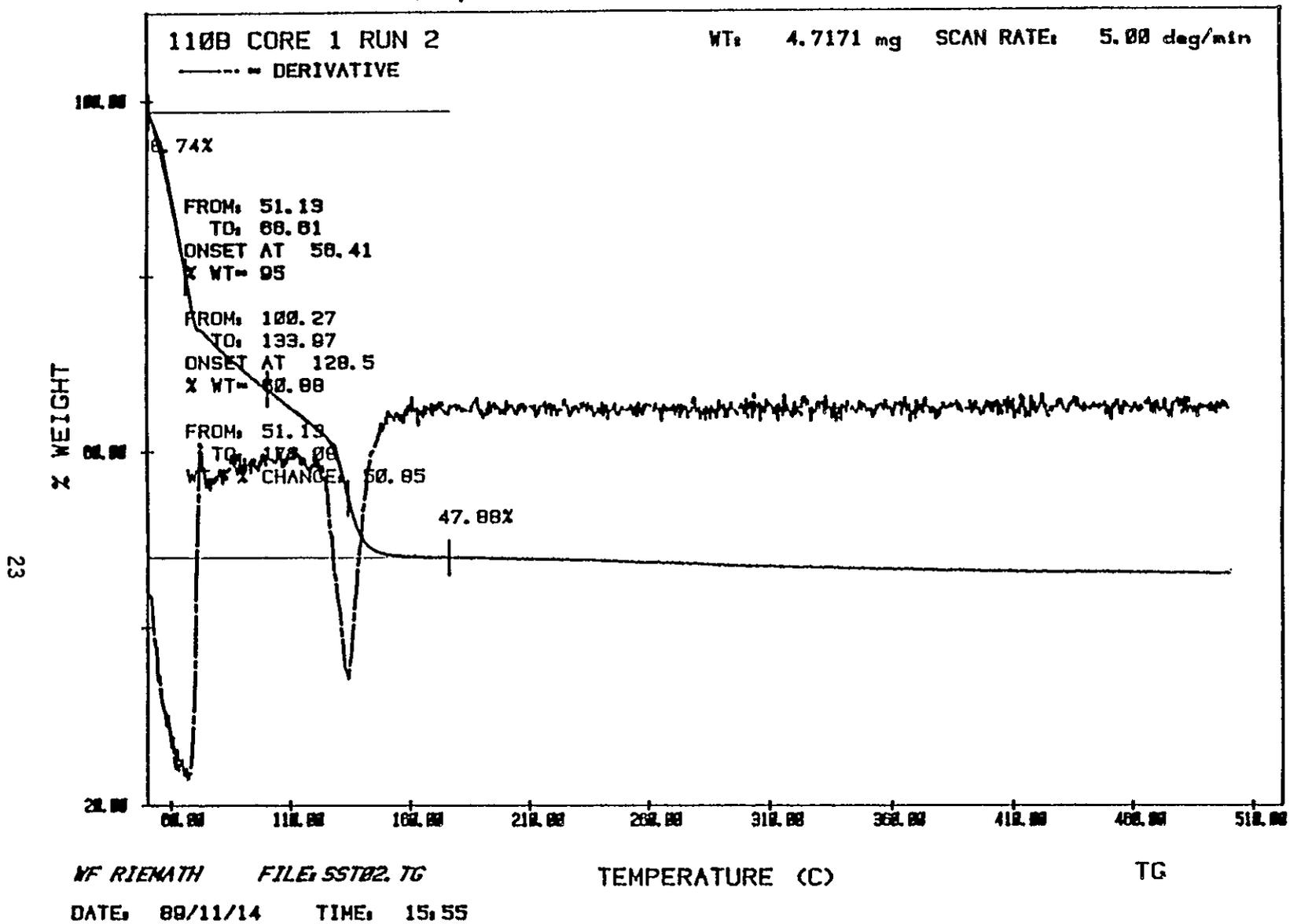


FIGURE 4. Core 1 Composite Thermogravimetric Curve, Run 1

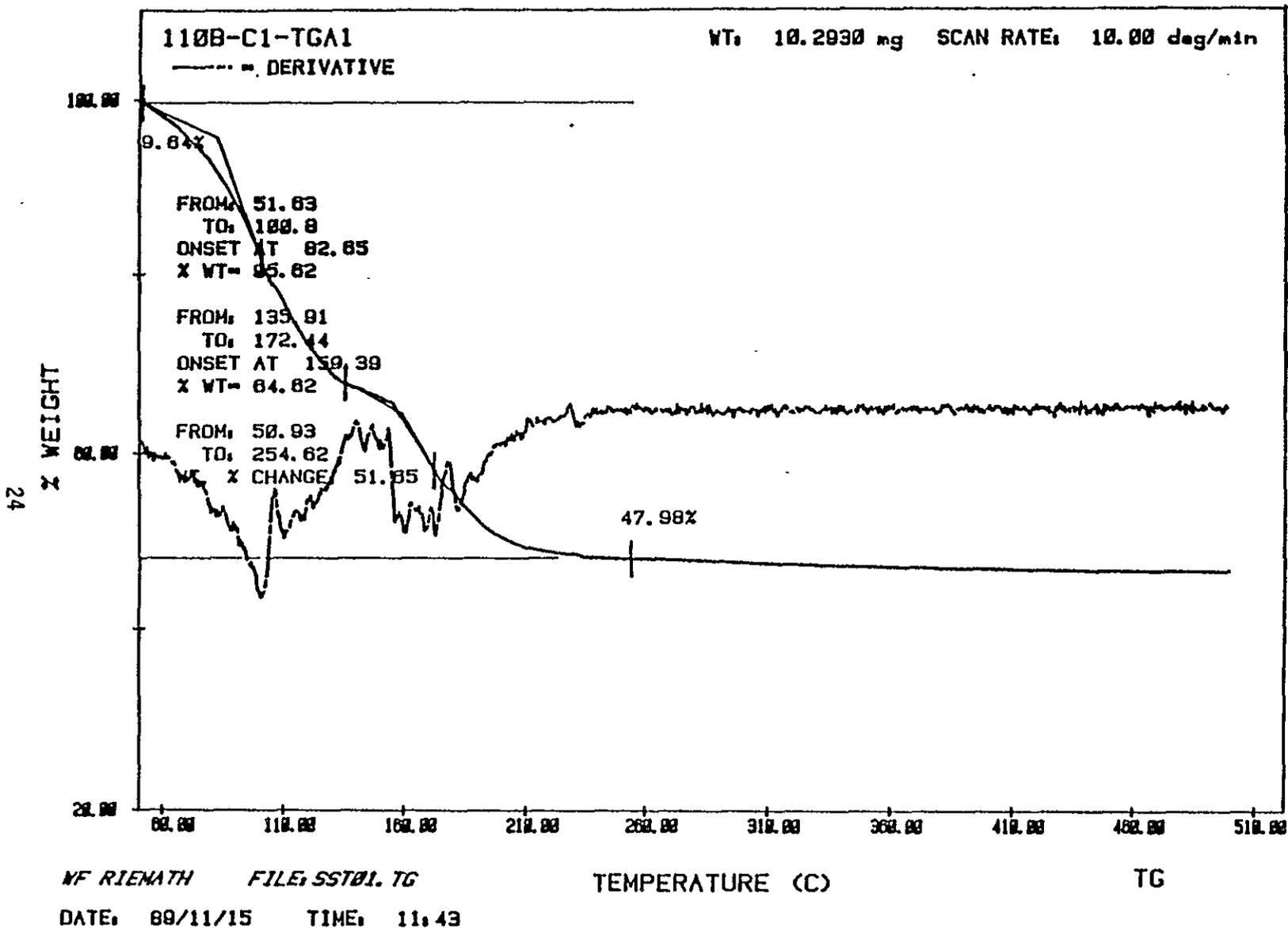


FIGURE 5. Core 1 Composite Thermogravimetric Curve, Run 2

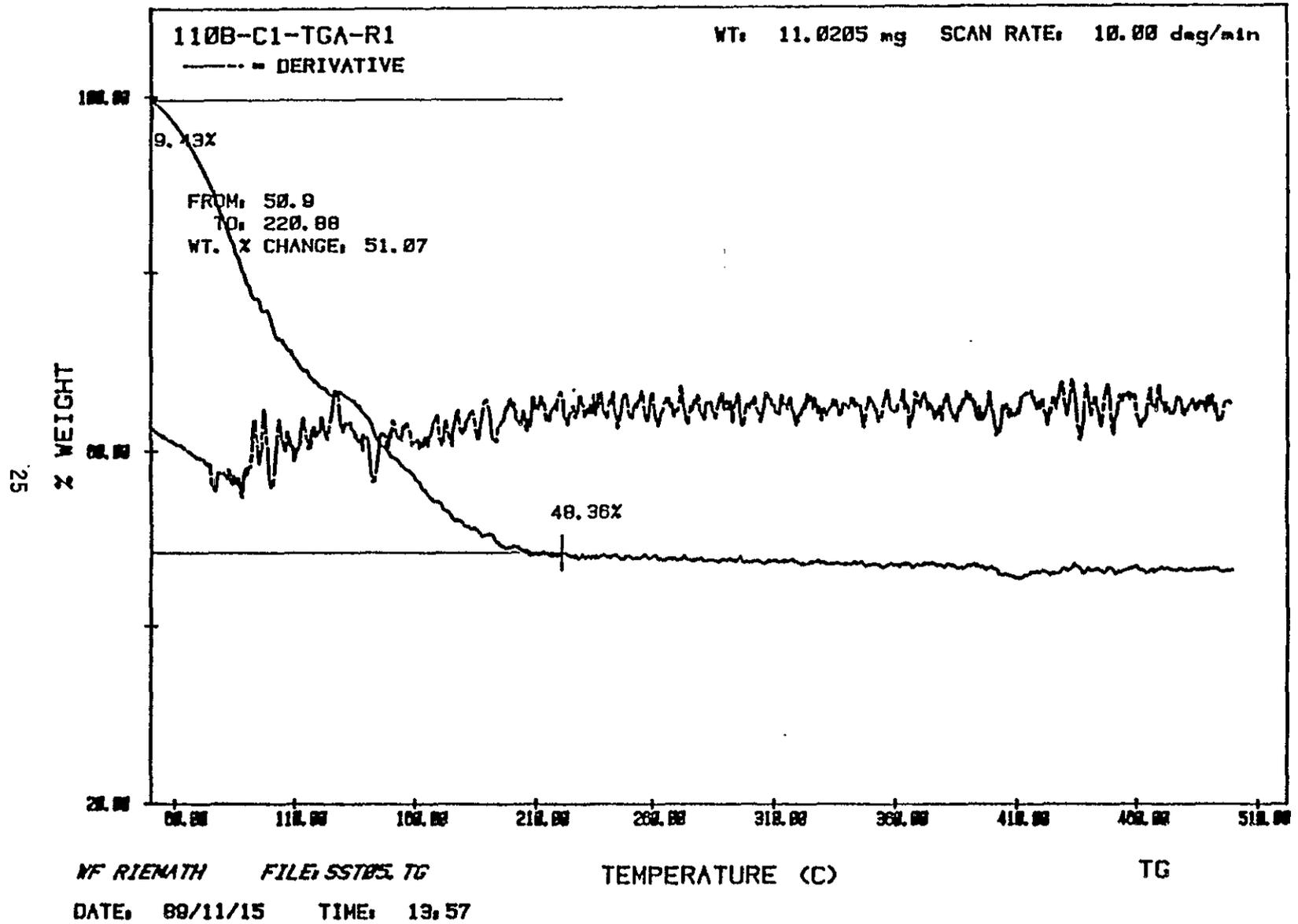


FIGURE 6. Core 1 Composite Thermogravimetric Curve, Run 3

TABLE 6: SST Core 1 Water Leach pH Values*

<u>Segment #</u>	<u>Sample #</u>	<u>pH</u>
2	89-0648-C-1	9.51
	89-0648-C-2	9.66
	89-0648-C-3	5.65
3	89-0649-C-1	8.94
	89-0649-C-2	8.99
	89-0649-C-3	5.42
4	89-0650-C-1	7.93
	89-0650-C-2	8.05
	89-0650-C-3	5.34
5	89-0651-C-1	8.21
	89-0651-C-2	7.94
	89-0651-C-3	5.79
Composite	89-0621-C-1	8.24
	89-0621-C-2	8.22
	89-0621-C-3	5.76

* Analysis completed following the method outlined in PNL-MA-599, Procedure WHC-053-1. Samples C-1 and C-2 are duplicates. Sample C-5 is a methods blank.

91120511909

TABLE 7: SST Core 1 Weight Percent Solid Values*

<u>Segment #</u>	<u>Sample #</u>	<u>Weight-% Solids</u>
2	89-0648-D-1	38.71
	89-0648-D-2	38.69
3	89-0649-D-1	40.62
	89-0649-D-2	40.78
4	89-0650-D-1	40.47
	89-0650-D-2	40.55
5	89-0651-D-1	41.36
	89-0651-D-2	41.39
Composite	89-0622-H-1	41.07
	89-0622-H-2	41.24
	89-0623-H-1	41.03
	89-0623-H-2	41.04

* Weight Percent Solids were determined following the method outlined in PNL-MA-599, Procedure PNL-ALO-504. Samples D-1/D-2 and H-1/H-2 are duplicates.

91120541910

PARTICLE SIZE
FOR
UNHOMOGENIZED SEGMENTS

9
1
1
2
5
4
1
9
1
1

THIS PAGE
INTENTIONALLY
LEFT BLANK

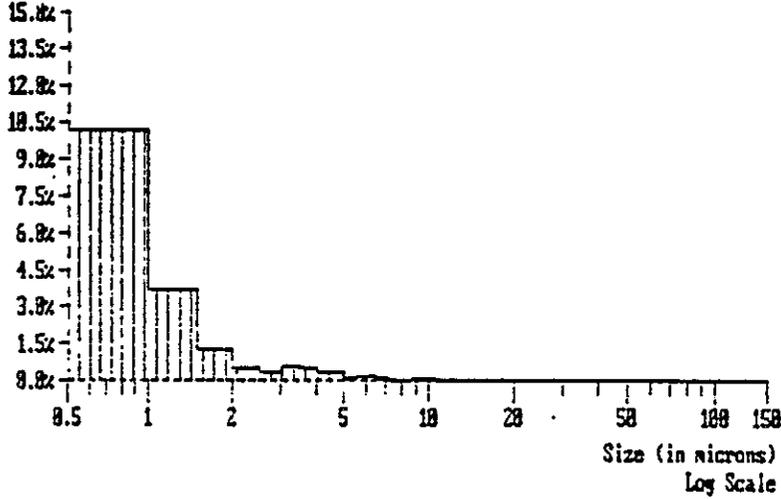
SST Core 1, Segment 2 Particle Size Analysis
 Sample # 89-0325

PROBABILITY NUMBER DENSITY GRAPH

Name: 89-0325
 1.6E+06 #/ml(100.0%)
 Mode at 0.75 μ m

Median : 0.84 μ m
 Mean(nl): 1.06 μ m
 S.D.(nl): 0.95 μ m
 Conf(nl):100.00 %

((SCALE RANGE (μ m): ADJUSTED))



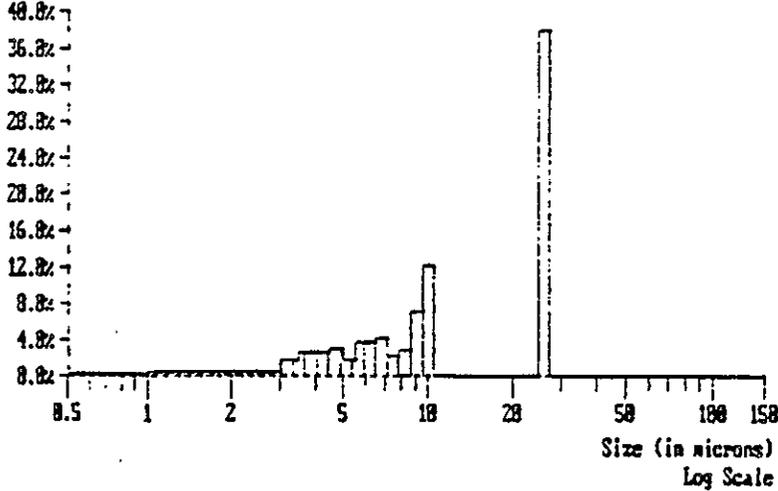
PROBABILITY VOLUME DENSITY GRAPH

Name: 89-0325
 9.4E-06 cc/ml(100.0%)
 Mode at 25.87 μ m

Mean(nv): 2.24 μ m
 S.D.(nv): 1.52 μ m

Median : 9.54 μ m
 Mean(vv): 13.43 μ m
 S.D.(vv): 9.56 μ m
 Conf(vv): 42.14 %

((SCALE RANGE (μ m): ADJUSTED))



