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Tank Characterization Report for Single-Shell Tank 241-BX-107

G. F. Raphael

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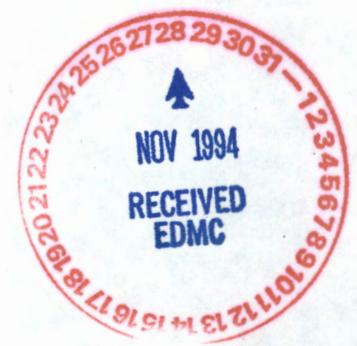
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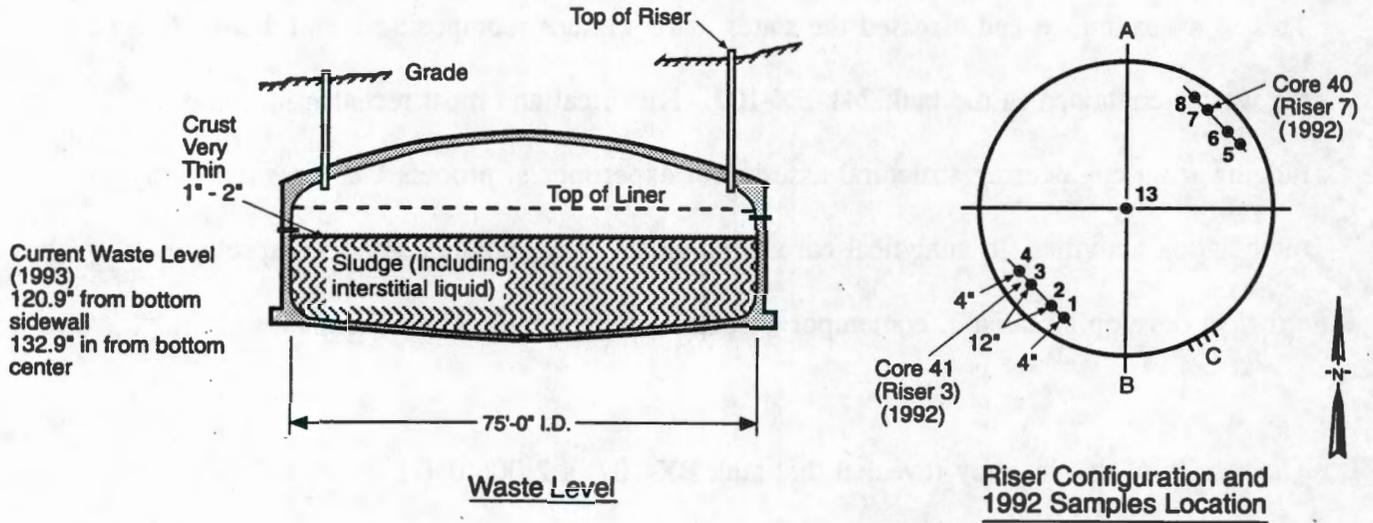
EXECUTIVE SUMMARY

This study examined and assessed the status, safety issues, composition, and distribution of the wastes contained in the tank 241-BX-107. Historical and most recent information, ranging from engineering structural assessment experiments, process history, monitoring and remediation activities, to analytical core sample data, were compiled and interpreted in an effort to develop a realistic, contemporary profile for the tank BX-107 contents.

The results of this study revealed that tank BX-107, a 2,006,050 L (530,000 gal) cylindrical single-shell, dished-bottom carbon-steel tank in the 200 East Area of the Hanford Site, was classified as sound. It has been interim stabilized and thus contains less than 189,250 L (50,000 gal) of interstitial liquid, and less than 18,925 L (5,000 gal) of supernatant. It has also been partially interim isolated, whereby all inlets to the tank are sealed to prevent inadvertent addition of liquid.

At a residual waste level of ~ 3.07 m (120.7 ± 2 in. from sidewall bottom or ~ 132.9 in. from center bottom), it is estimated that the tank BX-107 contents are equivalent to 1,305,825 L (345,000 gal). The vapor space pressure is at atmospheric. The latest temperature readings, which were taken in July 1994, show a moderate temperature value of 19 °C (66 °F). Two supernatant samples were collected in 1974 and 1990, prior to interim stabilization. Sludge core samples were obtained in 1979 and 1992 (see Figure ES-1).

Figure ES-1. Waste Level, Riser Configuration, and 1992 Samples Location.



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Based on available data, no evidence of significant vertical or horizontal waste layering was found. Table ES-1 provides summary information relative to tank BX-107 structure, status, and contents. Table ES-2 presents chemical and physical analysis as well as calculated inventory summaries.

The results of the analyses have been compared to the dangerous waste codes in the *Washington Administrative Code "Dangerous Waste Regulations"* (WAC 173-303)¹. This assessment was conducted by comparing tank analyses against dangerous waste characteristics ("D" waste codes) and Washington State waste codes. Tank analyses were not checked against "U," "P," "F," or "K" waste codes because applying these codes is dependent on the source of the waste and not on particular constituent concentrations. The results indicate that the waste in this tank is adequately described in the *Dangerous Waste Permit Application for the Single-Shell Tank System*².

¹WAC 173-30, 1990, Dangerous Waste Regulations," *Washington Administrative Code*, as amended.

²DOE-RL, 1993, *Dangerous Waste Permit Application for the Single-Shell Tank System*, DOE/RL-88-21, Rev. 3, U.S. Department of Energy, Richland Operations Office, Richland, Washington.

Table ES-1. Tank BX-107 Status Overview.

Parameter	Status	Basis
Structure	Qualify for high-hazard design	Postulated based on pilot experiments
Integrity	Sound, no known leaks	Based on dry well indications; no suspicious level drops in process history (FIC)
Mitigation activities	Interim stabilized	< 50,000 gal interstitial liquid < 5,000 gal supernatant
	Partially interim isolated	Sealed to prevent inadvertent liquid addition
Safety classification	<u>Non</u> Watch List	Based on safety criteria, analytical results, and surveillance data
Tank capacity	2,006,050 L (530,000 gal)	By design
Residue level in tank	~ 3.07 m (120.7 ± 2 in. from bottom sidewalls, 132.9 in. from bottom center)	Actual surface level measurement (includes supernatant)
Sludge calculated volume	1,302,040 L (344,000 ± 1,000 gal)	Based on surface level data includes drainable interstitial liquid
Sludge calculated weight	≈ 1,880,388 kg	From surface level measurements and corresponding calculated density and volume
Drainable interstitial liquid volume	~ 109,765 L (~ 29,000 gal)	Estimated from liquid level data
Supernatant calculated volume	2,271 L (600 gal)	Based on surface level data
Tank vapor space pressure	Atmospheric	Passive ventilation
Sludge color	Mostly grey with scattered red streaks	Based on physical observations (1979 core, 1992 hot cell)
Sludge consistency	Soft putty	Based on physical observations and viscosity experiments

Table ES-2. Chemical, Radiochemical, and Physical Analysis Summary.

Group	Mol. Wt.	1992 Cores 40 and 41 Average Total Concentration Wet Basis	Tank inventory Based on 1992 core data
Metals		Weight%	Kg-Carbon scale
Na	23	10.2	191800
Al	27	1.43	26890
Si	28	0.68	12755
P	31	2.35	44152
S	32	0.44	8349
K	39	0.03	495
Ca	40	0.10	1956
Cr	52	0.097	1820
Fe	56	1.11	20872
Bi	209	2.23	41933
U	238	0.23	4370
Total metals inventory (excluding phosphorus and sulfur)*		16.22	304230
Radionuclides		$\mu\text{Ci/g}$	KCi
AT		0.0963	0.1811
Pu ^{239/240}		0.0572	0.1076
U ²³⁸		7.467E-4	0.0014
TB		46.7	87.81
Cs ¹³⁷		17.4	32.72
Sr ⁹⁰		9.87	18.56
Total radionuclides inventory*		46.80	~ 88

Table ES-2. Chemical, Radiochemical, and Physical Analysis Summary.