9112051913

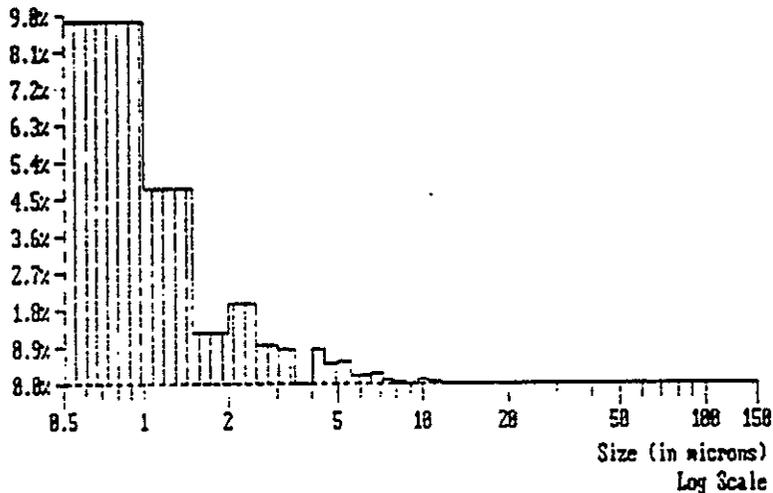
SST Core 1, Segment 3 Particle Size Analysis
89-0326

PROBABILITY NUMBER DENSITY GRAPH

Name: 89-0326
1.1E+06 #/ml(100.0%)
Mode at 0.75 μ m

Median : 0.89 μ m
Mean(n1) : 1.22 μ m
S.D.(n1) : 1.25 μ m
Conf(n1) : 99.84 %

<< SCALE RANGE (μ m): ADJUSTED >>



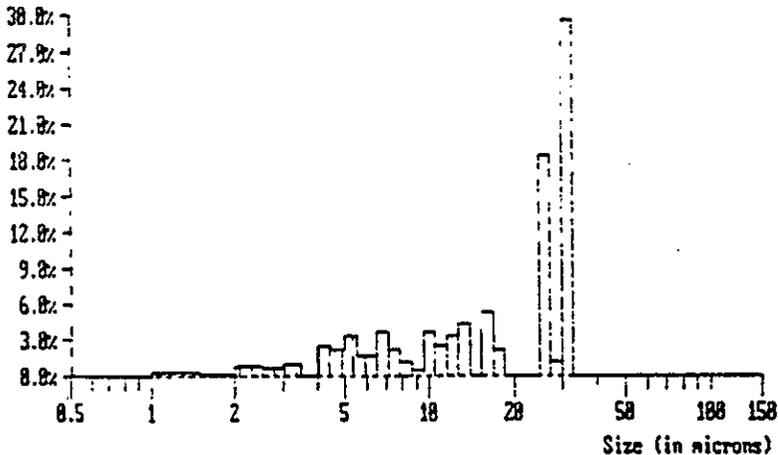
PROBABILITY VOLUME DENSITY GRAPH

Name: 89-0326
1.4E-05 cc/ml(100.0%)
Mode at 31.29 μ m

Mean(nv) : 2.94 μ m
S.D.(nv) : 2.13 μ m

Median : 16.95 μ m
Mean(vv) : 18.98 μ m
S.D.(vv) : 11.32 μ m
Conf(vv) : 49.27 %

<< SCALE RANGE (μ m): ADJUSTED >>



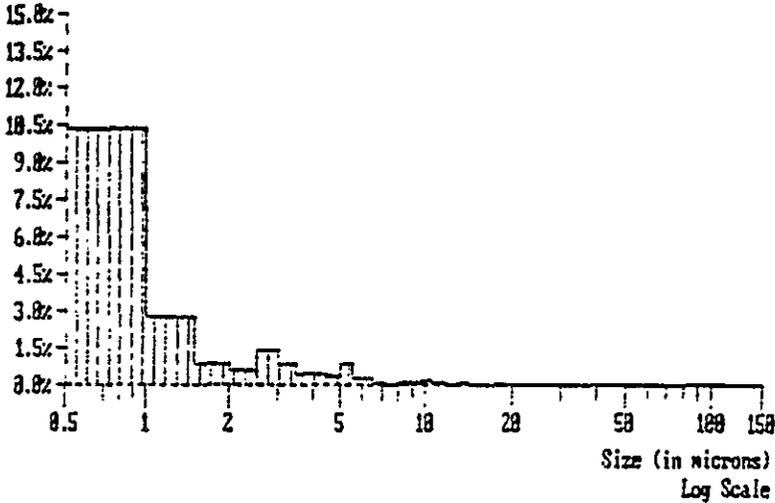
91120541313

SST Core 1, Segment 4 Particle Size Analysis
 Sample # 89-0327

PROBABILITY NUMBER DENSITY GRAPH

Name: 89-0327
 5.7E+05 #/ml(100.0%)
 Mode at 0.75 μ m
 ((SCALE RANGE (μ m): ADJUSTED))

Median : 0.83 μ m
 Mean(n1): 1.19 μ m
 S.D.(n1): 1.47 μ m
 Conf(n1): 99.38 %

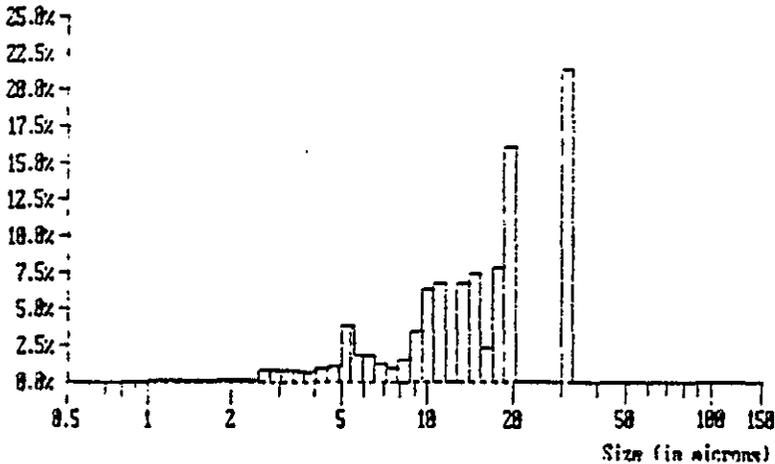


PROBABILITY VOLUME DENSITY GRAPH

Name: 89-0327
 9.7E+06 cc/ml(100.0%)
 Mode at 31.29 μ m
 ((SCALE RANGE (μ m): ADJUSTED))

Mean(nv): 3.19 μ m
 S.D.(nv): 2.48 μ m

Median : 15.80 μ m
 Mean(vv): 16.16 μ m
 S.D.(vv): 8.86 μ m
 Conf(vv): 52.89 %



91120511914

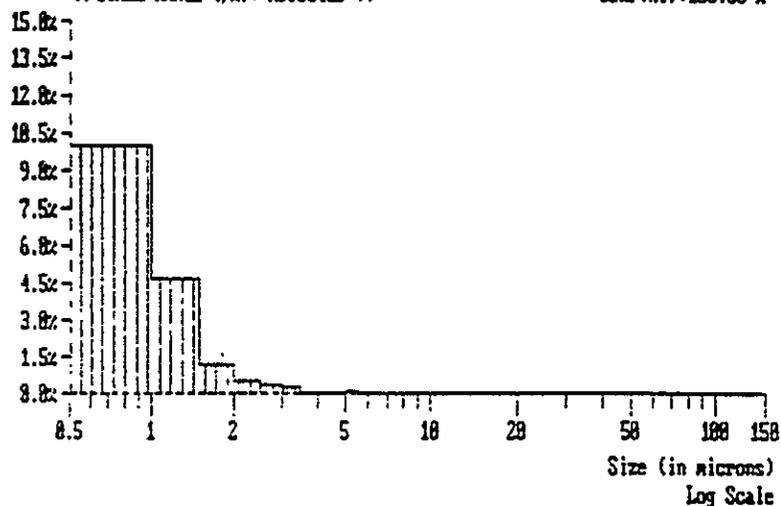
SST Core 1, Segment 5 Particle Size Analysis
89-0330

PROBABILITY NUMBER DENSITY GRAPH

Name: 89-0330
3.2E+06 #/ml(100.0%)
Mode at 8.75 μ m

Median : 8.84 μ m
Mean(nl): 8.96 μ m
S.D.(nl): 8.54 μ m
Conf(nl):100.00 %

<< SCALE RANGE (μ m): ADJUSTED >>



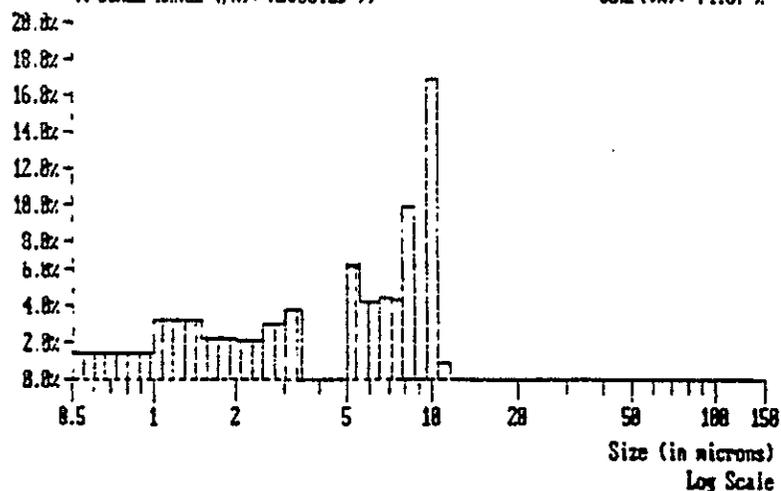
PROBABILITY VOLUME DENSITY GRAPH

Name: 89-0330
4.7E+06 cc/ml(100.0%)
Mode at 18.88 μ m

Mean(nv): 1.41 μ m
S.D.(nv): 8.78 μ m

Median : 5.87 μ m
Mean(vv): 4.93 μ m
S.D.(vv): 3.46 μ m
Conf(vv): 74.67 %

<< SCALE RANGE (μ m): ADJUSTED >>



91120541915

9 1 1 2 7 5 4 1 9 1 6

INORGANIC DATA TABLES

THIS PAGE
INTENTIONALLY
LEFT BLANK

INORGANIC FUSION ICP RESULTS

Selected segment or core composite samples were fused with potassium hydroxide (KOH), the resulting melt dissolved in an acid solution (PNL-ALO-102), and the solution was then analyzed by ICP spectroscopy (PNL-SP-7). In all cases samples were prepared in duplicate. A Methods Blank sample was included with Segment 2 samples of the Core 1. The Methods Blank involves the fusion of KOH without the addition of any sample. However, in all other respects the Methods Blank was prepared and analyzed exactly like the regular sample. With the exception of detection limits (DL) and quantitation limits (QL), all data are reported in units of weight percent (Wt %) wet sample. If concentrations fall below the estimated DL then "< DL" will be listed. If the value falls between the DL and QL the value will be reported in parentheses. Detection limits were estimated to be three times the standard deviation of the background. Quantitation limits were estimated as ten times the standard deviation of the background.

Samples for which data are included in the Core 1 Data Package are the Segments 2, 3, 4 and 5 homogenization check samples (Tables 8.1, 8.2, 8.3), Segment 2 analysis sample (Table 8.4), and two Core 1 Composite preparation/homogenization check samples (Tables 8.5 and 8.6). The segment or composite preparation/homogenization check analysis protocol includes duplicate sample prep and analyses of two samples, one from the top of the mixture and the other from the bottom.

In all of the Fusion ICP tables, column 1 contains a list of atomic symbols of 41 elements for which data are provided. In Tables 8.1, 8.2, 8.3, 8.5 and 8.6 columns 2 and 3 contain duplicate analyses of a sample. Column 4 lists the relative percent difference (RPD) between the two analyses. This format, where a set of duplicate analysis is followed by the RPD for those analyses, is repeated throughout these four tables. For each segment, the duplicate analyses of the "top" sample is listed before the duplicate analyses of the "bottom" sample. The last two columns contain the DL and QL, respectively, in units of micrograms per milliliter ($\mu\text{g/ml}$). The DL or QL in Wt % can be calculated for any element by multiplying the listed DL or QL (in $\mu\text{g/ml}$) by the appropriate Wt % Factor in row 3.

In Table 8.4, columns 2 and 3 contain duplicate analyses of homogenized Segment 2 sample. Column 4 lists data for the methods blank for this suite of analyses. Columns 5 and 6 list estimated DL and QL values in $\mu\text{g/ml}$. Column 7 lists the average values for columns 2 and 3, and the last column tabulates the RPD between the duplicates.

8
1
6
1
1
5
0
2
1
1
6

TABLE 8.1: SST Core 1 Segments 2 & 3 Fusion ICP, Homogenization Check

Samp Log#:	89-0331a189-0331b1		89-0332a189-0332b1		89-0333a189-0333b1						
Dilution:	1.00	1.00	1.00	1.00	1.00	1.00					
Wt% Factor	0.55432	0.57780	0.43540	0.49809	0.42060	0.41556					
ICP Run #	1287	1288	1289	1290	1291	1292	DL	QL			
	(Wt %)	(Wt %)	RPD	(Wt %)	(Wt %)	RPD	(Wt %)	(Wt %)	RPD	(ug/mL)	(ug/mL)
Ag	<DL	<DL		<DL	<DL		<DL	<DL		0.0310	0.1032
Al	(0.1513)	(0.2246)	39.0%	(0.1911)	(0.1693)	12.1%	<DL	(0.0664)		0.1350	0.4499
As	<DL	<DL		<DL	<DL		<DL	<DL		0.1623	0.5411
B	<DL	<DL		<DL	<DL		<DL	<DL		0.0910	0.3034
Ba	0.0089	0.0095	6.4%	0.0073	0.0085	15.6%	0.0069	0.0068	1.2%	0.0027	0.0090
Be	<DL	<DL		<DL	<DL		<DL	<DL		0.0007	0.0022
Ca	0.1314	0.1385	5.2%	0.1096	0.1031	6.1%	0.0690	0.0670	2.9%	0.0025	0.0083
Cd	<DL	<DL		<DL	<DL		<DL	<DL		0.0079	0.0263
Ce	<DL	<DL		<DL	<DL		<DL	<DL		0.1642	0.5473
Co	<DL	<DL		<DL	<DL		<DL	<DL		0.2373	0.7910
Cr	0.0737	0.1113	40.7%	0.0921	0.0800	14.0%	0.0787	0.0794	0.9%	0.0083	0.0276
Cu	<DL	<DL		<DL	<DL		<DL	(0.0066)		0.0153	0.0510
Dy	<DL	<DL		<DL	<DL		<DL	<DL		0.0084	0.0280
Fe	2.0434	2.3754	15.0%	1.8813	1.6729	11.7%	1.6420	1.6484	0.4%	0.0112	0.0373
K										0.2982	0.9941
La	<DL	(0.0128)		(0.0077)	<DL		<DL	<DL		0.0177	0.0589
Li	<DL	<DL		<DL	<DL		<DL	<DL		0.0079	0.0264
Mg	0.0246	0.0273	10.5%	0.0209	0.0199	4.7%	0.0150	0.0152	1.1%	0.0008	0.0027
Mn	0.0085	0.0116	30.3%	0.0127	0.0094	30.4%	0.0078	0.0085	9.2%	0.0019	0.0062
Mo	<DL	<DL		<DL	<DL		<DL	<DL		0.1063	0.3543
Na	6.5001	10.6543	48.4%	8.6944	7.6356	13.0%	9.0039	9.0254	0.2%	0.0732	0.2440
Nd	<DL	<DL		<DL	<DL		<DL	<DL		0.0915	0.3051
Ni										0.0312	0.1039
P	(1.1161)	1.7535	44.4%	1.4101	(1.2414)	12.7%	1.5887	1.6338	2.8%	0.8257	2.7524
Pb	(0.0953)	(0.1511)	45.3%	(0.1325)	(0.1086)	19.8%	<DL	<DL		0.1127	0.3755
Re	<DL	<DL		<DL	<DL		<DL	<DL		0.0404	0.1348
Rh	<DL	<DL		<DL	<DL		<DL	<DL		0.1361	0.4538
Ru	<DL	<DL		<DL	<DL		<DL	<DL		0.1325	0.4416
Sb	(0.1606)	<DL		(0.1107)	<DL		(0.1054)	<DL		0.2499	0.8331
Se	<DL	<DL		<DL	<DL		<DL	<DL		0.2915	0.9718
Si	0.8222	1.1281	31.4%	0.9291	0.8135	13.3%	0.8692	0.8691	0.0%	0.0679	0.2262
Sr	0.0206	0.0237	13.9%	0.0194	0.0169	13.8%	0.0188	0.0189	0.2%	0.0009	0.0031
Te	<DL	<DL		<DL	<DL		<DL	<DL		0.1113	0.3711
Th	<DL	<DL		<DL	<DL		<DL	<DL		0.1088	0.3626
Ti	<DL	<DL		<DL	<DL		<DL	<DL		0.0088	0.0294
Tl	<DL	<DL		<DL	<DL		<DL	<DL		1.3506	4.5020
U	<DL	<DL		<DL	<DL		<DL	<DL		1.1854	3.9512
V	<DL	<DL		<DL	<DL		<DL	<DL		0.0103	0.0345
Zn	0.0128	0.0170	28.3%	0.0138	0.0148	7.5%	0.0162	0.0150	8.2%	0.0028	0.0094
Zr	<DL	<DL		<DL	<DL		<DL	<DL		0.0128	0.0428
Bi	2.0621	2.3863	14.6%	1.8679	1.6437	12.8%	1.8464	1.7536	5.2%		