Group	Mol. Wt.	1992 Cores 40 and 41 Average Total Concentration Wet Basis	Tank inventory Based on 1992 core data
Anions		weight%	Kg
F-	19	0.92	17281
Cl-	35	0.11	2144
NO ₂	46	0.81	15269
NO ₃	62	13.7	257613
CO ₃ ⁻	60	0.58	10911
PO ₄ ⁻³ (calculated from total phosphorus)	95	7.26	136537
SO ₄ ⁻²	96	1.34	25197
Total anions inventory*		18.89	464409
Total metals and anions inventory		35.11	660190
Total organic carbon		0.07	1252
Physical properties			Units
pH (100 to 1 dilution of solids)		9.71	NA
Total solids (dry)		44.13	%
Water		~56	%
Undissolved solids		36.73	%
Temperature		66	°F
Bulk density		1.44	g/cm ³
Particle density (1979)		2.25	g/cm ³
Supernatant density		1.19	g/cm ³

Table ES-2. Chemical, Radiochemical, and Physical Analysis Summary.

Group	Mol. Wt.	1992 Cores 40 and 41 Average Total Concentration Wet Basis	Tank inventory Based on 1992 core data
Particle size distribution $\pm 20\%$			Number
0.5 - 2 μm		90	%
2 - 5 μm		9	%
5 - 150 μm		1	%

NOTE: This table includes analytes present in significant concentrations only. The total inventory values reflect total calculated from comprehensive data. Concentration on data and tank inventory values includes round-off error. Inventory data is based on bulk density and does not account for oxygen (metal oxides or hydroxides).

PREFACE

The goal of this report is to be customer and user friendly. The primary customer of Westinghouse Hanford Company, as the Operations and Maintenance contractor for the Hanford site, is the U.S. Department of Energy. In addition to the U.S. Department of Energy, data users include the program elements within Westinghouse Hanford Company that are tasked with the safe storage, retrieval, pretreatment, and final disposal of the tank waste. Other users of this document can range from State and Federal agencies such as the Washington State Department of Ecology and the U.S. Environmental Protection Agency, to private parties such as land owners and the Yakima Indian Sovereign Nation, to interested parties such as other U.S. Department of Energy sites and government contractors.

ACKNOWLEDGEMENTS

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LIST OF TERMS

AAS	Atomic absorption spectroscopy
AEA	Alpha energy analysis
AT	Total alpha reading
CRQLs	Contract Required Quantification Limits
CASS	Computer Automated Surveillance System
CVAA	Cold vapor technique
DOT	U.S. Department of Transportation
DSC	Differential scanning calorimetry
EPA	U.S. Environmental Protection Agency
EOX	Extractable halides
FIC	Food Instrument Corporation
GC/MS	Gas chromatography/mass spectroscopy
GEA	Gamma energy analysis
HAS	Hanford Analytical Services
HYAA	Hydride reduction method
IC	Ion chromatography
ICP	Inductively coupled plasma spectroscopy
IDL	Instrument detection limit
LANL	Los Alamos National Laboratory
LOW	Liquid observation wells
MDA	Method detectable activity
MDL	Method detection limit
MW	Metal waste
NA	Not applicable
NCPLX	Non-complexed
NPH	Normal paraffin hydrocarbon
NR	Not reported
PIC	Person in charge
PNL	Pacific Northwest Laboratory
SVOA	Semi-volatile organic analysis
TAP	Tank Advisory Panel
TB	Total beta
TBP	Tributyl phosphate
TCLP	Toxic characteristics leaching procedure
TCR	Tank characterization report
TGA	Thermal-gravimetric analysis
TIC	Total inorganic carbon
TOC	Total organic carbon
TOX	Total organic halides
TOX/EOX	Total/extractable organic halides
Tri-Party Agreement	<i>Hanford Federal Facility Agreement and Consent Order</i>

LIST OF TERMS (Continued)

TWRS
USQs
VOA

Tank Waste Remediation System
Unreviewed Safety Question
Volatile organic analysis

1.0 INTRODUCTION

Tank 241-BX-107, hereafter referred to as BX-107, is a 2,006,050 L (530,000 gal) tank that was built from 1946 to 1947 to support plutonium/uranium production and recovery activities at the Hanford Site. This report is designed to characterize the structure and contents of tank BX-107 based on a combination of process information, historical, and recent analytical data. In addition this report makes recommendations and conclusions concerning tank waste management issues.

1.1 PURPOSE

The objective of this report is to produce technical, user-friendly information that meets the Tank Waste Remediation Systems (TWRS) program and data quality requirements. This information, which includes physical, chemical, and radiological properties will be a major input into the decision process to appropriately retrieve and manage the material contained in tank BX-107 and the tank structure itself if necessary.

This document will primarily support the *Hanford Federal Facility Agreement and Consent Order* (Tri-Party Agreement) (Ecology et al. 1994) Milestone M-44-05. New information related to tank BX-107 will be incorporated into the BX-107 Tank Characterization Report (TCR) as it becomes available.

1.2 SCOPE

This report is an effort to concisely compile information on tank BX-107 and provide technical interpretation of the data. This effort includes reviewing all relevant tank records, including but not limited to process function and history, structural assessment, contents sampling, and analysis. Simulation model results, e.g., Track Radioactive Components (TRAC***), are compared against actual analytical values. Finally, specific safety issues and program element needs are addressed based on the results of the waste characterization.

***TRAC is a mathematical model developed to predict current tank content, based on historical process data.

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2.0 HISTORICAL TANK INFORMATION

This section will provide an overview of the Hanford Site radioactive-mixed waste SST farms and then focus on the history of tank BX-107 as it relates to its design specifications, process history, and contents.

2.1 TANK HISTORY

2.1.1 General Hanford Site Single-Shell Tank Farms History

Between 1943 and 1964, 149 SSTs, with capacities ranging from 208,175 - 3,785,000 L (55,000 to 1 M gal) were constructed to support the Hanford Site radioactive material production and recovery projects. In addition to the SSTs, 28 DSTs were constructed between 1966 and 1986, each with a capacity of 4,731,250 L (1.25 M gal).

The SSTs are located in 12 tank groups, called tank farms, which contain up to 18 tanks each, in the 200 East and 200 West Areas of the Hanford Site.

Additional generic information, related to process configuration, construction material, layout, structural integrity, and general use measures relative to the Hanford Site SSTs tanks are compiled in the Tank Characterization Reference Guide (WHC-SD-WM-TI-648).