03/30/90

61318502116

TABLE 8.2 SST Core 1 Segment 3 & 4 Fusion ICP, Homogenization Check

Samp Log#:	89-0334a189-0334b1		89-0335a189-0335b1		89-0336a189-0336b1						
Dilution:	1.00	1.00	1.00	1.00	1.00	1.00			DL	QL	
Wt% Factor	0.45387	0.44483	0.42066	0.39631	0.48433	0.41253					
ICP Run #	1293	1299	1300	1301	1306	1307					
	(Wt %)	(Wt %)	RPD	(Wt %)	(Wt %)	RPD	(Wt %)	(Wt %)	RPD	(ug/mL)	(ug/mL)
Ag	<DL	<DL		<DL	<DL		<DL	<DL		0.0310	0.1032
Al	<DL	(0.0716)		<DL	<DL		<DL	<DL		0.1350	0.4499
As	<DL	<DL		<DL	<DL		<DL	<DL		0.1623	0.5411
B	<DL	<DL		<DL	<DL		<DL	<DL		0.0910	0.3034
Ba	0.0072	0.0078	8.4%	0.0078	0.0068	12.9%	0.0066	<DL		0.0027	0.0090
Be	<DL	<DL		<DL	<DL		<DL	<DL		0.0007	0.0022
Ca	0.0720	0.0740	2.8%	0.0465	0.0522	11.6%	0.0515	0.0668	25.9%	0.0025	0.0083
Cd	<DL	<DL		<DL	(0.0037)		<DL	<DL		0.0079	0.0263
Ce	<DL	<DL		<DL	<DL		<DL	<DL		0.1642	0.5473
Co	<DL	<DL		<DL	<DL		<DL	<DL		0.2373	0.7910
Cr	0.0768	0.0797	3.7%	0.0669	0.0664	0.7%	0.0625	0.0653	4.3%	0.0083	0.0276
Cu	<DL	(0.0077)		(0.0120)	(0.0118)	2.1%	<DL	<DL		0.0153	0.0510
Dy	<DL	<DL		<DL	<DL		<DL	<DL		0.0084	0.0280
Fe	1.5834	1.5978	0.9%	1.4246	1.4289	0.3%	1.3748	1.4311	4.0%	0.0112	0.0373
K										0.2982	0.9941
La	<DL	<DL		<DL	<DL		<DL	<DL		0.0177	0.0589
Li	<DL	<DL		<DL	<DL		<DL	<DL		0.0079	0.0264
Mg	0.0152	0.0148	2.9%	0.0110	0.0122	11.1%	0.0111	0.0149	29.8%	0.0008	0.0027
Mn	0.0086	0.0098	13.5%	0.0080	0.0077	3.1%	0.0107	0.0125	15.5%	0.0019	0.0062
Mo	<DL	<DL		<DL	<DL		<DL	<DL		0.1063	0.3543
Na	8.6975	8.8418	1.6%	9.4833	9.3732	1.2%	9.3733	9.6695	3.1%	0.0732	0.2440
Nd	<DL	<DL		<DL	<DL		<DL	<DL		0.0915	0.3051
Ni										0.0312	0.1039
P	1.6442	1.7868	8.3%	1.6889	1.7781	5.1%	1.5847	1.5392	2.9%	0.8257	2.7524
Pb	<DL	(0.0512)		<DL	<DL		<DL	<DL		0.1127	0.3755
Re	<DL	<DL		<DL	<DL		<DL	<DL		0.0404	0.1348
Rh	<DL	<DL		<DL	<DL		<DL	<DL		0.1361	0.4538
Ru	<DL	<DL		<DL	<DL		<DL	<DL		0.1325	0.4416
Sb	(0.1276)	<DL		<DL	<DL		<DL	<DL		0.2499	0.8331
Se	<DL	<DL		<DL	<DL		<DL	<DL		0.2915	0.9718
Si	0.8430	0.8603	2.0%	0.8180	0.8115	0.8%	0.7810	0.7861	0.7%	0.0679	0.2262
Sr	0.0182	0.0183	0.7%	0.0157	0.0154	1.6%	0.0151	0.0154	2.1%	0.0009	0.0031
Te	<DL	<DL		<DL	<DL		<DL	<DL		0.1113	0.3711
Th	<DL	<DL		<DL	<DL		<DL	<DL		0.1088	0.3626
Ti	<DL	<DL		<DL	<DL		<DL	<DL		0.0088	0.0294
Tl	<DL	<DL		<DL	<DL		<DL	<DL		1.3506	4.5020
U	<DL	<DL		<DL	<DL		<DL	<DL		1.1854	3.9512
V	<DL	<DL		<DL	<DL		<DL	<DL		0.0103	0.0345
Zn	0.0139	0.0143	2.8%	0.0235	0.0236	0.5%	0.0241	0.0240	0.5%	0.0028	0.0094
Zr	<DL	<DL		<DL	<DL		<DL	<DL		0.0128	0.0428
Bi	1.8109	1.7482	3.5%	1.6153	1.5892	1.6%	1.5111	1.6171	6.8%		

03/30/90

0 0 1 1 3 0 0

TABLE 8.3 SST Core 1 Segment 5 Fusion ICP Homogenization Check

Samp Log#:	89-0337a189-0337b1		89-0338a189-0338b1								
Dilution:	1.00	1.00	1.00	1.00					DL	QL	
Wt% Factor	0.42285	0.40142	0.41882	0.39745							
ICP Run #	1308	1309	1310	1311							
	(Wt %)	(Wt %)	RPD	(Wt %)	(Wt %)	RPD	(Wt %)	(Wt %)	RPD	(ug/mL)	(ug/mL)
Ag	<DL	<DL		<DL	<DL					0.0310	0.1032
Al	<DL	<DL		<DL	<DL					0.1350	0.4499
As	<DL	<DL		<DL	<DL					0.1623	0.5411
B	<DL	<DL		<DL	<DL					0.0910	0.3034
Ba	<DL	<DL		(0.0014)	<DL					0.0027	0.0090
Be	<DL	<DL		<DL	<DL					0.0007	0.0022
Ca	0.0405	0.0365	10.4%	0.0369	0.0374	1.3%				0.0025	0.0083
Cd	<DL	<DL		<DL	<DL					0.0079	0.0263
Ce	<DL	<DL		<DL	<DL					0.1642	0.5473
Co	<DL	<DL		<DL	<DL					0.2373	0.7910
Cr	0.0641	0.0637	0.6%	0.0654	0.0642	1.8%				0.0083	0.0276
Cu	<DL	<DL		<DL	<DL					0.0153	0.0510
Dy	<DL	<DL		<DL	<DL					0.0084	0.0280
Fe	1.5791	1.5346	2.9%	1.6172	1.5874	1.9%				0.0112	0.0373
K										0.2982	0.9941
La	<DL	<DL		<DL	<DL					0.0177	0.0589
Li	<DL	<DL		<DL	<DL					0.0079	0.0264
Mg	0.0064	0.0064	0.5%	0.0065	0.0070	8.1%				0.0008	0.0027
Mn	0.0095	0.0091	3.6%	0.0072	0.0087	18.8%				0.0019	0.0062
Mo	<DL	<DL		<DL	<DL					0.1063	0.3543
Na	9.5584	9.4045	1.6%	9.6978	9.5574	1.5%				0.0732	0.2440
Nd	<DL	<DL		<DL	<DL					0.0915	0.3051
Ni										0.0312	0.1039
P	1.5150	1.4099	7.2%	1.4514	1.4980	3.2%				0.8257	2.7524
Pb	<DL	<DL		<DL	<DL					0.1127	0.3755
Re	<DL	<DL		<DL	<DL					0.0404	0.1348
Rh	<DL	<DL		<DL	<DL					0.1361	0.4538
Ru	<DL	<DL		<DL	<DL					0.1325	0.4416
Sb	<DL	<DL		<DL	<DL					0.2499	0.8331
Se	<DL	<DL		<DL	<DL					0.2915	0.9718
Si	0.8702	0.8520	2.1%	0.8811	0.8678	1.5%				0.0679	0.2262
Sr	0.0197	0.0186	6.2%	0.0201	0.0196	2.4%				0.0009	0.0031
Te	<DL	<DL		<DL	<DL					0.1113	0.3711
Th	<DL	<DL		<DL	<DL					0.1088	0.3626
Ti	<DL	<DL		<DL	<DL					0.0088	0.0294
Tl	<DL	<DL		<DL	<DL					1.3506	4.5020
U	<DL	<DL		<DL	<DL					1.1854	3.9512
V	<DL	<DL		<DL	<DL					0.0103	0.0345
Zn	0.0152	0.0160	5.2%	0.0136	0.0164	18.7%				0.0028	0.0094
Zr	<DL	<DL		<DL	<DL					0.0128	0.0428
Bi	1.3320	1.4331	7.3%	1.6837	1.5222	10.1%					

03/30/90

9112051131

TABLE 8.4 SST Core 1, Segment 2 Fusion ICP Data

Samp Log#:	89-0648a1	89-0648a2	89-0648a3			89-0648a1						
Dilution:	1.00	1.00	1.00					89-0648a2				
Wt% Factor	0.46478	0.43078	0.44713			DL	QL	Average				
ICP Run #	1713	1714	1715									
	(Wt %)	(Wt %)	%Dif	(Wt %)	(Wt %)	%Dif	(Wt% *)	(Wt% *)	(ug/mL)	(ug/mL)	(Wt %)	RPD
Ag	<DL	<DL		<DL	<DL		<DL	<DL	0.0303	0.1010	N/A	
Al	(0.1734)	(0.1677)		<DL	<DL		<DL	<DL	0.2048	0.6827	0.1706	3.3%
As	<DL	<DL		<DL	<DL		<DL	<DL	0.1619	0.5396	N/A	
B	<DL	<DL		<DL	<DL		<DL	<DL	0.0927	0.3089	N/A	
Ba	(0.0023)	(0.0026)		<DL	<DL		<DL	<DL	0.0024	0.0079	0.0024	12.5%
Be	<DL	<DL		<DL	<DL		<DL	<DL	0.0008	0.0027	N/A	
Ca	0.1199	0.1141		<DL	<DL		0.0262	<DL	0.0027	0.0090	0.1170	4.9%
Cd	(0.0053)	(0.0037)		<DL	<DL		<DL	<DL	0.0079	0.0262	0.0045	35.2%
Ce	<DL	<DL		<DL	<DL		<DL	<DL	0.1800	0.6000	N/A	
Co	<DL	<DL		<DL	<DL		<DL	<DL	0.2543	0.8476	N/A	
Cr	0.0941	0.0900		<DL	<DL		<DL	<DL	0.0074	0.0246	0.0921	4.4%
Cu	<DL	<DL		<DL	<DL		<DL	<DL	0.0146	0.0486	N/A	
Dy	<DL	<DL		<DL	<DL		<DL	<DL	0.0092	0.0306	N/A	
Fe	1.9677	1.9228		<DL	<DL		(0.0056)	<DL	0.0104	0.0346	1.9453	2.3%
K	<DL	<DL		<DL	<DL		<DL	<DL	0.3111	1.0372	N/A	
La	<DL	<DL		<DL	<DL		<DL	<DL	0.0193	0.0642	N/A	
Li	<DL	<DL		<DL	<DL		<DL	<DL	0.0118	0.0393	N/A	
Mg	0.0235	0.0220		<DL	<DL		(0.0016)	<DL	0.0014	0.0045	0.0228	6.4%
Mn	0.0112	0.0099		<DL	<DL		<DL	<DL	0.0023	0.0078	0.0105	12.7%
Mo	<DL	<DL		<DL	<DL		<DL	<DL	0.0888	0.2959	N/A	
Na	8.9564	8.6648		<DL	<DL		(0.0666)	<DL	0.0626	0.2088	8.8106	3.3%
Nd	<DL	<DL		<DL	<DL		<DL	<DL	0.1114	0.3713	N/A	
Ni	<DL	<DL		<DL	<DL		<DL	<DL	0.0294	0.0978	N/A	
P	1.4283	1.4593		<DL	<DL		<DL	<DL	0.7219	2.4064	1.4438	2.1%
Pb	(0.1200)	(0.1282)		<DL	<DL		<DL	<DL	0.1186	0.3955	0.1241	6.6%
Re	<DL	<DL		<DL	<DL		<DL	<DL	0.0363	0.1209	N/A	
Rh	<DL	<DL		<DL	<DL		<DL	<DL	0.1267	0.4224	N/A	
Ru	<DL	<DL		<DL	<DL		<DL	<DL	0.0621	0.2070	N/A	
Sb	(0.2228)	<DL		<DL	<DL		(0.2101)	<DL	0.3363	1.1210	N/A	
Se	<DL	<DL		<DL	<DL		<DL	<DL	0.2765	0.9216	N/A	
Si	0.9245	0.9012		<DL	<DL		<DL	<DL	0.0554	0.1847	0.9129	2.6%
Sr	0.0200	0.0194		<DL	<DL		<DL	<DL	0.0020	0.0066	0.0197	3.5%
Te	<DL	<DL		<DL	<DL		<DL	<DL	0.1006	0.3354	N/A	
Th	<DL	<DL		<DL	<DL		<DL	<DL	0.1193	0.3976	N/A	
Ti	<DL	<DL		<DL	<DL		<DL	<DL	0.0096	0.0322	N/A	
Tl	<DL	<DL		<DL	<DL		<DL	<DL	0.9976	3.3254	N/A	
U	<DL	<DL		<DL	<DL		<DL	<DL	1.2691	4.2302	N/A	
V	<DL	<DL		<DL	<DL		<DL	<DL	0.0091	0.0303	N/A	
Zn	0.0165	0.0119		<DL	<DL		0.0054	<DL	0.0026	0.0087	0.0142	32.5%
Zr	<DL	<DL		<DL	<DL		<DL	<DL	0.0113	0.0378	N/A	
Bi	1.9242	1.9730		<DL	<DL		0.0268	<DL	<DL	<DL	1.9486	2.5%

* Methods blank - average sample weight used to calculate wt%.

03/30/90

911275192

TABLE 8.5 SST Core 1, Composite Fusion ICP Data

Samp Log#: 89-0622a189-0622b1		90-0623a189-0623b1								DL	QL
Dilution: 1.00 1.00		1.00 1.00									
Wt% Factor 0.47389 0.58427		0.49264 0.41852									
ICP Run # 1653 1654		1655 1656									
	(Wt %)	(Wt %)	RPD	(Wt %)	(Wt %)	RPD	(Wt %)	(Wt %)	RPD	(ug/mL)	(ug/mL)
Ag	<DL	<DL		<DL	<DL					0.0292	0.0972
Al	0.4132	0.3806	8.2%	0.5860	0.3134	60.6%				0.1926	0.6419
As	<DL	<DL		<DL	<DL					0.1673	0.5576
B	0.9551	1.0564	10.1%	1.7755	0.8148	76.2%				0.0944	0.3146
Ba	0.1378	0.1239	10.7%	0.2071	0.0921	76.9%				0.0022	0.0075
Be	<DL	<DL		<DL	<DL					0.0006	0.0022
Ca	0.2780	0.2715	2.4%	0.3852	0.2062	60.5%				0.0026	0.0086
Cd	<DL	<DL		<DL	<DL					0.0076	0.0252
Ce	<DL	<DL		<DL	<DL					0.1595	0.5315
Co	<DL	<DL		<DL	<DL					0.2267	0.7557
Cr	0.0738	0.0738	0.0%	0.0757	0.0730	3.6%				0.0069	0.0231
Cu	<DL	<DL		<DL	<DL					0.0136	0.0453
Dy	<DL	<DL		<DL	<DL					0.0088	0.0292
Fe	1.7649	1.7370	1.6%	1.8111	1.7455	3.7%				0.0097	0.0324
K										0.3652	1.2172
La	<DL	<DL		<DL	<DL					0.0151	0.0504
Li	<DL	<DL		<DL	<DL					0.0106	0.0353
Mg	0.0193	0.0196	1.8%	0.0229	0.0173	28.0%				0.0013	0.0045
Mn	0.0076	0.0071	6.3%	0.0084	0.0081	3.7%				0.0023	0.0077
Mo	<DL	<DL		<DL	<DL					0.0912	0.3039
Na	10.5761	10.5357	0.4%	11.7806	10.0219	16.1%				0.0719	0.2397
Nd	<DL	<DL		<DL	<DL					0.0855	0.2849
Ni										0.0257	0.0858
P	1.4822	1.5122	2.0%	1.5951	1.5105	5.4%				0.7406	2.4687
Pb	(0.0651)	(0.0657)	0.9%	(0.1078)	(0.0856)	23.0%				0.1112	0.3706
Re	<DL	<DL		<DL	<DL					0.0340	0.1133
Rh	<DL	<DL		<DL	<DL					0.1174	0.3914
Ru	<DL	<DL		<DL	<DL					0.1099	0.3664
Sb	(0.1385)	<DL		<DL	<DL					0.2494	0.8312
Se	<DL	<DL		<DL	<DL					0.2659	0.8865
Si	1.3614	1.7936	27.4%	2.3916	1.5868	40.5%				0.0573	0.1909
Sr	0.0215	0.0211	2.0%	0.0231	0.0202	13.1%				0.0020	0.0065
Te	<DL	<DL		<DL	<DL					0.0972	0.3240
Th	<DL	<DL		<DL	<DL					0.1070	0.3568
Ti	<DL	<DL		(0.0056)	(0.0036)	43.8%				0.0076	0.0252
Tl	<DL	<DL		<DL	<DL					1.1836	3.9455
U	<DL	<DL		<DL	<DL					1.1456	3.8186
V	<DL	<DL		<DL	<DL					0.0094	0.0312
Zn	0.0169	0.0250	38.9%	0.0210	0.0142	38.7%				0.0024	0.0082
Zr	<DL	<DL		<DL	<DL					0.0105	0.0348
Bi	1.7818	1.8229	2.3%	2.0789	1.9294	7.5%				N/A	N/A

03/30/90

9112951193

TABLE 8.6: SST Core 1, Composite Fusion ICP Data

Samp Log#:	90-1125a1	90-1125a2	90-1125a3			90-1125a1		
Dilution:	1.00	1.00	1.00			90-1125a2		
Wt% Factor	0.48722	0.47555	0.48131			Average		
ICP Run #	864	865	863					
	(Wt %)	(Wt %)	%Dif	(Wt %)	(Wt %)	%Dif	(Wt% *)	(Wt% *)
	(ug/mL)	(ug/mL)		(ug/mL)	(ug/mL)			
								RPD
Ag	<DL	<DL		<DL	<DL			
Al	0.1188	(0.1148)		<DL	0.0728	0.2426	0.1168	3.4%
As	<DL	<DL		<DL	0.0534	0.1782	N/A	
B	<DL	<DL		<DL	0.0628	0.2093	N/A	
Ba	(0.0028)	(0.0024)		<DL	0.0027	0.0089	0.0026	17.2%
Be	<DL	<DL		<DL	0.0002	0.0007	N/A	
Ca	0.1144	0.0746		0.0144	0.0004	0.0013	0.0945	42.0%
Cd	<DL	<DL		<DL	0.0051	0.0170	N/A	
Ce	<DL	<DL		<DL	0.1487	0.4958	N/A	
Co	<DL	<DL		<DL	0.2590	0.8634	N/A	
Cr	0.0818	0.0798		<DL	0.0040	0.0132	0.0808	2.5%
Cu	(0.0082)	(0.0087)		<DL	0.0096	0.0319	0.0084	5.6%
Dy	<DL	<DL		<DL	0.0096	0.0318	N/A	
Fe	2.8417	1.7961		(0.0057)	0.0062	0.0205	2.3189	45.1%
K					0.3617	1.2058		
La	(0.0086)	(0.0080)		<DL	0.0126	0.0420	0.0083	7.6%
Li	<DL	<DL		<DL	0.0077	0.0255	N/A	
Mg	0.0174	0.0151		0.0011	0.0004	0.0013	0.0163	14.3%
Mn	0.0159	0.0091		(0.0008)	0.0012	0.0041	0.0125	54.1%
Mo	<DL	<DL		<DL	0.0104	0.0347	N/A	
Na	10.0982	9.6515		(0.0888)	0.0555	0.1851	9.8748	4.5%
Nd	<DL	<DL		<DL	0.0644	0.2146	N/A	
Ni					0.0145	0.0483		
P	1.7251	1.6969		<DL	0.1935	0.6449	1.7110	1.6%
Pb	0.1102	0.1127		<DL	0.0508	0.1694	0.1115	2.2%
Re	<DL	<DL		<DL	0.0120	0.0400	N/A	
Rh	<DL	<DL		<DL	0.0874	0.2914	N/A	
Ru	<DL	<DL		<DL	0.0277	0.0922	N/A	
Sb	<DL	<DL		<DL	0.3957	1.3191	N/A	
Se	<DL	<DL		<DL	0.1852	0.6173	N/A	
Si	0.9483	0.9366		<DL	0.0385	0.1284	0.9424	1.2%
Sr	0.0207	0.0203		<DL	0.0011	0.0036	0.0205	2.0%
Te	<DL	<DL		<DL	0.0449	0.1497	N/A	
Th	<DL	<DL		<DL	0.1029	0.3431	N/A	
Ti	<DL	<DL		<DL	0.0068	0.0227	N/A	
Tl	<DL	<DL		<DL	1.2592	4.1975	N/A	
U	<DL	<DL		<DL	1.1133	3.7109	N/A	
V	<DL	<DL		<DL	0.0070	0.0235	N/A	
Zn	0.2611	0.0124		(0.0015)	0.0023	0.0076	0.1367	181.9%
Zr	0.0798	<DL		<DL	0.0071	0.0237	N/A	
Bi	1.8368	1.7405		<DL			1.7887	5.4%

* Methods blank - average sample weight used to calculate wt%.

04/20/90

91120511304

TABLE 8.6: SST Core 1, Composite Fusion ICP Data (Cont'd)

Samp Log#:	90-1126a1	90-1126a2	90-1126a3	90-1126a1 90-1126a2								
Dilution:	1.00	1.00	1.00	DL	QL	Average						
Wt% Factor	0.49668	0.48465	0.49059									
ICP Run #	870	871	869									
	(Wt %)	(Wt %)	%Dif	(Wt %)	(Wt %)	%Dif	(Wt% *)	(Wt% *)	(ug/mL)	(ug/mL)	(Wt %)	RPD
Ag	<DL	<DL		<DL	<DL				0.0104	0.0347	N/A	
Al	(0.1155)	(0.1107)		<DL	<DL				0.0728	0.2426	0.1131	4.2%
As	<DL	<DL		<DL	<DL				0.0534	0.1782	N/A	
B	<DL	<DL		<DL	<DL				0.0628	0.2093	N/A	
Ba	(0.0013)	(0.0013)		<DL	<DL				0.0027	0.0089	0.0013	2.5%
Be	<DL	<DL		<DL	<DL				0.0002	0.0007	N/A	
Ca	0.0788	0.0798		<DL	<DL				0.0004	0.0013	0.0793	1.3%
Cd	<DL	<DL		<DL	<DL				0.0051	0.0170	N/A	
Ce	<DL	<DL		<DL	<DL				0.1487	0.4958	N/A	
Co	<DL	<DL		<DL	<DL				0.2590	0.8634	N/A	
Cr	0.0815	0.0780		<DL	<DL				0.0040	0.0132	0.0797	4.5%
Cu	(0.0054)	(0.0058)		<DL	<DL				0.0096	0.0319	0.0056	6.3%
Dy	<DL	<DL		<DL	<DL				0.0096	0.0318	N/A	
Fe	1.8692	1.7969		<DL	<DL		(0.0036)		0.0062	0.0205	1.8331	3.9%
K									0.3617	1.2058		
La	<DL	<DL		<DL	<DL				0.0126	0.0420	N/A	
Li	<DL	<DL		<DL	<DL				0.0077	0.0255	N/A	
Mg	0.0143	0.0150		<DL	<DL		0.0009		0.0004	0.0013	0.0146	4.6%
Mn	0.0084	0.0073		<DL	<DL				0.0012	0.0041	0.0079	13.1%
Mo	<DL	<DL		<DL	<DL				0.0104	0.0347	N/A	
Na	9.8419	9.5870		<DL	<DL		(0.0564)		0.0555	0.1851	9.7145	2.6%
Nd	<DL	<DL		<DL	<DL				0.0644	0.2146	N/A	
Ni									0.0145	0.0483		
P	1.6776	1.6689		<DL	<DL				0.1935	0.6449	1.6732	0.5%
Pb	0.0977	0.0920		<DL	<DL				0.0508	0.1694	0.0949	6.0%
Re	<DL	<DL		<DL	<DL				0.0120	0.0400	N/A	
Rh	<DL	<DL		<DL	<DL				0.0874	0.2914	N/A	
Ru	<DL	<DL		<DL	<DL				0.0277	0.0922	N/A	
Sb	<DL	<DL		<DL	<DL				0.3957	1.3191	N/A	
Se	<DL	<DL		<DL	<DL				0.1852	0.6173	N/A	
Si	0.9496	0.9329		<DL	<DL				0.0385	0.1284	0.9413	1.8%
Sr	0.0202	0.0202		<DL	<DL				0.0011	0.0036	0.0202	0.3%
Te	<DL	<DL		<DL	<DL				0.0449	0.1497	N/A	
Th	<DL	<DL		<DL	<DL				0.1029	0.3431	N/A	
Ti	<DL	<DL		<DL	<DL				0.0068	0.0227	N/A	
Tl	<DL	<DL		<DL	<DL				1.2592	4.1975	N/A	
U	<DL	<DL		<DL	<DL				1.1133	3.7109	N/A	
V	<DL	<DL		<DL	<DL				0.0070	0.0235	N/A	
Zn	0.0123	0.0105		<DL	<DL		(0.0012)		0.0023	0.0076	0.0114	15.4%
Zr	0.0829	<DL		<DL	<DL				0.0071	0.0237	N/A	
Bi	1.7384	1.7302		<DL	<DL						1.7343	0.5%

* Methods blank - average sample weight used to calculate wt%.

04/20/90

91120511005

INORGANIC ACID LEACH ICP RESULTS

Selected segment or core composite samples were leached with nitric acid following the EPA SW-846 protocols (PNL-ALO-101). For a single segment or core composite sample five aliquots were prepared for analysis. These include duplicate sample preps, a spiked sample prep, a spiked control sample, and a methods blank. The Spike Control is prepared by adding the spike solution to an appropriate amount of deionized water. Each of these aliquots were analyzed by ICP spectroscopy at a 1X or 5X dilution (PNL-SP-7). ICP analysis data are reported for each dilution, as well as a percent difference (% Dif) between the analyses. With the exception of detection limits (DL) and quantitation limits (QL), all data are reported in units of weight percent (Wt %) wet sample. If concentrations fall below the estimated DL then "< DL" will be listed. If the value falls between the DL and QL the value will be reported in parentheses. Detection limits were estimated to be three times the standard deviation of the background. Quantitation limits were estimated as ten times the standard deviation of the background.

Acid Leach ICP samples in the Core 1 Data Package include Segments 2 through 5 (Tables 8.7, 8.8, 8.9) and the Core Composite (Table 8.10). Column 1 of each of these tables list the atomic symbols of each of the elements for which data are reported. In Tables 8.7, 8.8, and 8.10 columns 2 and 3 contain duplicate analyses of a 5X dilution of the sample. Column 4 contains the data for the methods blank. In Table 8.9, columns 2 and 3 contain duplicate analyses of a 5X dilution of the sample, and column 4 lists the relative percent difference (RPD) between the two analyses. This format, where a set of duplicate analysis is followed by the RPD for those analyses is repeated in columns 5, 6, and 7 in Table 8.9. In each of the tables the last two columns contain the DL and QL, respectively, in units of micrograms per milliliter ($\mu\text{g/ml}$). The DL or QL in Wt % can be calculated for any element by multiplying the listed DL or QL (in $\mu\text{g/ml}$) by the appropriate Wt % Factor in row 3.

The second page of Tables 8.7, 8.8 and 8.10 contains primarily quality control (QC) data. Column 2 lists the average for the duplicate analyses. Column 3 provides the relative percent difference (RPD) between the analyses. Column 4 tabulates the calculated Wt % Spike added to spiked sample. Columns

5 lists the data for 5X dilution on the spiked sample, and Column 6 lists the percent recovery (% Rec). Columns 7 and 8 report values for the 1X and 5X dilutions of the Spike Control sample and Column 9 reports the % Dif. Column 10 lists the calculated concentrations of elements in the Spike Control and Column 11 reports % Rec in the Spike Control sample analysis.