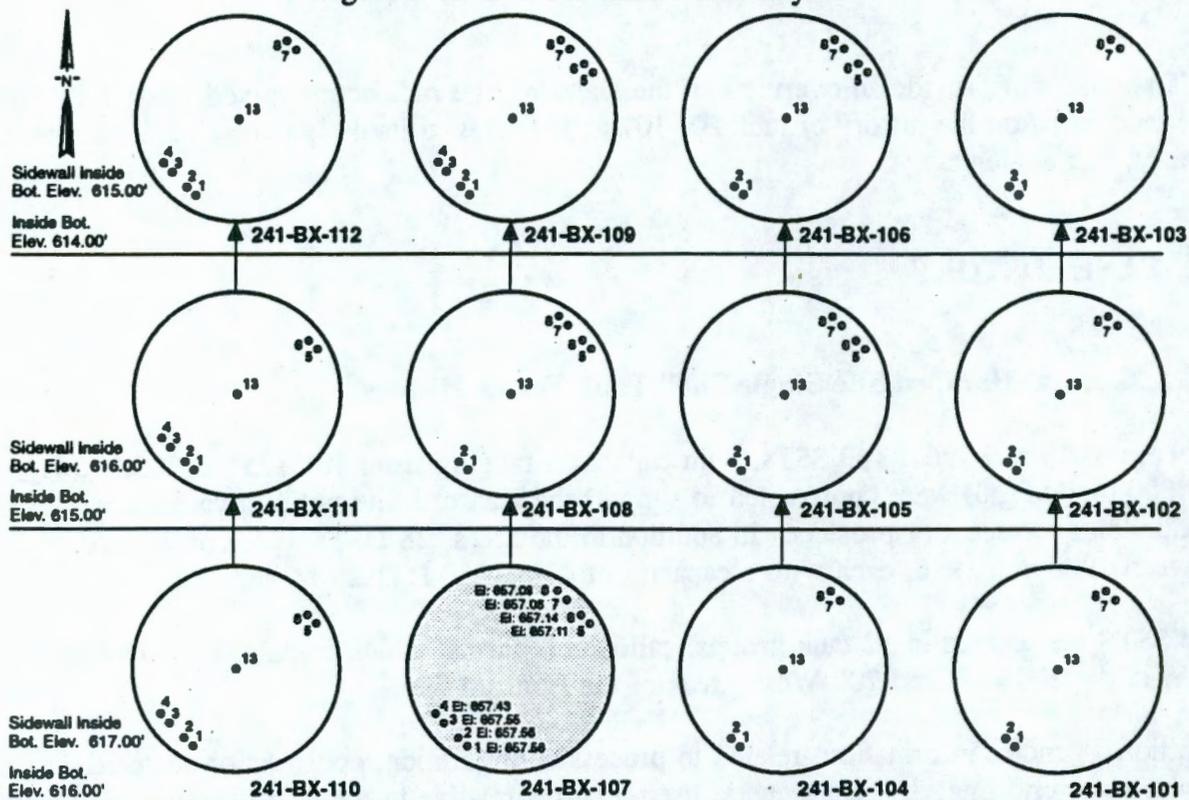
2.1.2 241-BX Tank Farm Surveillance

The BX Tank Farm is located in the 200 East Area of the Hanford Site. It consists of twelve (530,000 gal) (2,006,050 L) tanks as shown in Figure 2-1.

The BX Tank Farm is regularly monitored for possible leaks using liquid level indicators, drywells, and liquid observation wells. Figure 2-2 shows a typical vertical section layout configuration. The liquid level in each tank is scanned using automatic liquid level gauges that feed into a Computer Automated Surveillance System (CASS). Manual tapes are used as a backup. External vertical dry wells are monitored using scintillation probes. Neutron and gamma probes are used alternately on a quarterly basis to monitor liquid observation wells (LOW). Temperature, using thermocouple trees, and headspace pressure, are continuously monitored. Tank dome elevation surveys are also taken to measure any indication of a tank dome collapse (Weltey 1989).

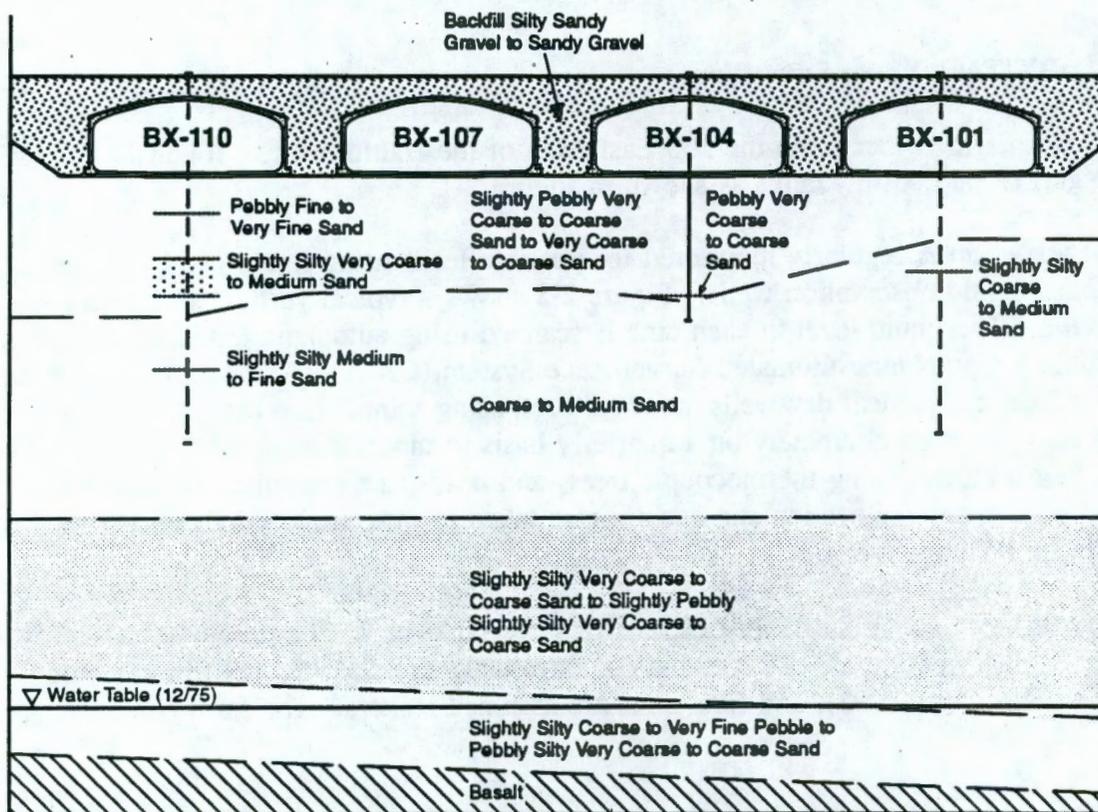
The BX Tank Farm consists of four cascades of three tanks each. Its primary function was to support B Plant operations. Two of the BX farm tanks are classified as ferrocyanide Watch List tanks (June 1993), and five tanks are assumed leakers. The farm contains

Figure 2-1. 241-BX Tank Farm Layout.



29-02002.2

Figure 2-2. 241-BX Tank Farm Vertical Section Layout.