91120311307

TABLE 8.7 SST Core 1, Segment 2 Acid Leach ICP

Samp Log#:	89-0648b1	89-0648b2	89-0648b5						
Dilution:	5.00	5.00	1.00						
Wt% Factor	0.09069	0.06990	0.01579						
ICP Run #	1767R	1768	1787					DL	QL
	(Wt %)	(Wt %) %dif	(Wt %)	(Wt %) %dif	(Wt% *)	(Wt% *) %dif		(ug/mL)	(ug/mL)
Ag	0.0063		0.0063		<DL			0.0093	0.0311
Al	0.2077		0.2046		<DL			0.2007	0.6689
As	<DL		<DL		<DL			0.1376	0.4587
B	<DL		<DL		(0.0049)			0.0998	0.3328
Ba	0.0019		0.0018		<DL			0.0041	0.0136
Be	<DL		<DL		<DL			0.0010	0.0032
Ca	0.1214		0.1205		0.0133			0.0027	0.0089
Cd	(0.0007)		(0.0009)		0.0020			0.0067	0.0222
Ce	(0.0261)		(0.0183)		<DL			0.2102	0.7006
Co	<DL		<DL		<DL			0.2878	0.9593
Cr	0.0966		0.0968		0.0008			0.0080	0.0265
Cu	(0.0029)		(0.0025)		0.0015			0.0160	0.0534
Dy	<DL		<DL		<DL			0.0135	0.0450
Fe	2.0426		2.0433		0.0046			0.0096	0.0319
K	(0.0706)		(0.0593)		0.0328			0.3343	1.1143
La	0.0130		0.0127		<DL			0.0199	0.0664
Li	(0.0042)		0.0037		0.0020			0.0154	0.0512
Mg	0.0256		0.0247		0.0028			0.0010	0.0034
Mn	0.0092		0.0095		0.0002			0.0026	0.0087
Mo	(0.0017)		(0.0011)		0.0005			0.0089	0.0296
Na	9.1388		9.0783		0.0936			0.1264	0.4213
Nd	(0.0146)		(0.0108)		<DL			0.1382	0.4605
Ni	(0.0031)		(0.0026)		<DL			0.0247	0.0823
P	1.6200		1.6149		0.0486			0.2885	0.9617
Pb	0.1636		0.1635		(0.0019)			0.0592	0.1973
Re	<DL		<DL		<DL			0.0250	0.0832
Rh	<DL		<DL		<DL			0.1425	0.4751
Ru	<DL		<DL		<DL			0.0450	0.1499
Sb	<DL		<DL		<DL			0.4673	1.5577
Se	(0.0124)		(0.0090)		<DL			0.1251	0.4170
Si	0.0444		0.0426		0.0071			0.0664	0.2212
Sr	0.0203		0.0203		(0.0000)			0.0022	0.0072
Te	<DL		<DL		<DL			0.0763	0.2544
Th	<DL		<DL		<DL			0.1747	0.5823
Ti	(0.0018)		(0.0015)		<DL			0.0120	0.0399
Tl	<DL		<DL		<DL			1.5447	5.1491
U	(0.1808)		(0.1210)		<DL			1.6265	5.4217
V	<DL		<DL		<DL			0.0098	0.0328
Zn	0.0071		0.0068		0.0031			0.0042	0.0142
Zr	(0.0018)		(0.0015)		<DL			0.0119	0.0396
Bi	1.9705		2.0727		<DL				

* Methods blank - average sample weight used to calculate wt%.

03/29/90

91127511928

TABLE 8.7 SST Core 1, Segment 2 Acid Leach ICP (Cont.)

	89-0648b1		89-0648b3		<Sample ID>		89-0648b4		89-0648b4		Spike	
	89-0648b2		5.00		<Dilution>		1.00		5.00		STD	
	(Wt %)	RPD	Spike Added (Wt %)	Spike+ Sample (Wt %)	Spike+ Sample (Wt %)	% Dif	% Rec	Spike Control (ug/mL)	Spike Control (ug/mL)	% Dif	Spike STD (ug/mL)	% rec
Ag	0.0063	0.3%		0.0064								
Al	0.2062	1.5%		0.2059								
As	N/A		0.0345	(0.0312)		90.7%		74.7	65.3	12.7%	80.0	93.4%
B	N/A			<DL								
Ba	0.0018	5.8%	0.0345	0.0350		96.4%		75.1	75.2	0.1%	80.0	93.8%
Be	N/A		0.0009	0.0008		96.3%		1.9	1.8	1.9%	2.0	93.5%
Ca	0.1209	0.7%		0.1207								
Cd	0.0008	31.5%	0.0009	0.0020		142.3%		5.1	3.7	26.0%	2.0	252.9%
Ce	0.0222	34.9%		(0.0223)								
Co	N/A		0.0086	<DL		163.5%		18.8	32.7	73.9%	20.0	93.9%
Cr	0.0967	0.2%	0.0034	0.1002		99.9%		9.3	8.5	8.5%	8.0	116.0%
Cu	0.0027	14.4%	0.0043	0.0072		103.7%		11.9	11.1	6.6%	10.0	119.0%
Dy	N/A			<DL								
Fe	2.0430	0.0%		2.0388								
K	0.0650	17.4%		(0.0689)								
La	0.0129	2.2%		0.0128								
Li	0.0040	13.4%		(0.0040)								
Mg	0.0252	3.5%		0.0252								
Mn	0.0094	2.5%	0.0086	0.0178		97.4%		19.3	19.6	1.4%	20.0	96.6%
Mo	0.0014	40.1%		(0.0016)								
Na	9.1086	0.7%		9.1444								
Nd	0.0127	29.7%		(0.0127)								
Ni	0.0028	18.6%	0.0086	0.0111		96.1%		19.7	20.7	4.7%	20.0	98.7%
P	1.6175	0.3%		1.6149								
Pb	0.1636	0.1%	0.0086	0.1672		42.3%		22.1	22.5	1.9%	20.0	110.3%
Re	N/A			<DL								
Rh	N/A			<DL								
Ru	N/A			<DL								
Sb	N/A		0.0086	<DL		-15.5%		18.3			20.0	91.3%
Se	0.0107	31.6%	0.0345	(0.0328)		64.0%		68.1	73.0	7.2%	80.0	85.1%
Si	0.0435	4.1%		0.0769								
Sr	0.0203	0.1%		0.0203								
Te	N/A			<DL								
Th	N/A			<DL								
Ti	0.0017	18.3%		(0.0016)								
Tl	N/A		0.0345	<DL		342.3%		64.3	220.4	242.9%	80.0	80.3%
U	0.1509	39.6%		(0.1518)								
V	N/A		0.0086	0.0087		101.1%		18.7	18.8	0.5%	20.0	93.6%
Zn	0.0070	4.2%	0.0086	0.0157		100.9%		22.1	22.5	1.9%	20.0	110.4%
Zr	0.0017	16.5%		(0.0016)								
Bi	2.0216	5.1%		2.0789								

03/29/90

911211319

TABLE 8.8 SST Core 1, Segment 3 Acid Leach ICP

Samp Log#:	89-648b1	89-649b2	89-649b5	89-649b5				
Dilution:	5.00	5.00	1.00					
Wt% Factor	0.11038	0.12543	0.02348					
ICP Run #	1809	1810	1817					
	(Wt %)	(Wt %)	%Dif	(Wt %)	(Wt %)	%Dif	DL	QL
	(ug/mL)	(ug/mL)		(ug/mL)	(ug/mL)		(ug/mL)	(ug/mL)
Ag	<DL	<DL		<DL	<DL		0.0126	0.0419
Al	0.0643	0.0697		<DL	<DL		0.0886	0.2953
As	<DL	<DL		<DL	<DL		0.1567	0.5223
B	<DL	<DL		<DL	<DL		0.0718	0.2395
Ba	0.0016	0.0016		<DL	<DL		0.0033	0.0112
Be	<DL	<DL		<DL	<DL		0.0004	0.0012
Ca	0.0724	0.0771		0.0155			0.0017	0.0058
Cd	<DL	<DL		<DL	<DL		0.0060	0.0201
Ce	<DL	<DL		<DL	<DL		0.2083	0.6942
Co	<DL	<DL		<DL	<DL		0.3038	1.0128
Cr	0.0850	0.0885		(0.0003)			0.0065	0.0218
Cu	(0.0023)	<DL		<DL	<DL		0.0177	0.0589
Dy	<DL	<DL		<DL	<DL		0.0163	0.0545
Fe	1.8147	1.8813		0.0284			0.0067	0.0222
K	<DL	<DL		<DL	<DL		0.4545	1.5150
La	<DL	<DL		<DL	<DL		0.0435	0.1448
Li	<DL	<DL		(0.0009)			0.0138	0.0462
Mg	0.0191	0.0201		0.0034			0.0012	0.0038
Mn	0.0061	0.0064		0.0003			0.0021	0.0069
Mo	<DL	<DL		<DL	<DL		0.0717	0.2391
Na	9.1813	9.4701		0.0550			0.1494	0.4979
Nd	<DL	<DL		<DL	<DL		0.1056	0.3519
Ni	(0.0023)	(0.0025)		<DL	<DL		0.0173	0.0578
P	1.7769	1.8457		<DL	<DL		0.6388	2.1295
Pb	0.0493	0.0554		<DL	<DL		0.0909	0.3028
Re	<DL	<DL		<DL	<DL		0.0164	0.0547
Rh	<DL	<DL		<DL	<DL		0.3368	1.1227
Ru	<DL	<DL		<DL	<DL		0.0354	0.1180
Sb	<DL	<DL		<DL	<DL		0.6341	2.1135
Se	<DL	<DL		<DL	<DL		0.1113	0.3710
Si	0.0518	0.0500		0.0095			0.0560	0.1867
Sr	0.0205	0.0212		0.0001			0.0011	0.0036
Te	<DL	<DL		<DL	<DL		0.0809	0.2696
Th	<DL	<DL		<DL	<DL		0.1847	0.6157
Ti	<DL	<DL		<DL	<DL		0.0090	0.0300
Tl	<DL	<DL		<DL	<DL		2.6893	8.9644
U	<DL	<DL		<DL	<DL		1.7634	5.8779
V	<DL	<DL		<DL	<DL		0.0084	0.0279
Zn	0.0070	0.0071		0.0004			0.0046	0.0153
Zr	(0.0011)	(0.0017)		<DL	<DL		0.0088	0.0292
Bi	0.4519	0.4300		0.0054				

* Methods blank - average sample weight used to calculate wt%.

03/29/90

01011507116

TABLE 8.8 SST Core 1, Segment 3 Acid Leach ICP (Cont.)

	89-648b1		89-649b3		<Sample ID>		89-649b4		89-649b4		Spike	
	89-649b2		5.00		<Dilution>		1.00		5.00		STD	
	(Wt %)	RPD	(Wt %)	(Wt %)	(Wt %)	% Dif	% Rec	(ug/mL)	(ug/mL)	% Dif	(ug/mL)	% rec
Ag	N/A			<DL								
Al	0.0670	8.1%		0.0689								
As	N/A		0.0386	(0.0319)		82.6%		78.3	71.0	9.3%	80.0	97.9%
B	N/A			<DL								
Ba	0.0016	2.1%	0.0386	0.0392		97.3%		78.9	77.1	2.3%	80.0	98.6%
Be	N/A		0.0010	0.0010		100.8%		1.9	2.0	1.6%	2.0	96.3%
Ca	0.0747	6.3%		0.0751								
Cd	N/A		0.0010	(0.0022)		229.6%		3.3	3.8	14.1%	2.0	166.8%
Ce	N/A			<DL								
Co	N/A		0.0097	<DL		53.9%		18.2	2.3	87.5%	20.0	90.8%
Cr	0.0867	4.1%	0.0039	0.0922		140.9%		8.3	8.5	2.1%	8.0	103.7%
Cu	N/A		0.0048	0.0072		148.3%		11.0	10.7	3.0%	10.0	110.3%
Dy	N/A			<DL								
Fe	1.8480	3.6%		1.8863								
K	N/A			<DL								
La	N/A			<DL								
Li	N/A			<DL								
Mg	0.0196	5.2%		0.0197								
Mn	0.0063	4.4%	0.0097	0.0160		100.4%		20.2	19.8	2.0%	20.0	101.1%
Mo	N/A			<DL								
Na	9.3257	3.1%		9.4292								
Nd	N/A			<DL								
Ni	0.0024	9.4%	0.0097	0.0123		101.9%		20.3	20.7	2.3%	20.0	101.4%
P	1.8113	3.8%		1.7964								
Pb	0.0523	11.7%	0.0097	0.0658		140.0%		24.6	28.4	15.3%	20.0	123.1%
Re	N/A			<DL								
Rh	N/A			<DL								
Ru	N/A			<DL								
Sb	N/A		0.0097	<DL		141.3%		21.6	33.4	54.8%	20.0	107.8%
Se	N/A		0.0386	(0.0418)		108.2%		66.3	94.8	42.9%	80.0	82.9%
Si	0.0509	3.6%		0.0742								
Sr	0.0209	3.1%		0.0212								
Te	N/A			<DL								
Th	N/A			<DL								
Ti	N/A			<DL								
Tl	N/A		0.0386	<DL		-145.5%		68.9			80.0	86.1%
U	N/A			<DL								
V	N/A		0.0097	0.0099		102.6%		19.4	19.9	2.6%	20.0	97.0%
Zn	0.0070	1.2%	0.0097	0.0164		97.2%		20.5	20.8	1.1%	20.0	102.7%
Zr	0.0014	42.7%		<DL								
Bi	0.4409	5.0%		0.8584								

03/29/90

9112511871

TABLE 8.9 SST Core 1, Segments 4 and 5 Acid Leach ICP

Samp Log#:	89-650b1	89-650b2		89-651b1	89-651b2							
Dilution:	5.00	5.00		5.00	5.00							
Wt% Factor	0.11795	0.10786		0.12025	0.11115							
ICP Run #	1803	1804		1805	1806					DL	QL	
	(Wt %)	(Wt %)	RPD	(Wt %)	(Wt %)	RPD	(Wt %)	(Wt %)	RPD	(ug/mL)	(ug/mL)	
Ag	<DL	<DL		<DL	<DL					0.0126	0.0419	
Al	(0.0126)	<DL		(0.0288)	(0.0263)	9.3%				0.0886	0.2953	
As	<DL	<DL		<DL	<DL					0.1567	0.5223	
B	(0.0089)	<DL		<DL	<DL					0.0718	0.2395	
Ba	(0.0010)	(0.0008)	26.8%	0.0013	0.0013	5.2%				0.0033	0.0112	
Be	<DL	<DL		<DL	<DL					0.0004	0.0012	
Ca	0.0518	0.0495	4.4%	0.0515	0.0491	4.8%				0.0017	0.0058	
Cd	(0.0009)	<DL		(0.0008)	<DL					0.0060	0.0201	
Ce	<DL	<DL		<DL	<DL					0.2083	0.6942	
Co	<DL	<DL		<DL	<DL					0.3038	1.0128	
Cr	0.0964	0.0894	7.5%	0.0977	0.0943	3.5%				0.0065	0.0218	
Cu	(0.0030)	(0.0022)	29.9%	(0.0029)	(0.0025)	13.4%				0.0177	0.0589	
Dy	<DL	<DL		<DL	<DL					0.0163	0.0545	
Fe	1.6853	1.6409	2.7%	1.9130	1.9061	0.4%				0.0067	0.0222	
K	(0.0578)	<DL		<DL	<DL					0.4545	1.5150	
La	<DL	<DL		<DL	<DL					0.0435	0.1448	
Li	<DL	<DL		<DL	<DL					0.0138	0.0462	
Hg	0.0154	0.0151	2.1%	0.0113	0.0109	3.8%				0.0012	0.0038	
Mn	0.0068	0.0062	9.6%	0.0071	0.0068	4.9%				0.0021	0.0069	
Mo	<DL	<DL		<DL	<DL					0.0717	0.2391	
Na	9.8866	9.7769	1.1%	9.9590	9.9912	0.3%				0.1494	0.4979	
Nd	<DL	<DL		<DL	<DL					0.1056	0.3519	
Ni	0.0138	0.0107	25.4%	0.0143	0.0122	15.5%				0.0173	0.0578	
P	1.7649	1.7181	2.7%	1.6763	1.6432	2.0%				0.6388	2.1295	
Pb	<DL	<DL		(0.0229)	(0.0221)	3.5%				0.0909	0.3028	
Re	<DL	<DL		<DL	<DL					0.0164	0.0547	
Rh	<DL	<DL		<DL	<DL					0.3368	1.1227	
Ru	<DL	<DL		<DL	<DL					0.0354	0.1180	
Sb	<DL	<DL		<DL	<DL					0.6341	2.1135	
Se	(0.0166)	<DL		(0.0248)	(0.0133)	60.1%				0.1113	0.3710	
Si	0.0393	0.0394	0.1%	0.0798	0.0906	12.7%				0.0560	0.1867	
Sr	0.0170	0.0167	1.6%	0.0224	0.0226	0.7%				0.0011	0.0036	
Te	<DL	<DL		<DL	<DL					0.0809	0.2696	
Th	<DL	<DL		<DL	<DL					0.1847	0.6157	
Ti	<DL	<DL		<DL	<DL					0.0090	0.0300	
Tl	<DL	<DL		<DL	<DL					2.6893	8.9644	
U	<DL	<DL		<DL	<DL					1.7634	5.8779	
V	<DL	<DL		<DL	<DL					0.0084	0.0279	
Zn	0.0184	0.0170	8.2%	0.0136	0.0131	3.3%				0.0046	0.0153	
Zr	<DL	<DL		(0.0011)	<DL					0.0088	0.0292	
Bi	0.6289	0.4263	38.4%	0.4180	0.4213	0.8%						

03/29/90

91120511772

TABLE 8.10 SST Core 1, Composite Acid Leach ICP (Cont.)