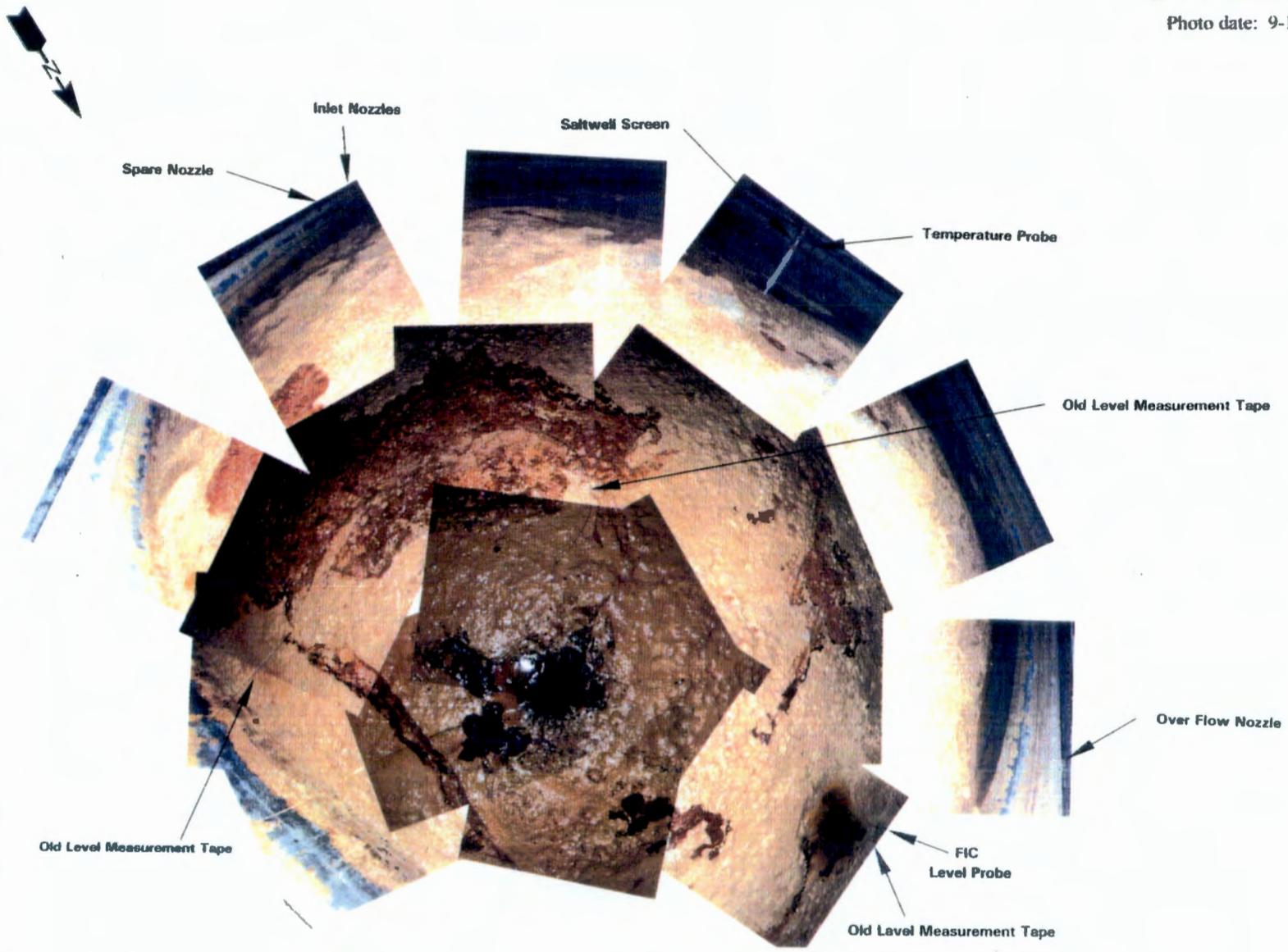
29402002.3

241-BX-107

Photo date: 9-11-90

WHC-EP-0739

Color Figure 2-1. Interior Photograph of Tank 241-BX-107.



CF 2-1

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approximately 5,124,890 L (1,354 Kgal) of sludge, 586,675 L (155 Kgal) of salt cake, plus 189,250 L (50 Kgal) of supernatant liquids, thus adding up to a total volume of 5,900,815 L (1,559 Kgal) (Hanlon 1993).

2.1.3 BX-107 Tank Specifications

The configuration of tank BX-107 is similar to the first generation SSTs. Figure 2-3 is a two-dimensional sketch of tank BX-107. Tank BX-107 is a 22.9-m dia. (75-ft dia.), 4.88-m (16-ft) elevation tank with a .3-m (12-in.) deep dished bottom. Although 8 vertical risers and one centrally located vertical salt well pit are shown, tank BX-107 has a total of 12 risers. Figure 2-4 depicts a generic SST instrumentation configuration. Tank BX-107 has a 2,006,050 L (530,000 gal) capacity. It consists of an ASTM A283 grade C carbon steel liner constructed within a reinforced concrete tank and dome.

2.2 PROCESS KNOWLEDGE

Tank BX-107 received primarily B Plant first-cycle waste and cell 23 concentrated waste (Prosk 1986). Because of the waste management operations between 1952 and 1980, process effluents from REDOX, PUREX, and other plants may have found their way into tank BX-107.

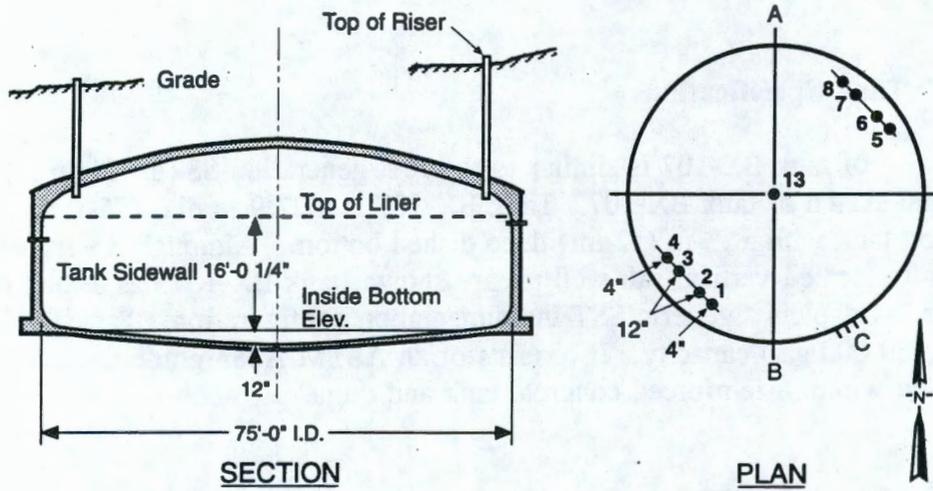
In September 1948, the tank began filling with byproduct cake solution from the first decontamination cycle residues generated in the canyon building, which supported the BiPO_4 process (waste type 1C). The tank's contents recorded volume during the third quarter of 1952 was 2,006,050 L (530,000 gal).

By the end of 1952 when all released supernatant liquid was pumped out of tank BX-107, there were 1,654,045 L (437,000 gal) of settled sludge left in the tank. During the second quarter of 1953, 352,050 L (93,000 gal) of tributyl phosphate (TBP) waste were added to tank BX-107.

Between 1953 and 1968, the volume of the sludge remained between 1,619,895 L to 1,619,980 L (428 to 447 Kgal) while liquid volumes fluctuated between 34,065 L to 492,050 L (9 to 130 Kgal).

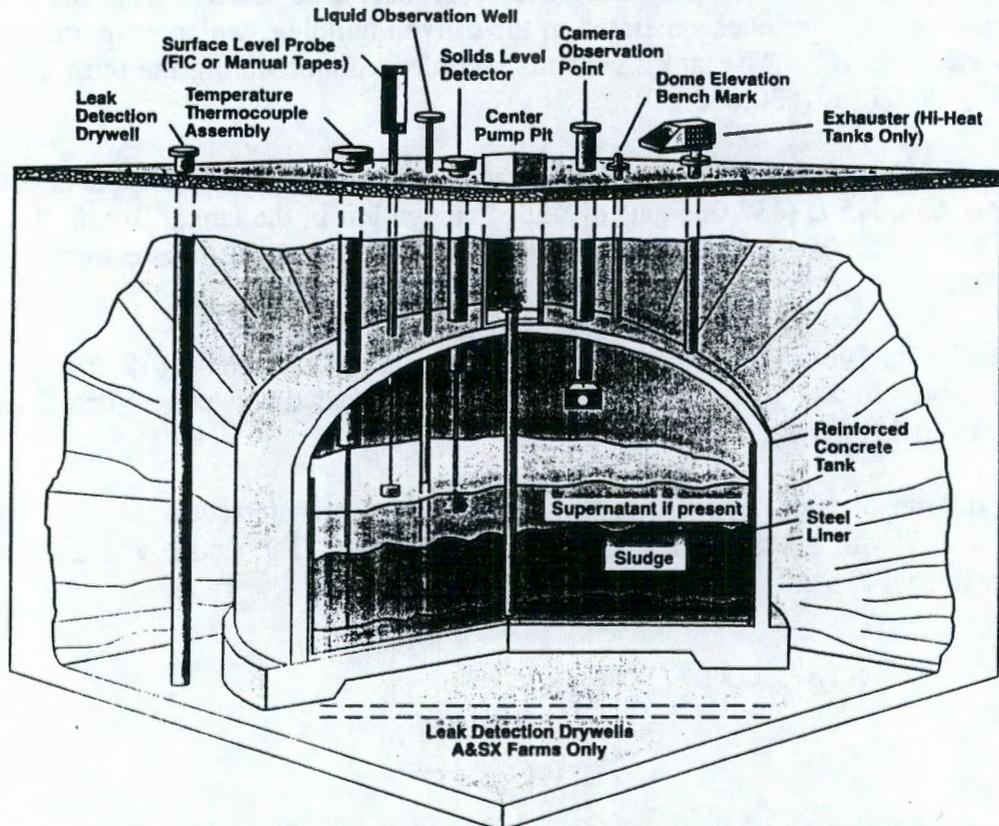
In the last quarter of 1968, 598,030 L (158,000 gal) were transferred to tank BX-106, which left tank BX-107 with 1,548,065 L (409,000 gal) of sludge. The sludge was a mixture of 1C and TBP residues.

Figure 2-3. Tank 241-BX-107 Configuration.
75 Ft. Tanks



Tank 241-BX-107								
Riser Number and Elevation								
1	2	3	4	5	6	7	8	13
657.54	657.57	657.57	657.39	657.11	657.13	657.06	657.09	

Figure 2-4. Generic Single-Shell Tank Instrument Configuration.



In the third quarter of 1969, tank BX-107 began receiving 1X type waste*. Records show that the sludge volume decreased to 1,423,160 L (376,000 gal) and the liquid volume increased to 624,525 L (165,000 gal).

Those levels remained approximately constant until 1974 when 249,810 L (66,000 gal) of tank BX-107 supernatant liquid were transferred to tank BX-106 for solidification. The remaining 374,715 L (99,000 gal) of liquid were used as evaporator feed in 1977, leaving the tank with 1,423,160 L (376,000 gal) of residual sludge from 1977 to 1980. In 1977, the tank was decommissioned (Anderson 1990).

Figure 2-5 shows the level history for tank BX-107 between 1948 and 1993. A detailed model that depicts process history by waste type was developed by Los Alamos National Laboratory (LANL) (Agnew 1994).

2.3 TANK CURRENT STATUS

2.3.1 Structure

The tank BX-107 structure is qualified for the current high-hazard design spectrum of 0.2 times gravity (Becker 1990). No corrosion counter measures, such as cathodic protection or the addition of chemical inhibitors, are currently being applied to the tank's steel shell. Tank BX-107 is classified as sound; i.e., surveillance data indicate no loss of liquid attributed to a breach of tank integrity (Hanlon 1993). Dome loading and deflection are reported to be within the structural limitation specifications.

2.3.2 Contents Overview

2.3.2.1 Physical Description. The contents of tank BX-107 consist of a water-saturated sludge layer. The most recent tank photographs were taken in September 1990 (Hanlon 1990). Color Figure 2-1 is a top view photo collage. The liquid filled well in the center of that figure was left by the salt well pump, which was used for interim stabilization. In general, Color Figure 2-1 shows a relatively heterogeneous top layer with a mostly grey texture (note the brownish color in the center photos is probably due to lighting conditions) with red and bluish streaks. The blue-colored markings are obvious on the internal side of the tank's steel shell and could be attributed to ferrous phosphate and/or sulfate crystals

*Generated from the cesium recovery process at B Plant, 1X waste (largely liquid, with very little suspended solids) included ion exchange column waste, column wash waste, and cesium purification waste.

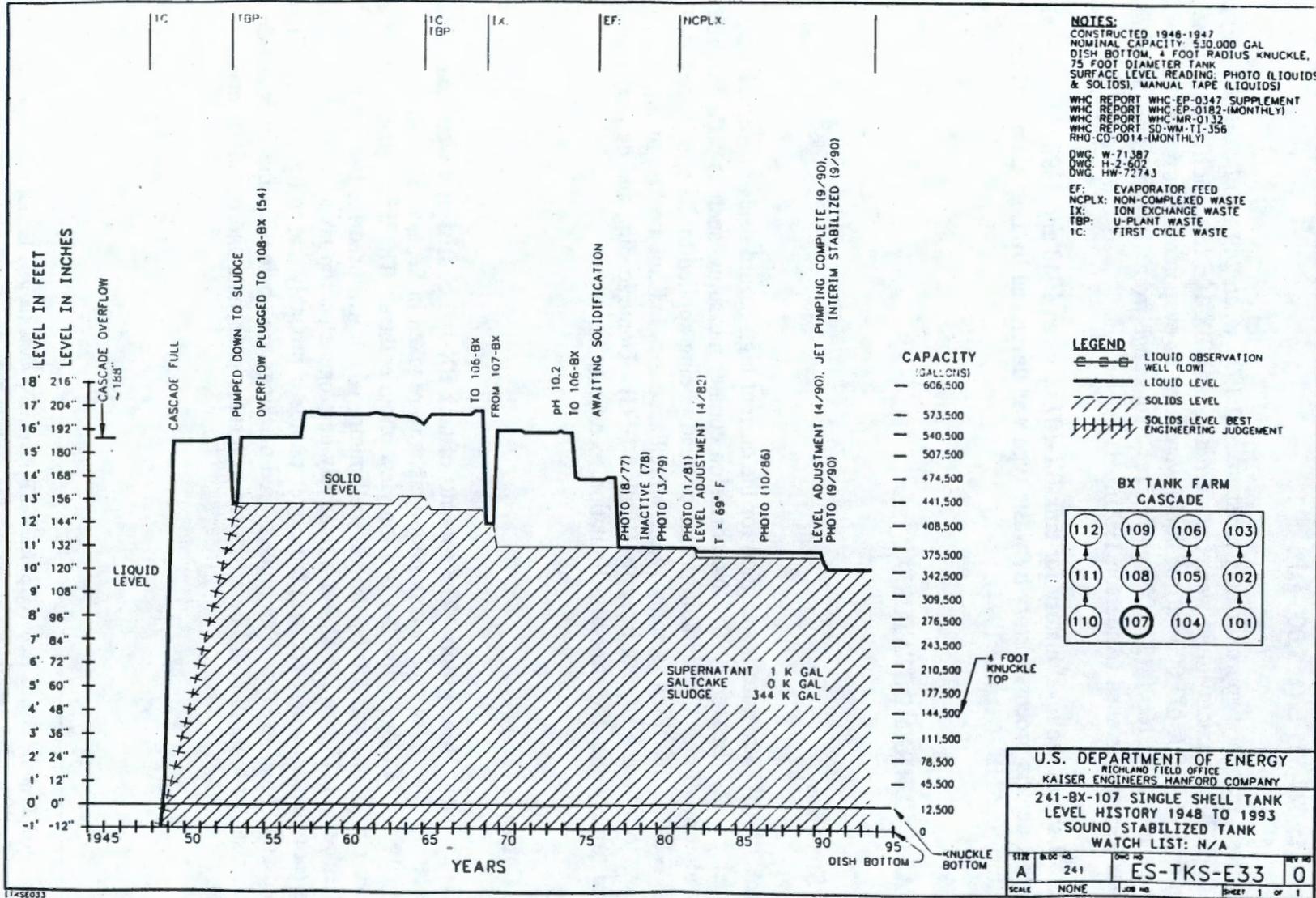


Figure 2-5. Tank 241-BX-107 Waste Level History.