	89-0621a1		89-0621a3		<Sample ID>		89-0621a4		89-0621a4		Spike	
	89-0621a2	Average	Spike Added	Spike+ Sample	<Dilution>		1.00	5.00	Spike Control	Spike Control	STD	
	(Wt %)	RPD	(Wt %)	(Wt %)	(Wt %)	% Dif	% Rec	(ug/mL)	(ug/mL)	% Dif	(ug/mL)	% rec
Ag	0.0021	20.0%		(0.0029)								
Al	0.1158	1.9%		0.1175								
As	N/A		0.0388	(0.0325)		83.8%	74.3	91.7	23.4%	80.0	92.9%	
B	N/A			<DL								
Ba	0.0013	1.3%	0.0388	0.0392		97.7%	76.7	75.6	1.5%	80.0	95.9%	
Be	N/A		0.0010	0.0009		97.6%	1.9	1.9	1.7%	2.0	93.2%	
Ce	0.0863	1.7%		0.0853								
Cd	N/A		0.0010	(0.0021)		211.8%	3.1	3.6	16.0%	2.0	156.4%	
Ce	N/A			<DL								
Co	N/A		0.0097	<DL		-55.9%	15.1	6.0	60.1%	20.0	75.6%	
Cr	0.0824	0.4%	0.0039	0.0879		141.2%	8.1	8.6	6.7%	8.0	101.3%	
Cu	N/A		0.0048	(0.0066)		137.1%	10.2	9.6	5.7%	10.0	101.8%	
Dy	N/A			<DL								
Fe	1.9433	1.6%		1.9565								
K	N/A			<DL								
La	0.0063	5.4%		(0.0063)								
Li	N/A			<DL								
Hg	0.0193	1.4%		0.0192								
Mn	0.0073	1.7%	0.0097	0.0169		99.0%	19.8	19.7	0.5%	20.0	98.9%	
Mo	N/A			<DL								
Na	9.6977	0.7%		9.8027								
Nd	N/A			<DL								
Ni	N/A		0.0097	0.0115		119.2%	19.6	19.6	0.3%	20.0	98.2%	
P	1.6536	3.9%		1.6983								
Pb	0.1132	5.7%	0.0097	0.1231		101.5%	19.9	19.2	3.6%	20.0	99.5%	
Re	N/A			<DL								
Rh	N/A			<DL								
Ru	N/A			<DL								
Sb	N/A		0.0097	<DL		572.0%	26.1	89.3	241.4%	20.0	130.7%	
Se	N/A		0.0388	(0.0327)		84.5%	61.6	78.6	27.6%	80.0	76.9%	
Si	0.0572	30.0%		0.0830								
Sr	0.0208	0.8%		0.0209								
Te	N/A			<DL								
Th	N/A			<DL								
Ti	N/A			<DL								
Tl	N/A		0.0388	<DL		102.9%	47.1	88.8	88.7%	80.0	58.8%	
U	N/A			<DL								
V	N/A		0.0097	0.0091		94.2%	18.8	18.4	2.3%	20.0	94.1%	
Zn	0.0092	1.1%	0.0097	0.0187		97.6%	19.9	19.8	0.5%	20.0	99.5%	
Zr	N/A			(0.0012)								
Bi	0.6795	84.1%		1.8327								

03/29/90

91120511974

INORGANIC WATER LEACH ICP DATA

A Core Composite sample was leached with deionized water (PNL-ALO-103), filtered, and distributed for analyses including analysis by ICP spectroscopy (PNL-SP-7). The sample preparation was completed in duplicate and included a Methods Blank. With the exception of detection limits (DL) and quantitation limits (QL), all data are reported in units of weight percent (Wt %) wet sample. If concentrations fall below the estimated DL then "< DL" will be listed. If the value falls between the DL and QL the the value will be reported in parentheses. Detection limits were estimated to be three times the standard deviation of the background. Quantitation limits were estimated as ten times the standard deviation of the background.

The Water Leach ICP sample in the Core 1 Data Package is from the Core Composite (Table 8.11). Column 1 of this table lists the atomic symbols of the 41 elements for which data are reported. Columns 2 and 3 tabulate the results for the analyses of the duplicate samples, and column 4 reports results for the Methods Blank. Columns 5 and 6 list DL and QL values, respectively, in units of micrograms per milliliter ($\mu\text{g/ml}$). The DL or QL in Wt % can be calculated for any element by multiplying the listed DL or QL (in $\mu\text{g/ml}$) by the appropriate Wt % Factor in row 3. Column 7 tabulates the average value for the duplicate analyses and Column 8 reports the relative percent difference (RPD).

911201975

TABLE 8.11 SST Core 1, Composite Water Leach ICP

	89-0621c1		89-0621c2		89-0621c3		89-0621c1		89-0621c2		RPD	
	Dilution:	10.00	10.00	10.00	1.00	1.00	Average	Average				
	Wt% Fctr:	0.10767	0.08969	0.00980			DL	QL				
	ICP Run#:	1740	1741	1739			(ug/mL)	(ug/mL)	(Wt %)			
	(Wt %)	(Wt %)	XDif	(Wt %)	(Wt %)	XDif	(Wt% *)	(Wt% *)	(ug/mL)	(ug/mL)	(Wt %)	RPD
Ag	<DL	<DL		<DL	<DL		<DL	<DL	0.0318	0.1060	N/A	
Al	<DL	<DL		<DL	<DL		<DL	<DL	0.2131	0.7103	N/A	
As	<DL	<DL		<DL	<DL		<DL	<DL	0.1670	0.5568	N/A	
B	<DL	<DL		<DL	<DL		<DL	<DL	0.0936	0.3120	N/A	
Ba	<DL	<DL		<DL	<DL		<DL	<DL	0.0029	0.0097	N/A	
Be	<DL	<DL		<DL	<DL		<DL	<DL	0.0009	0.0031	N/A	
Ca	0.0086			0.0072			0.0002		0.0026	0.0085	0.0079	18.0%
Cd	<DL	<DL		<DL	<DL		<DL	<DL	0.0091	0.0305	N/A	
Ce	<DL	<DL		<DL	<DL		<DL	<DL	0.2101	0.7005	N/A	
Co	<DL	<DL		<DL	<DL		<DL	<DL	0.2819	0.9396	N/A	
Cr	0.0065			0.0065			<DL	<DL	0.0089	0.0298	0.0065	0.8%
Cu	(0.0055)			(0.0018)			<DL	<DL	0.0174	0.0579	0.0036	101.3%
Dy	<DL	<DL		<DL	<DL		<DL	<DL	0.0120	0.0401	N/A	
Fe	0.0197			0.0126			<DL	<DL	0.0119	0.0396	0.0161	44.2%
K	<DL	<DL		<DL	<DL		<DL	<DL	0.3691	1.2303	N/A	
La	<DL	<DL		<DL	<DL		<DL	<DL	0.0211	0.0705	N/A	
Li	<DL	<DL		<DL	<DL		<DL	<DL	0.0148	0.0492	N/A	
Mg	0.0011			0.0010			0.0001		0.0012	0.0040	0.0010	14.5%
Mn	<DL	<DL		<DL	<DL		<DL	<DL	0.0026	0.0085	N/A	
Mo	<DL	<DL		<DL	<DL		<DL	<DL	0.0883	0.2945	N/A	
Na	8.6479			8.6702			(0.0014)		0.0587	0.1957	8.6591	0.3%
Nd	<DL	<DL		<DL	<DL		<DL	<DL	0.1370	0.4566	N/A	
Ni	<DL	<DL		<DL	<DL		<DL	<DL	0.0321	0.1070	N/A	
P	0.7796			0.7657			<DL	<DL	0.6650	2.2165	0.7727	1.8%
Pb	<DL	<DL		<DL	<DL		<DL	<DL	0.1055	0.3516	N/A	
Re	<DL	<DL		<DL	<DL		<DL	<DL	0.0363	0.1211	N/A	
Rh	<DL	<DL		<DL	<DL		<DL	<DL	0.1390	0.4633	N/A	
Ru	<DL	<DL		<DL	<DL		<DL	<DL	0.0624	0.2081	N/A	
Sb	(0.0521)			(0.0368)			<DL	<DL	0.3958	1.3194	0.0444	34.5%
Se	<DL	<DL		<DL	<DL		<DL	<DL	0.2546	0.8488	N/A	
Si	0.0396			0.0382			<DL	<DL	0.0657	0.2191	0.0389	3.6%
Sr	<DL	<DL		<DL	<DL		<DL	<DL	0.0021	0.0071	N/A	
Te	<DL	<DL		<DL	<DL		<DL	<DL	0.1019	0.3397	N/A	
Th	<DL	<DL		<DL	<DL		<DL	<DL	0.1577	0.5258	N/A	
Ti	<DL	<DL		<DL	<DL		<DL	<DL	0.0125	0.0417	N/A	
Tl	<DL	<DL		<DL	<DL		<DL	<DL	1.0988	3.6628	N/A	
U	<DL	<DL		<DL	<DL		<DL	<DL	1.5188	5.0628	N/A	
V	<DL	<DL		<DL	<DL		<DL	<DL	0.0104	0.0348	N/A	
Zn	(0.0007)			(0.0008)			<DL	<DL	0.0039	0.0131	0.0008	1.3%
Zr	<DL	<DL		<DL	<DL		<DL	<DL	0.0129	0.0429	N/A	
Bi											N/A	

* Methods blank - average sample weight used to calculate wt%.

03/29/90

91120511076

INORGANIC WATER LEACH ANION ANALYSIS RESULTS

An aliquot of the Water Leach sample preparation solution (PNL-ALO-102) was analyzed for anions by ion chromatography (7-40.8) and total carbon/total inorganic carbon (7-40.8). Each sample was prepared in duplicate. In addition, an anion spike was added to a third sample. A Spike Control sample was prepared by adding the same spiking solution to deionized water. Finally, each suite of analyses include a Methods Blank.

The Core 1 Data Package contains anion analysis data for Segments 2 through 5 and the Core Composite (Table 9.1 and 9.2). Column 1 of each of these tables list the anions for which data are reported. Columns 2 and 3 tabulate the analytical results for the analyses of the duplicate samples and column 4 lists the relative percent difference (RPD) between the samples. Column 5 lists the "as prepared" concentrations of the anion spiking solution and column 6 lists the "measured" concentrations that are derived from the Spike Control analysis data. Column 7 lists the calculated value of the "spike" added to the spiked sample. Column 8 reports the results from the spiked sample analysis. Percent recoveries (% Spk Rec) are listed in column 9. Estimated method detection limits are shown in column 10.

A comparison of data in columns 5 for the "as prepared" anion spike concentration with the measured concentrations in column 6 indicates that some of the nitrite in the spiking standard has decomposed to nitrate.

7
2
1
5
0
2
1
1
9

TABLE 9.1 SST Core 1, Segments 2, 3 and 4 Anion Analyses by IC/TOC Data

Sample: 89-0648
03/07/90

	C1 (mg/kg)	C2 (mg/kg)	RPD	-----Spike*Control--(a)-					C5 (c) (mg/kg)
				True (ug/mL)	C4----> (ug/mL)	C3 Spk (mg/kg)	C3 (mg/kg)	% Spk Rec (b)	
F	1540	1740	12.2%	100	104	592	2130	82.8%	<21
Cl	1030	1030	0.0%	150	122	693	1900	125.5%	<21
NO2	17000	17400	2.3%	1000	887	5030	22100	97.4%	<41
NO3	127000	131000	3.1%	1500	1833	10400	141000	(b)	187
PO4	18400	21000	13.2%	3000	3153	17900	36000	91.1%	<41
SO4	10400	10400		500	575	3260	13200	85.9%	508
TIC	1860	1800	3.3%						114
TOC	497	493	0.8%						114

- (a) The spike's measured concentration has been used to calculate the quantity of spike added in mg/kg.
- (b) The % spike recovery is valid only if the spike level is over 20% of the sample's base value.
- (c) The average Wt-gm of Sample 1 & 2 is used to calculate blank in mg/kg.

TABLE 9.1 SST Core 1, Segments 2, 3 and 4 Anion Analyses by IC/TOC Data (Cont.)

Sample: 89-0649
03/07/90

	C1 (mg/kg)	C2 (mg/kg)	RPD	-----Spike*Control--(a)--			C3 (mg/kg)	% Spk Rec (b)	C5 (c) (mg/kg)
				True (ug/mL)	C4---> (ug/mL)	C3 Spk (mg/kg)			
F	1690	1660	1.8%	100	102	438	2190	117.6%	<19
Cl	1060	856	21.3%	150	134	573	1560	105.1%	82
NO2	12000	12000	0.0%	1000	851	3640	15800	104.4%	<38
NO3	152000	158000	3.9%	1500	1868	8000	168000	(b)	157
PO4	27800	26400	5.2%	3000	3422	14700	36200	61.9%	<38
SO4	11300	11700	3.5%	500	629	2690	13900	89.2%	421
TIC	739	741	0.3%						<95
TOC	659	561	16.1%						156

- (a) The spike's measured concentration has been used to calculate the quantity of spike added in mg/kg.
- (b) The % spike recovery is valid only if the spike level is over 20% of the sample's base value.
- (c) The average Wt-gm of Sample 1 & 2 is used to calculate blank in mg/kg.

TABLE 9.1 SST Core 1, Segments 2, 3 and 4 Anion Analyses by IC/TOC Data (Cont.)

Sample: 89-0650
03/07/90

	C1 (mg/kg)	C2 (mg/kg)	RPD	-----Spike*Control--(a)-					C5 (c) (mg/kg)
				True (ug/mL)	C4----> (ug/mL)	C3 Spk (mg/kg)	C3 (mg/kg)	% Spk Rec (b)	
F	1750	1740	0.6%	100	89	377	1930	49.1%	<18
Cl	1080	1050	2.8%	150	120	511	1760	136.0%	<18
NO2	6880	7060	2.6%	1000	846	3610	10100	86.7%	<37
NO3	169000	176000	4.1%	1500	1809	7710	176000	(b)	148
PO4	23500	24700	5.0%	3000	2990	12700	33900	77.2%	<37
SO4	11100	11600	4.4%	500	564	2410	13100	72.6%	332
TIC	383	366	4.5%						92
TOC	423	392	7.6%						138

- (a) The spike's measured concentration has been used to calculate the quantity of spike added in mg/kg.
- (b) The % spike recovery is valid only if the spike level is over 20% of the sample's base value.
- (c) The average Wt-gm of Sample 1 & 2 is used to calculate blank in mg/kg.

TABLE 9.2 SST Core 1, Segments 5 and Core 1 Composite Anion Analyses by IC/TOC Data

Sample: 89-0651
03/07/90

	C1 (mg/kg)	C2 (mg/kg)	RPD	-----Spike*Control--(a)--			C3 (mg/kg)	% Spk Rec (b)	C5 (c) (mg/kg)
				True (ug/mL)	C4----> (ug/mL)	C3 Spk (mg/kg)			
F	1400	1410	0.7%	100	94	502	1990	116.5%	<18
Cl	950	967	1.8%	150	120	638	1930	152.3%	45
NO2	4490	4550	1.3%	1000	852	4530	9100	101.1%	<36
NO3	178000	183000	2.8%	1500	1798	9560	187000	(b)	153
PO4	22300	24200	8.2%	3000	2911	15500	34400	71.9%	<36
SO4	11100	11000	0.9%	500	574	3050	13400	77.0%	332
TIC	275	292	6.0%						<90
TOC	506	557	9.6%						117

- (a) The spike's measured concentration has been used to calculate the quantity of spike added in mg/kg.
- (b) The % spike recovery is valid only if the spike level is over 20% of the sample's base value.
- (c) The average Wt-gm of Sample 1 & 2 is used to calculate blank in mg/kg.

TABLE 9.2 SST Core 1, Segments 5 and Core 1 Composite Anion Analyses by IC/TOC Data (Cont.)

Sample: 89-0621
03/07/90

	C1 (mg/kg)	C2 (mg/kg)	RPD	-----Spike*Control--(a)--					C5 (c) (mg/kg)
				True (ug/mL)	C4---> (ug/mL)	C3 Spk (mg/kg)	C3 (mg/kg)	% Spk Rec (b)	
F	1600	1700	6.1%	100	89	484	2100	93.0%	<20
Cl	1050	987	6.2%	150	124	677	1770	111.0%	39
NO2	9850	9870	0.2%	1000	788	4300	14800	114.9%	<39
NO3	155000	168000	8.0%	1500	1763	9620	178000	(b)	44
PO4	24200	26500	9.1%	3000	2836	15500	41200	102.3%	<39
SO4	10300	10700	3.8%	500	492	2690	14800	159.9%	55
TIC	1200	1070	11.5%						<98
TOC	398	439	9.8%						147

- (a) The spike's measured concentration has been used to calculate the quantity of spike added in mg/kg.
- (b) The % spike recovery is valid only if the spike level is over 20% of the sample's base value.
- (c) The average Wt-gm of Sample 1 & 2 is used to calculate blank in mg/kg.

OTHER INORGANIC RESULTS

Graphite furnace atomic absorption spectroscopy analyses were performed on samples prepared by the acid leach method (PNL-ALO-101). This procedure leads to significant sample dilutions and this is reflected in the reported detection limits.

The colorimetric chromium (VI) and ammonia were completed on aliquots of the water leach sample. Mercury analyses sample preparation followed the protocols outlined in the EPA-CLP statement of work. The analyses were completed in duplicate, a third sample split was spiked with mercury and a fourth sample was prepared by spiking the mercury standard into deionized water. Finally, a methods blank was run.

No cyanide data are available at this time.

91120541913

TABLE 10. SST Core 1, Other Inorganic Analyses

Sample Number 89-621

Graphite-Furnace Atomic Absorption Data

Element	Sample B-1 mg/kg	Sample B-2 mg/kg	Calc. Spike Added mg/kg	Sample B-3 with Spike mg/kg	Spike Control B-4 Calculated ml/l	Measured ml/l	Methods Blank- B-5 mg/kg
As	< 23	< 25	347	335	80	76.2	< 2.5
Se	< 23	< 25	347	321	80	72.5	< 2.5

Colorimetric Cr(VI) Analysis Results*

Sample C-1	Sample C-2	Methods Blank C-5
67 mg/kg	67 mg/kg	< 20 mg/kg

* Cr(VI) results may be unreliable. Subsequent work has indicated that sample matrix effects cause a positive bias.

Ammonia Analysis Results

Sample C-1	Sample C-2	Methods Blank C-5
288 mg/kg	230 mg/kg	8 mg/kg

Mercury Results for Core 1 Composite

Sample D-1 mg/kg	Sample D-2 mg/kg	Calc. Spike Added mg/kg	Sample D-3 with Spike mg/kg	Spike Control D-4 Calculated ml/l	Measured ml/l	Methods Blank- D-5 mg/kg
0.9130	1.1270	0.2570	1.1380	0.0500	0.0550	< 0.018

Cyanide Results are unavailable at this time.

91120511914

RADIOCHEMICAL DATA TABLES

9 1 1 2 0 5 1 1 9 1 5

THIS PAGE
INTENTIONALLY
LEFT BLANK

RADIOCHEMICAL RESULTS

Radiochemical analyses were completed on selected segment and core composite samples. The solution resulting from the potassium hydroxide (KOH) fusion (PNL-ALO-102) were split between the inorganic and radiochemistry groups. Radiochemical analyses completed on this solution include Gamma Energy Analysis (GEA), Total Alpha, Total Beta, and Total Uranium. These radiochemical analyses were also completed for the Water Leach sample (PNL-ALO-102). GEA and uranium data are reported in Table 11. The rest of the radiochemistry results are reported in Table 12.

Plutonium and uranium isotopic analyses are unavailable because their levels in water leach and KOH fusions samples are too low for existing radiochemistry procedures within the 325 Building.

9 1 1 2 3 5 4 1 8 1 6

TABLE 11. SST CORE 1 - GAMMA ENERGY ANALYSIS DATA*

Sample #	Cs-137 uCi/g-wet 662 keV	Percent Error +/-	Eu-155 uCi/g-wet 86.6 keV	Percent Error +/-	Eu-154 uCi/g-wet 1274 keV	Percent Error +/-
Segment 2						
89-0331-A-2	13.04	0.43	1.192	3.47	0.768	3.82
89-0331-B-2	37.92	0.26	1.23	3.6	0.867	3.6
89-0332-A-2	17.50	0.33	0.932	3.5	0.612	3.8
89-0332-B-2	17.20	0.11	0.954	1.2	0.592	1.32
89-0648-A-1	17.61	0.33	0.977	2.43	0.657	3.75
89-0648-A-2	16.99	0.32	1.018	3.39	0.671	3.55
89-0648-A-3**	0.043	1.590	0.003	13.580	0.007	9.7
Segment 3						
89-0333-A-2	14.14	0.35	0.331	6.8	0.166	7.9
89-0333-B-2	13.35	0.36	0.271	7.4	0.140	8.2
89-0334-A-2	12.31	0.40	0.253	7.9	0.140	8.6
89-0334-B-2	13.73	0.37	0.267	7.8	0.148	8.0
Segment 4						
89-0335-A-2	11.81	0.39	-	-	-	-
89-0335-B-2	11.33	0.38	-	-	-	-
89-0336-A-2	11.66	0.42	-	-	-	-
89-0336-B-2	11.20	0.39	-	-	-	-
Segment 5						
89-0337-A-2	13.70	0.36	0.089	16.6	0.062	13
89-0337-B-2	13.35	0.35	0.112	13.6	0.065	11
89-0338-A-2	11.31	0.40	0.101	13.3	0.067	11.1
89-0338-B-2	13.01	0.36	0.127	11.0	0.070	12
Core 1 Composite Homogenization Check Samples						
89-0622-A-2	14.40	0.26	0.601	3.34	0.391	3.55
89-0622-B-2	14.66	0.29	0.617	3.67	0.384	3.85
89-0623-A-2	15.01	0.26	0.617	3.41	0.382	3.56
89-0623-B-2	14.58	0.26	0.601	3.17	0.401	3.17
Core 1 Composite - Water Leach						
89-0621-C1	8.44	0.17	0.029	11.7	0.012	11.
89-0621-C-2	8.41	0.15	0.020	14.94	0.0084	12.
89-0621-C-5**	0.005	5.6	0.009	3.7	0.007	

* Samples A-2 and B-2 are duplicates.

** Methods Blanks

9112751917

TABLE 11. SST CORE 1 - GAMMA ENERGY ANALYSIS DATA (Cont.)

(CONT) Sample #	SST Nuclide	Additional Nuclides		Percent Error +/-
		Energy keV	uCi/g-wet	
Segment 2				
89-0331-A-2	Am-241	59.50	0.297	11.5
	Ce-144	133.54	0.396	20.2
89-0331-B-2	Am-241	59.50	0.327	12
	Ce-144	133.54	0.463	21.8
	Cs-134	795.76	0.111	9.1
89-0332-A-2	Am-241	59.50	0.223	11.50
	Ce-144	133.54	0.306	23
89-0332-B-2	Am-241	59.50	0.212	4.4
	Ce-144	133.54	0.311	7.4
	Cs-134	795.76	0.037	6.5
89-0648-A-1	Am-241	59.50	.231	11.4
	Ce-144	133.54	0.338	21
89-0648-A-2	Am-241	59.50	0.241	10.5
	Ce-144	133.54	0.273	25
89-0648-A-3**	Cs-134	795.76	0.30	24
	Co-60	1332.47	0.03	
Segment 3				
89-0333-A-2	Am-241	59.50	0.068	22
	Ce-144	133.54	0.159	24.5
89-0333-B-2	Am-241	59.50	0.055	26
	Ce-144	133.54	0.188	24.2

* Samples A-2 and B-2 are duplicates.

** Methods Blank

TABLE 11. SST CORE 1 - GAMMA ENERGY ANALYSIS DATA (Cont.)

Sample #	Nuclide	Additional Nuclides		Percent Error +/-
		Energy keV	uCi/g-wet	
89-0334-A-2	Am-241	59.50	0.056	25
	Ce-144	133.54	0.214	24
89-0334-B-2	Am-241	59.50	0.079	18
	Ce-144	133.54	0.210	25
Segment 5				
89-0337-A-2	Am-241	59.50	0.025	44
	Ce-144	133.54	0.141	28.3
	Cs-134	795.76	0.0099	23
	Ru-106	622.20	0.151	39
89-0337-B-2	Am-241	59.50	0.036	25
	Ce-144	133.54	0.074	52.5
89-0338-A-2	Am-241	59.50	0.025	36
	Ce-144	133.54	0.292	14.6
	Cs-134	795.76	0.034	10.3
	Ru-106	622.20	0.158	34
89-0338-B-2	Am-241	59.50	0.035	31
	Ce-144	133.54	0.206	21
	Cs-134	795.76	0.014	17
	Ru-106	622.20	0.188	32
Composite				
89-0621-C-1	Cs-134	795.76	0.0043	16
89-0621-C-2	CS-134	795.76	0.0046	15
89-0622-A-2	Am-241	59.50	0.1362	10.8
	Ce-144	133.54	0.269	17.5
89-0622-B-2	Am-241	59.50	.139	11.9
	Ce-144	133.54	0.235	20.0
89-0623-A-2	Am-241	59.50	.134	11.4
	Ce-144	133.54	0.228	19.0
89-0623-B-2	Am-241	59.50	0.145	9.5
	Ce-144	133.54	0.213	19.0
	Ru-106	622.20	0.121	33.0

* Samples A-2 and B-2 are duplicates.

9112051319

TABLE 11: SST Data Results for Core 1 - GEA Data (Cont'd)

Customer No.	Counting Time	Cs-137		Eu-155		Eu-154		Additional Nuclides			
		uCi/g-wet 662 keV	+/- % error	uCi/g-wet 105 keV	+/- % error	uCi/g-wet 1274 keV	+/- % error	Nuclide	Energy keV	uCi/g-wet	+/- % error
Composite 4											
89-0980-A-2	30 min.	14.49	0.31	0.2312	6.20	0.2259	5.58	Co-60	1332.47	0.00711	21.42
								Gd-153	103.2	0.2232	6.52
								Am-241	59.5	0.067	17.86
89-0980-B-2	30 min.	14.38	0.30	---		0.6040	3.16	Am-241	59.5	0.07611	18.64
								Gd-153	103.2	0.91830	1.85
89-0981-A-2	30 min.	14.39	0.3	0.2278	5.33	0.2759	5.03	Cs-134	604.66	0.01602	33.75
								Am-241	59.5	0.07498	17.98
								Co-60	1332.47	0.005409	28.85
								Gd-153	103.2	0.2881	4.27
89-0981-B-2	30 min.	15.03	0.3	0.2405	5.14	0.2689	4.73	Cs-134	604.66	0.0224	22.86
								Am-241	59.5	0.08568	16.01
								Co-60	1332.47	0.01183	16.19
								Gd-153	103.2	0.2453	5.01

All errors are due to counting statistics only and are at the one sigma level.

0 - 3 1 5 2 1 1 9

TABLE 12. SST CORE 1 RADIOCHEMISTRY DATA*
TOTAL ALPHA, TOTAL BETA AND URANIUM

Sample No.	Tot alpha dpm/g *	+/- % Uncert.	Tot beta (dpm/g) @Sr90-Y90	+/- % Uncert.	Uranium ug/g wet wt.	+/- % Uncert.
Segment 2						
	Fusion Samp					
89-0331-A-2	6.89E+05	30	1.94E+09	4.6	470	6
89-0331-B-2	7.84E+05	30	2.34E+09	3.8	600	5.2
89-0332-A-2	7.94E+05	30	1.91E+09	3.8	522	5.1
89-0332-B-2	7.11E+05	30	1.78E+09	3.8	470	5.8
89-0648-A-1	7.71E+05	30	2.00E+09	4.8	546	8.6
89-0648-A-2	7.94E+05	30	1.95E+09	4.6	570	5.5
89-0648-A-3**	3.50E+03	30	<1.5E+06		1.62	24.0
Segment 3						
	Fusion Sample					
89-0333-A-2	3.34E+05	30	5.68E+08	3.9	230	7.8
89-0333-B-2	2.96E+05	30	5.57E+08	3.8	220	7.8
89-0334-A-2	3.48E+05	30	5.63E+08	3.8	210	8.8
89-0334-B-2	3.56E+05	30	5.80E+08	3.8	220	8.2
Segment 4						
	Fusion Sample					
89-0335-A-2	1.17E+05	30	5.14E+07	3.6	45	8.5
89-0335-B-2	1.40E+05	30	4.89E+07	3.6	43	8.2
89-0336-A-2	1.29E+05	30	5.02E+07	3.6	44	9.6
89-0336-B-2	1.08E+05	30	4.86E+07	3.6	42	8.8
Segment 5						
	Fusion Sample					
89-0337-A-2	2.00E+05	30	2.21E+08	3.5	35	8.4
89-0337-B-2	1.91E+05	30	2.23E+08	3.5	36	8.3
89-0338-A-2	2.12E+05	30	2.17E+08	3.6	37	8.3
89-0338-B-2	2.02E+05	30	2.25E+08	3.6	58	8.3
Core Composite						
	Fusion Sample					
89-0622-A-2	5.27E+05	30	1.12E+09	4.7	236	8.8
89-0622-B-2	4.86E+05	30	1.13E+09	4.9	240	11
89-0623-A-2	4.97E+05	30	1.11E+09	4.8	231	10
89-0623-B-2	4.75E+05	30	1.16E+09	4.6	233	8.9
Core Composite						
	Water Leach					
89-0621-C-1	1.54E+04	30	3.75E+07	3.7	0.89	10
(rerun)			3.53E+07	3.0		
89-0621-C-2	1.04E+04	30	2.67E+07	3.6	0.88	10
(rerun)			2.53E+07	3.5		
89-0621-C-5**	8.40E-01	30	9.80E+03	7.0	< 1	

* Samples A-2 and B-2 are duplicates

** Methods Blank

TABLE 12. SST CORE 1 RADIOCHEMISTRY DATA (Cont.)