[$\text{Fe}_3(\text{PO}_4)_2$ (insoluble), $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (soluble)]. The reddish spots could be attributed to ferric oxide/hydroxide compounds (such as $\text{FeO}(\text{OH})$ insoluble crystals). Other ferric and ferrous compounds such as phosphates ($\text{Fe}(\text{H}_2\text{PO}_2)_3$, $\text{Fe}_4(\text{P}_2\text{O}_7)_3 \cdot \text{H}_2\text{O}$, FePO_4 , $\text{Fe}(\text{PO}_4)_2$), nitrates ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), and sulfates ($\text{Fe}_2(\text{SO}_4)_3$) are grayish in color and can easily blend into the dominant grey color of tank BX-107 waste making identification of potential species by visual observation infeasible. Stabilization records indicate that the surface is a light brown, sludgy fine material with a pool of liquid located north of riser #4 (Boyles 1990).

2.3.2.2 Waste Volume. The material in tank BX-107 was classified as non-complexed (NCPLX); or largely inorganic wastes containing low levels of organic complexants. It consists of approximately 1,302,040 L (344,000 gal) of sludge, of which 109,765 L (29,000 gal) are interstitial liquid, and 3,785 L (~1,000 gal) supernatant. Those figures were valid as of September 1990 (Hanlon 1993) and were calculated based on actual in-tank level measurements (stabilization records indicate 3.07 ± 0.05 m or 120.7 in. (± 2 in.) as of September 1990) and actual tank geometry.

2.3.3 Contents Remediation Activities

Tank BX-107 is not classified as a Watch List tank, nor does it have Unreviewed Safety Questions (USQs) associated with it. It was interim stabilized in September 1990 by jet pumping supernatant liquid and pumpable interstitial liquid (see Appendix A).

Tank BX-107 is also partially interim isolated in 1990, as the tank piping configuration was appropriately modified to prevent inadvertent addition of liquids.

While tank BX-107 does not have an operational exhauster, it is passively ventilated. It is fitted with a Food Instrument Corporation (FIC) liquid-level gauge relayed to a CASS, a manual temperature reading well, one screen/jet pump system, and two radiation monitoring dry wells on one side of its perimeter (drywell 21-07-05 is no longer active).

2.3.4 Contents Surveillance Activities

No headspace pressure data were found on tank BX-107. However, temperature data were found for the period between 1975 and 1980. The last temperature reading was reported in July 1994 and averaged about 66 °F. The thermocouple tree, containing 14 thermocouple elements, was previously declared out of service in April 1983.

Recent radiation (gamma and neutron) activity readings for tank BX-107 were not found. No liquid level reading has been taken since April 1993 (120.7 in. ± 2 in.). The FIC liquid-level gauge was repaired on May 10, 1994 (per work package No. 2E-94-00273). The thermocouple repair work package (No. 2E-93-01348) has been executed (Gutierrez 1994). Table 2-1 shows the latest temperature data from operating thermocouples.

Table 2-1. Temperature Data - July 21, 1994.

Thermocouple	1	2	3	4	5	6	7	11	Average
Temperature °F	67.82	68.54	64.76	64.58	64.76	65.48	66.92	67.46	66.12

2.4 HISTORICAL ESTIMATION OF THE TANK'S CONTENTS

2.4.1 Estimate - Based on Process Knowledge

2.4.1.1 Nature of the Material That Entered the Tank. As outlined in Section 2.1.3.2, 1C, TBP, and 1X residues were primarily transferred in and out of tank BX-107. Estimated compositions for the above-mentioned residues are summarized in Tables 2-2 and 2-3.

The 1C, or byproduct cake solution, and first cycle decontamination residue was generated from the BiPO₄ process in the T and B Plants (Anderson 1990). This waste was often combined with alkaline coating removal and/or stack drainage residues. Water content in 1C waste was reported to be around 83%.

TBP residue resulted from the uranium scavenging activities at the 241-CR Facility, which is also known as the Uranium Recovery Plant. It was systematically combined on a 1 to 1 ratio with metal waste (MW) from the BiPO₄ process. The pH of the final residue was over 9.5. 1X, as mentioned in Section 2.1.3.2 of this report, and included ion-exchange column residues and wash solutions, as well as cesium purification residues generated during the B Plant recovery process.

2.4.1.2 Estimated Residual Amounts in Tank. The records for the many sluicing, slurry, and waste transfer operations in tank BX-107 during its service life are not sufficiently accurate to reconstruct a detailed material balance.

2.4.1.3 Character of Tank Residues. Tank BX-107 residue is expected to contain the elements and compounds listed in Table 2-3. Sodium (Na⁺), nitrate (NO₃⁻), and nitrite (NO₂⁻) are expected to have the highest concentrations, followed by carbonates (CO₃⁻²), sulfates (SO₄⁻²), phosphates (PO₄⁻³), ammonium (NH₄⁺), silica (Si), aluminum (Al), fluoride (F⁻), and hydroxide (OH⁻).

It is expected that other fission products (e.g., Sr^{89/90}, I¹²⁹, and C¹⁴) and organics will be found in tank BX-107 waste.

Table 2-2. 1C and TBP Waste Composition Estimates (Anderson 1990).

Chemical compound	Approximate concentration moles/L (wet basis)	Residue type
CePO ₄	<0.01	1C
Zn ₃ (PO ₄) ₂	<0.01	1C
NaNO ₃	0.85	1C
Fe ₂ (SO ₄) ₃	0.07	1C
Na ₃ PO ₄	0.75	1C
Cs(NO ₃)	0.01	1C
(NH ₄) ₂ SO ₄	0.04	1C
(NH ₄) ₂ (SiF ₆)	0.07	1C
NH ₄ NO ₃	0.06	1C
Pu	0.4% weight, 1% weight	TBP, 1C
U	0.4%	TBP
P	1.29	TBP
UNH	0.0026	TBP
SO ₄ ⁻²	0.346	TBP
PO ₄ ⁻³	0.25	TBP
NO ₃ ⁻	6.14	TBP
Cl ⁻	0.022	TBP
Na ⁺	7.57	TBP
OH ⁻	0.094	TBP
K ₄ Fe[CN] ₆	Not reported	TBP
Cs	Not reported	TBP

NOTE: No unit conversion was performed to preserve data authenticity. Subsequent sections, however, contain unit converted data for comparison purposes.

Table 2-3. 1C, TBP, and 1X Waste Species Concentration
Estimate-Wet Basis.

Chemical compound	Approximate concentration moles/L	Residue type
Al	0.38	1C
Bi	0.012	1C
Ce	0.0002	1C
Cl ⁻	0.0025	TBP
CO ₃ ⁻²	0.65	1X
Cr	0.003	1C
F ⁻	0.19	1C
Fe	0.025, 0.03	1C, TBP
NH ₄ ⁺	0.11	1C
Na ⁺	3.34, 8.87, 3.9	1C, TBP, 1X
NO ₂ ⁻	0.28, 1.9	1C, 1X
NO ₃ ⁻	1.54, 7.35, 0.49	1C, TBP, 1X
OH ⁻	0.28, 0.09	1C, TBP
PO ₄ ⁻³	0.28, 0.3	1C, TBP
Pu	0.000002, 0.00000067	1C, TBP
Si	0.034	1C
SiF ₆	0.031	1C
SO ₄ ⁻²	0.052, 0.31, 0.085	1C, TBP, 1X
U	0.0061	TBP
Zr	0.0003	1C

No unit conversion was performed to preserve data authenticity.
Subsequent sections contain unit converted data for comparison purposes.

NOTE: Figure 2-6 is a reflection of this data.

Source: Anderson (1990).

Waste Species Concentration Estimates (Wet Basis)

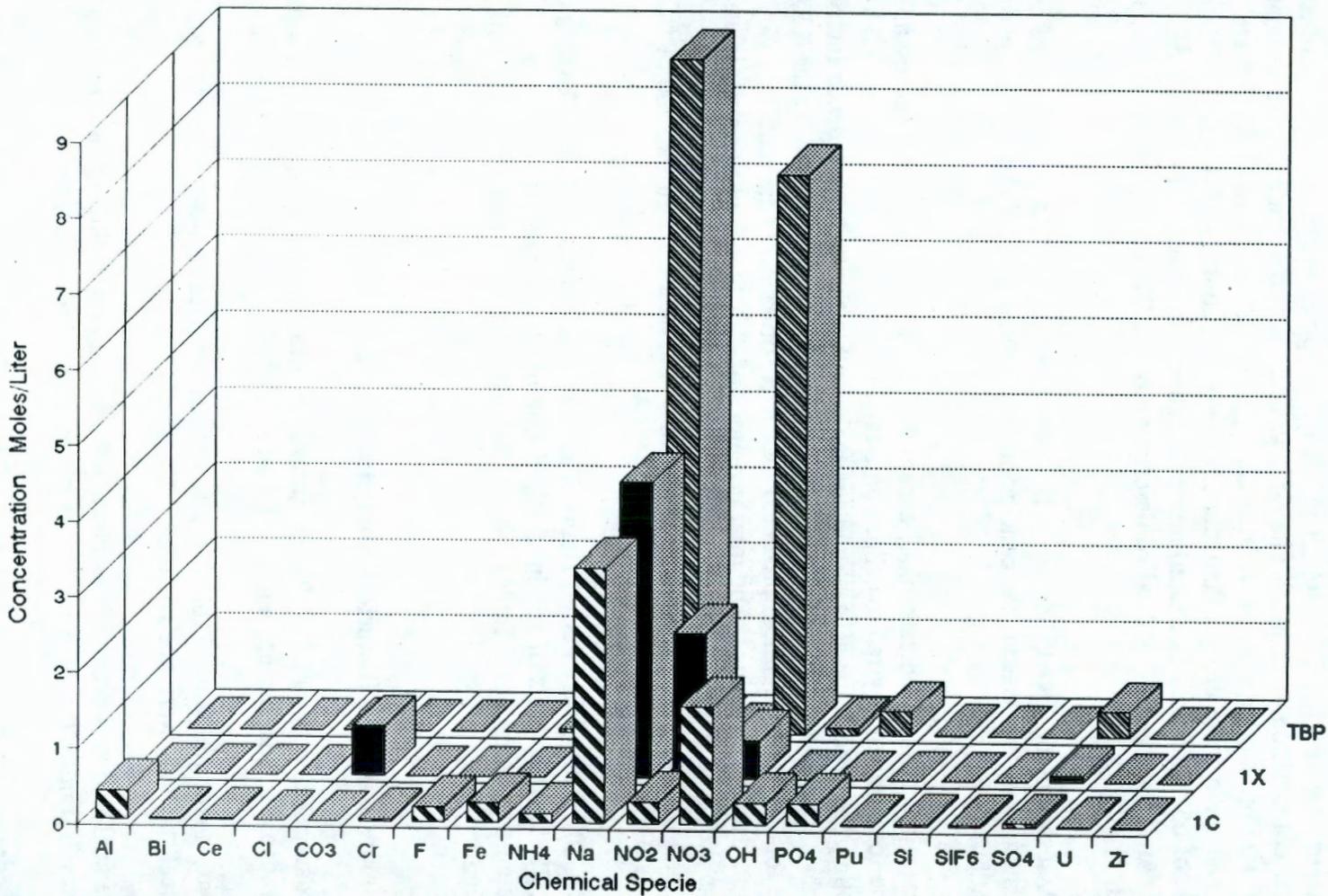


Figure 2-6. Waste Species Concentration Estimates (Wet Basis).

2.4.2 Estimate - Based on TRAC Model

2.4.2.1 Overview of TRAC Model. The TRAC computer program was developed to estimate the composition of Hanford Site tank wastes. Waste inventories are estimated based on nuclear fuel production models, reprocessing and waste management flowsheets, tank transfers, and radioactive decay calculations. The output consists of the approximate inventories of 65 radionuclides and 30 non-radioactive chemical constituents in each of the 149 SSTs (Anderson 1990). A detailed description of the TRAC model can be found in Jungfleisch (1984).

2.4.2.2 Assessment of TRAC Model. The codes used in the TRAC model have not yet been validated. For this reason, the conclusions and model outputs should be regarded as approximations (Anderson 1990).

A preliminary assessment indicates that, except for C^{14} , the TRAC model successfully predicts the locations of key radionuclides on a tank-by-tank basis. Moreover, TRAC agreed with sampling predictions for at least half the tanks within the 95% confidence interval. Although the locations of key radionuclides are reasonably well predicted by the TRAC model, the total inventory (curies) predicted by TRAC of each key radionuclide varies from that found to date by sampling. The total inventory of Tc^{99} found by sampling is essentially the same as predicted by the TRAC model. However, TRAC under-estimates the inventory of $Pu^{239/240}$ and over-estimates the amounts of Am^{241} , I^{129} , and C^{14} . In the case of ^{14}C , the factor is 1,000 times (Jensen et al. 1986).

2.4.2.3 BX-107 TRAC Calculated Data. The latest data available from TRAC is dated March 12, 1985. It consists of an alphabetic listing of several analytes and their corresponding concentrations. Tables 2-4 and 2-5 list a selective excerpt of the tank BX-107 reported data.*

2.4.3 Estimate Based on Historical Analytical Results

Historical data relative to tank BX-107 is scarce. Records available show two sets of analysis available for the period before 1980 when all SSTs were decommissioned.

2.4.3.1 Data Prior to 1980. Table 2-6 summarizes the data in Walser (1974). Sampling and analytical methods were not specified.

The sample analyzed was described as clear, yellow, with no visible solids. Radiation emission was 300 mRad/hr. No exotherms below 200 °C were reported.

In 1979, a core was sampled and analyzed. Tables 2-7 and 2-8 summarize the data reported. No exotherms were reported in any of the samples. Composite bulk density and percent

*Unit conversion of this data will be performed in subsequent sections.

Table 2-4. Select Radiochemical Analytes as Calculated by TRAC.