Sr-90, Tc-99 and I-129 DATA

Customer No.	Sr-90 dpm/g	+/- % Uncert.	Tc-99 dpm/g	+/- % Uncert.	I-129 dpm/g	+/- % Uncert.
Core Composite	Fusion Samp					
89-0622-A-2	4.72E+08	8.7	4.33E+04	9.2	<3.1E+01	
89-0622-B-2	5.23E+08	8.7	4.85E+04	9.3	<3.8E+01	
89-0623-A-2	5.12E+08	8.6	4.69E+04	9.0	6.70E+01	50
89-0623-B-2	4.21E+08	8.6	4.56E+04	9.1	6.60E+01	43
Core Composite	Water Leach					
89-0621-C-1	4.36E+05	11.0	4.17E+04	4.0	NA	
89-0621-C-2	5.40E+05	9.7	3.86E+04	4.0	<2.2E+00	
89-0621-C-5**	9.80E+02	25.0	2.60E+02	48.0	Blank	

ALPHA ENERGY ANALYSIS OF Pu and Np

Sample No.	Pu 239&240 dpm/g	+/- % Uncert.	Pu 238 dpm/g	+/- % Uncert.	Np 237 dpm/g	+/- % Uncert.
Core Composite	Fusion Samp					
89-0622-A-2	2.43E+05	6.8	7.7E+03	21	2.10E+02	33.00
89-0622-B-2	2.50E+05	7.1	7.9E+03	21	2.40E+02	22.00
89-0623-A-2	2.58E+05	6.8	9.0E+03	21	1.10E+03	30.00
89-0623-B-2	2.14E+05	6.8	7.4E+03	21	6.70E+02	16.00
Core Composite	Water Leach					
89-0621-C-1	3.22E+03	11.0	NA			
(rerun)	4.47E+03	9.8	2.7E+02	42		
89-0621-C-2	4.25E+03	9.7	NA			
(rerun)	3.43E+03	10.2	2.0E+02	42		
89-0621-C-5**	3.10E+02	12.0				

ALPHA ENERGY ANALYSIS OF Am AND Cm

Sample No.	Am 241 dpm/g	+/- % Uncert.	Cm 243&244 dpm/g	+/- % Uncert.
Core Composite	Fusion Samp			
89-0622-A-2	3.05E+05	8.1	7.4E+03	21
89-0622-B-2	2.93E+05	7.1	---	
89-0623-A-2	2.93E+05	7.3	---	
89-0623-B-2	3.12E+05	7.9	4.9E+03	19
89-0621-C-1	1.20E+04	9.2	---	
89-0621-C-2	8.12E+03	9.7	1.7E+02	48
89-0621-C-5	<2.1E+02			

CORE 1 COMPOSITE TRITIUM AND CARBON 14 RESULTS

Sample No.	H-3 dpm/g	+/- % Uncert.	C-14 dpm/g	+/- % Uncert.
89-0621-C-1	3000	22		
89-0621-C-2	3500	20		
89-0621-C-3			54000	NA

* Samples A-2 and B-2 are duplicates

** Methods Blank

TABLE 12. SST CORE 1 RADIOCHEMISTRY DATA (Cont.)

URANIUM MASS SPEC. ISOTOPE RATIOS

Sample No.	U-234/U238	+/- 2 sigma	U-235/U-238	+/- 2 sigma	U-236/U-238	+/- 2 sigma
Core Composite	Fusion Samp					
89-0622-A-2	0.000060	0.000018	0.006752	0.000075	0.000119	0.000019
89-0622-B-2	0.000078	0.000071	0.006727	0.000186	0.000120	0.000036
89-0623-A-2	0.000062	0.000020	0.006729	0.000077	0.000109	0.000029
89-0623-B-2	0.000056	0.000010	0.006748	0.000036	0.000115	0.000010

PLUTONIUM MASS SPEC. ISOTOPE RATIOS

Sample No.	Pu238/Pu239	+/- 2 sigma	Pu240/Pu239	+/- 2 sigma	Pu241/Pu239	+/- 2 sigma
Core Composite	Fusion Samp					
89-0622-A-2	0.000832	0.000120	0.029735	0.000219	0.000634	0.000034
89-0622-B-2	0.000265	0.000043	0.029072	0.000301	0.000508	0.000029
89-0623-A-2	0.000387	0.000107	0.029805	0.000215	0.000549	0.000029
89-0623-B-2	0.000897	0.000406	0.029925	0.000306	0.000657	0.000043

Customer No.	Pu242/Pu239	+/- 2 sigma
Core Composite	Fusion Samp	
89-0622-A-2	0.000369	0.000026
89-0622-B-2	0.000193	0.000035
89-0623-A-2	0.000205	0.000029
89-0623-B-2	0.000374	0.000033

* Samples A-2 and B-2 are duplicates

** Methods Blank

ORGANIC DATA TABLES

91120541374

THIS PAGE
INTENTIONALLY
LEFT BLANK

ORGANIC COMPOUND ANALYSIS REPORT

SAMPLE ANALYSIS REPORTED

Analysis for Semivolatile organic compounds in Core 1 Composite samples is the subject of this report.

SAMPLE DESCRIPTION AND PREPARATION

Four Core 1 Composite samples of approximately 1-g each plus a method blank were analyzed. There is no blank soil/sediment available, so only the methylene chloride solvent with sodium sulfate and the spiked surrogate compounds were carried through the procedure. Two of the samples were treated as duplicates and the others were used for the matrix spike (MS) and matrix spike duplicate (MSD).

The preparation of these samples was performed in the 325 building hot cells. After addition of sodium sulfate, spiking compounds, and methylene chloride to the samples, the mixtures were sonicated, and aliquots were taken for GC screens. Results indicated that a 5-fold dilution would be required if the normal concentration of the 5-ml analytical aliquot were performed. Consequently, rather than doing the evaporation (which results in a 5-fold concentration) as called for in the procedure, the unconcentrated samples were taken for the GC/MS analyses.

ANALYSIS METHOD

The procedures followed for these analyses was PNL-ALO-340 for the screen and PNL-ALO-345 for the GC/MS. The HP-5890 GC (WB60701) for the screens is in lab 330 and the HP-5890/5970 GC/MS (WB38473) for the analyses is the #1 system in lab 427.

QUALITY CONTROL

The QC procedures were followed. The following is a list of the CLP form reporting the QC results.

<u>Form</u>	<u>Information</u>	<u>Comments</u>
2D	Surrogate Recovery	All Passed
3D	MS/MSD Recovery	Some Outside Limits
4B	Method Blank Summary	Meets Requirements
5B	Tune/Mass Calibration	2 ea, Meet Requirements
6B,C	Initial Calibration	5-Point, Meets Requirements
7B,C	Daily Calibration	2-Sets, Meet Requirements
8B,C	Internal Standards	2-Sets, Meet Requirements

The only deviations from QC requirements were in the matrix spike recoveries which were all high. The reason for the high results is not known.

DATA

The data and calibration files are archived on magnetic tape in lab 427 in the 325 building. The following is the list of the pertinent files.

<u>File Name</u>	<u>Sample Number</u>	<u>GC/MS Analysis</u>
>AL15A		DFTPP Tune Check
>AL15B		Daily Calibration
>AL15C		Method Blank
>AL15D	89-621-E-1	C-1 Composite
>AL15E	89-621-E-2	C-1 Composite
>AL15F	89-621-E-3	C-1 Composite MS
>AL21A		DFTPP Tune Check
>AL21B		Daily Calibration
>AL21C	89-621-E-4	C-1 Composite MSD

RESULTS

CLP Target Compounds: No CLP target compounds were detected. The Form 1B,C's list the target compounds, the Contract Required Quantitation Limits (the value followed by the U flag), and the notation that the sample was diluted by the D flag. Note that the concentration values are ug/g (ppm).

Tentatively Identified Compounds (TIC): The only TIC's found were the NPH alkanes. Concentration estimates of the TIC's are made by assuming that the response factors are unity. Then the peak areas of the TIC's are compared to the nearest internal standard (as for the target compounds) to estimate the concentrations. The results for these compounds are shown in the CLP Forms 1F and summarized as follows.

<u>Sample</u>	<u>Compound</u>	<u>Concentration, ug/g</u>
Core 1 Composite	Dodecane	360
	Tridecane	1100
	Tetradecane	700
Core 1 Composite	Dodecane	490
	Tridecane	1500
	Tetradecane	940
Core 1 composite MS	Dodecane	410
	Tridecane	1200
	Tetradecane	830
Core 1 Composite MSD	Dodecane	390
	Tridecane	1100
	Tetradecane	760

91120571376

COMMENTS

There are problems in taking samples and maintaining them until they are received in the organic labs that lead to concern about the results. Lack of temperature control can lead to loss of organics. Of more concern is the introduction of foreign substances during the sampling process. The use of NPH makes it necessary to dilute the samples such that any organics native to the samples may not be detected. One fortunate aspect to the dilution is that the severity of the effects of the added silicone lubricant and Led Plate on the GC/MS systems is lessened. There are components in one or both of these two formulations that have a serious effect on both the semivolatile and volatile GC/MS systems. The problems are seen in loss of spike recoveries, shifts in chromatographic retention times, general degradation of results, and recurrent instrumental problems that require extensive corrective action and maintenance.

ANALYST *Richard* DATE *1/6/90* REVIEW *C. K. Jones* DATE *1-9-90*

7
1
3
4
5
6
7
8
9
1
1
2
3
4
5
6
7
8
9

ORGANIC COMPOUND ANALYSIS REPORT

SAMPLE ANALYSIS REPORTED

Analysis for **Volatile** organic compounds in Core 1, segments 2 through 5 are the subject of this report.

SAMPLE DESCRIPTION AND PREPARATION

Samples were received from the Shielded Analytical Laboratory on August 23, 1989. Aliquots of approximately one gram were provided in pre-cleaned and vendor certified 20 ml vials. Silicone/Teflon septa were used for the closures. Considerable headspace was noted above the samples. Additionally, the samples were received at room temperature.

The sample preparation and storage observations are contrary to proper treatment as specified in "USEPA Contract Laboratory Program, Statement of Work for Organic Analysis", February 1988. The method specifies minimum headspace above solid samples and storage at 4 ± 2 degrees centigrade.

Hexadecane extractions for screen analysis were performed according to SW-846, "Test Methods for Evaluating Solid Waste", method 3820. Sample and solvent quantities were modified. Four mls of blank water and 1 ml of hexadecane were added to one gram samples. Standards and spikes were performed by addition of 20 ul of desired compounds in methanol. Standard and target spike levels were 2000 ug/Kg.

Screen data indicated substantial dilutions of the samples were required in order to perform analysis within the working range of the GC/MS system. Samples were prepared for GC/MS analysis by methanol extraction according to method PNL-ALO-335, "GC/MS Analysis of Volatile Organic Compounds". In this method, a quantity of methanol is added to the sample. An aliquot is taken and diluted into 5 mls of blank water and purged. No surrogate was employed during the dilution process. Surrogates and spikes were used only during the purging procedure.

91120541339

9 1 1 2 0 5 1 1 9 9

ANALYSIS METHOD

Headspace screens were performed according to document PNL-ALO-331, "Screening of Headspace Extracts for Volatile Organic Compounds" (in approval process). The method is a modification with respect to sample size and spike levels from those specified in SW-846, "Test Methods for Evaluating Solid Waste", method 3810. One gram of sample was contained in a 20 ml vial. The sample was not transferred to another vial. The sample was heated in the vial as provided to minimize volatile compound losses. Standard and target spike levels were 2000 ug/Kg.

Hexadecane residues were analyzed directly following extraction as described above. The analysis employed method PNL-ALO-330, "Screening of Hexadecane Extracts for Volatile Organic Compounds". Both screening methods employed a HP-5890 GC (WB60701) located in lab 330, 325 building.

GC/MS analysis was performed using method PNL-ALO-335. This employed a HP-5890/5970 GC/MS (WB38464) and is described as system 2 in lab 427, 325 building.

QUALITY CONTROL

Analysis by method PNL-ALO-331, headspace screens, were performed on 9/7/89, this exceeds the 14 day holding time specified in SW-846 by 2 days.

Analysis of samples 89-322, 89-323, and 89-329 using method PNL-ALO-330, hexadecane volatile screens, were performed on 10/11/89, this exceeds the 14 day holding time by 36 days. Sample 89-324 was analyzed on 1/4/90, exceeding the holding time by 120 days. It was uncertain if there was sufficient sample to perform the hexadecane screen on sample 89-324 so analysis was performed at the later date.

GC/MS analyses, by method PNL-ALO-335, were performed on 12/7/89 for samples 89-322, 323, and 324, this exceeds the 14 day holding time by 91 days. Sample 89-329 and spikes on 89-324 were performed on 12/13/89, this exceeds the holding time by 97 days. While every attempt is made to meet holding times, the ability to do so was effected by sample priority, instrument down time, and personnel availability.

Quality control procedures for these methods were followed. The following is a summary of the reporting forms.

<u>Form</u>	<u>Information</u>	<u>Comments</u>
2B	Surrogate Recovery	2 sets. Some outside limits (All of the first eluting 1,2-Dichloroethane-d4 passed QC limits).
3B	MS/MSD Recovery	2 sets. Some outside limits.
4A	Method Blank Summary	2 sets. Meets all requirements.
5A	Tune/ Mass Calibration	2 sets. Meets all requirements.
6A	Initial Calibration Data	5 point. Meets all requirements.
7A	Daily Calibration	2 sets. Meets all requirements.
8A	Internal Standards	2 sets. Some outside limits.

Deviations from quality control requirements for surrogate and spike compound recoveries were observed for samples on each day of analysis. For all samples, when deviations were observed, they were high. This indicates matrix interference contributing abundance at the primary, quantitative, mass. In addition, interferences covered a broad mass range and contributed to the secondary ions as well. While this skewed mass ratios for some compounds, they were still able to meet the specified criteria and resulted in elevated recoveries. The interfering compounds also carried over from one analysis to the next under the specified run parameters for this method. The carry over inhibits comparison of duplicate analysis and resulted in some relative percent difference data to exceed QC limits also.

Response of the Internal Standards was shown to trend downward and sometimes exceeded the quality control limits (Form 8A). While lower response of internal standards could contribute to high recoveries being calculated for other compounds, no similar trend was observed for the surrogate recovery data.

The concentration of surrogate compounds for the continuing calibration performed on 12/13/89 were one and two thirds too high. The surrogate recoveries are calculated based on the true concentration as reflected on Form 2B for 12/13/89.

0
1
2
3
4
5
6
7
8
9

DATA

The data and calibration files are archived on magnetic tape in lab 427 in the 325 building. The pertinent data files are listed on form 4A as "Lab File ID".

RESULTS

CLP Target Compounds: No CLP target compounds were detected above the Contract Required Quantitation Limits. Form 1A for each sample lists the target compounds. The column "Q" on the Form 1A indicates the EPA defined data qualifier as defined below:

<u>"Q" Flag</u>	<u>Definition</u>
U	Indicates compound was analyzed for but not detected
J	Indicates an estimated value. Spectra meet criteria but response is below a quantifiably confident level
B	Indicates compound was found in the blank
D	Indicates analysis was performed on a diluted sample

Tentatively Identified Compounds (TIC): Primarily the only TIC's found were the normal paraffin hydrocarbon alkanes. Concentration estimates of TIC's are made by assuming that the response factors are unity. Quantitation is then based on the nearest eluting internal standard. The results for these compounds are shown on Form 1E for each sample. The three typical identified compounds were dodecane, tridecane, and tetradecane ranging in concentration from 4000 to 66000 ug/Kg each.

COMMENTS

Samples were received and stored at ambient temperatures during sample processing. Only after final processing in the shielded "B" hot cell facility were samples stored at the required four degrees centigrade. One gram aliquots of sample intended for volatile analysis were placed in 20 ml sample bottles resulting in large headspace. Volatile compounds can then be lost by diffusing into the large headspace, particularly while stored at room temperature.

Percent levels of the normal paraffin hydrocarbons, primarily in the form of Dodecane, Tridecane, and Tetradecane were present in each segment of the

core samples. The large quantity of these compounds present required that the samples be substantially diluted before GC/MS analysis could be performed. The dilutions resulted in considerably elevated detection limits than those specified by the method. In addition, even with the dilutions, the difficulties with these analyses included poor surrogate and spike compound recoveries, substantial chromatographic performance degradation and retention time shifts. The instrumentation had considerable downtime and extensive maintenance following analysis of these samples. Attempts at corrective action to maintain the instrument response and retention time windows have failed.

A "trip" and "sample processing" blank and spike (of actual samples) at levels reasonable to analyze is needed to assess the effects of current sample handling techniques. The spike should be at more than one level and include all of the target compounds.

ANALYST Mark M. Bunn DATE 1-24-90 REVIEW [Signature] DATE 1-24-90

CORE 1 VOLATILE TIC COMPOUNDS*

	<u>Segment 2</u>	<u>Segment 2-Dup</u>	<u>Segment 3</u>	<u>Segment 4</u>	<u>Segment 5</u>
Dodecane	9000	8200	37000	22000	22000
Tridecane	14000	25000	51000	58000	66000
Tetradecane	4000		15000	7800	49000

* $\mu\text{g/Kg}$

9112031972