Component	Inventory (moles)	Activity (curies)
Am ²⁴¹	4E-3	3
Ba ¹³⁷	2E-7	2E+4
Pu ²³⁹	3	5E+1
Pu ²⁴⁰	8E-2	5
Cs ¹³⁷	1	2E+4
Sr ⁹⁰	4	5E+4
Tc ⁹⁹	3	5
U ²³⁵	7E+1	3E-2
U ²³⁸	1E+4	8E-1
Y ⁹⁰	1E-3	6E+4
Zr ⁹³	1E+1	2

Table 2-5. Select Inorganic Analytes as Calculated by TRAC.

Analytes	Inventory (moles)
Al	1E+5
Ba	3
Bi	7E+4
CO ₃ ⁻	8E+5
Cr	1E+4
F	1E+5
Fe	2E+5
NO ₂ ⁻	8E+4
NO ₃ ⁻	1E+6
Na ⁺	4E+6
OH ⁻	6E+5
PO ₄ ⁻³	3E+5
SiO ₃ ⁻²	2E+4
SO ₄ ⁻²	2E+5
ZrO	2E+4

Table 2-6. Summary of 1974 Data for Supernatant Sample T-5921.

Analyte	Concentration
Ionic compounds	Moles/L
OH ⁻	0.723
Al ⁺³	1.53E-3
Na ⁺	5.29
NO ₂ ⁻	1.82
NO ₃ ⁻	1.51
PO ₄ ⁻³	6.15E-2
F ⁻	2.16E-2
CO ₃ ⁻²	0.674
Physical properties	
Specific gravity	1.238
Water	75.28%
pH	10.18
Radionuclides	μCi/gal
Cs ¹³⁴	7.38E+2
Cs ¹³⁷	3.4E+5

Table 2-7. 1979 Core Chemical Data (Wet Basis).

Analyte	Composite H ₂ O soluble Weight %	Composite H ₂ O insoluble Weight %
Metallic elements		
Al	NR	1.64
Bi	< 3.4E-2	1.61
Cd	NR	1.5E-3
Cr	6.87E-3	NR
Fe	NR	1.43
Hg	NR	1.31E-1 ^a
La	NR	4.38E-2
Mn	NR	5.27E-3
Na	6.48	1.1E+1
Ni	< 1.6E-3	5.04E-1 ^b
Pb	< 2E-3	NR
Si	1.8E-2	0.127
Ionic species		
CO ₃ ⁻²	0.1	NR
F ⁻	8.7E-2	0.1
NO ₃ ⁻	14	1.3
OH ⁻	< 5E-2	NR
PO ₄ ⁻³	1.5	7.2
SO ₄ ⁻²	0.81	0.12
SiO ₄	0.02	0.03
Organics		
TOC	NR	7.3E-4

^aPossibly attributed to reporting error.

^bPossibly attributed to KOH fusion in nickel-impregnated crucibles. See discussion in Section 7.0.

Table 2-8. 1979 Core Radiochemical Data (Wet Basis).

Radionuclides	Segment 1	Segment 2	Segment 3	Composite (water soluble)	Composite (water insoluble)	Units
Am ²⁴¹	NR	NR	NR	NR	2.7E-9	g/g
Cs ¹³⁴	8.8	3.3	5.2	NR	NR	μCi/kg
Cs ¹³⁷	2.01E+5	8.21E+4	2.74E+3	2.74E+3	8.37E+3	μCi/kg
Eu ¹⁵⁴	24	12	21	NR	NR	μCi/kg
Eu ¹⁵⁵	1.35E+3	68	1.01E+3	NR	NR	μCi/kg
Pu	NR	NR	NR	8.51E-11	1.75E-6	g/g
Sr ^{89/90}	NR	NR	NR	2.3E-3	14.2	μCi/g
U	NR	NR	NR	5.5E-7	7.99E-5	g/g
Ce ¹⁴⁴	NR	NR	NR	<2.54E-2	8.37	μCi/g

water were reported at 1.46 g/cm³ and 54% respectively. Endotherms, however, were exhibited in all three segments at 105 °C and 290 °C approximately. Reported physical properties are compiled in Table 2-9.

While sampling and analytical methods were not discussed, it was reported that the three-segment core yielded a total of 127 cm (50 in.). The solids were 45% water soluble, yellowish in color with red streaking throughout, and had the consistency of soft putty. Core segment No. 3 had various size chunks of a hard material ranging from pea to marble size (Horton 1979).

Table 2-9. 1979 Core Physical Data.

Physical properties	Segment 1	Segment 2	Segment 3	Units
Water content	53.12	55.05	52.83	Weight %
Bulk density	1.422	1.424	1.541	g/cm ³
Particle density	1.834	2.19	2.726	g/cm ³

An attempt was made to estimate the data values for the missing core segments. Results can be found in Hill (1979).

2.4.3.2 Data Between 1980 and 1992. Analysis results of a supernatant liquid sample from tank BX-107 were reported in Weiss (1990). The results are provided in Table 2-10. However, Weiss (1990) states that the total alpha reading (AT) values may have been influenced by the significant quantities of cesium present in the sample. Weiss (1990) also adds that the reported hydroxide (OH⁻) concentration was biased by the neutralization of the second phosphate charge present in the sample. Again, sampling or analytical methods were not discussed.

2.4.3.3 Comparison of Supernatant Analytical Data. As outlined in the previous section, supernatant data dates back to 1974 and 1990. Analytes reported in considerable concentrations are summarized in Tables 2-11, 2-12, and 2-13. Cesium-134/137 concentrations, reported in $\mu\text{Ci/gal}$ in 1974, were converted to $\mu\text{Ci/L}$.

Total organic carbon (TOC) was reported at 2.7 g/L in 1990. Hereafter, comments relative to the data in Tables 2-11, 2-12, and 2-13 will be limited to observations.

- Higher amounts of soluble solids were present in the 1990 supernatant sample, possibly because of (1) the formation of additional soluble salts and soluble metal oxides; (2) the change in thermodynamic equilibrium state; or (3) the analytical variance (based on the sampling location and analytical procedure).
- pH decreased to 9.64 in 1990, indicating a loss in soluble [OH⁻] ions, possibly because of the conversion of some hydroxides to oxides (or salt + water), formation (or absorption) of CO₂, as well as other thermodynamic factors resulting in lower solubility of hydroxide species.
- Lower soluble Na⁺ concentration in 1990 could be attributed to (1) analytical variance; (2) a decrease in solubility because of alteration in the thermodynamic equilibrium; or (3) the incorporation of Na⁺ ions into a less soluble matrix.
- Lower soluble NO₂⁻ and higher soluble NO₃⁻ concentrations were possibly because of the oxidation of nitrite to nitrate.
- Increase in PO₄⁻³ concentration in 1990 was possibly because of analytical variance, or a change in the medium's thermodynamic conditions, for example, the formation of additional soluble phosphate compounds (by ion-exchange, substitution, and other mechanisms such as the ones supporting Le Chatelier's principle).

Table 2-10. 1990 Supernatant Liquid Profile: Sample Number R6038.

Analyte	Concentration	
	Moles/L	Weight percent
Metallic elements		
Ba	6.53E-5	0
Cr	6.59E-3	0.03
Fe	7.84E-5	0
Hg	0	0
K	1.23E-2	0.04
Mo	6.13E-4	0
Na	4.22	7.94
P	1.7E-1	0.43
Pb	1.64E-4	0
Si	9.29E-3	0.02
U grams/L	8.58E-1	0.07
Anions	Moles/L	
Cl ⁻	6.09E-2	0.18
F ⁻	<7.47E-2	
NO ₂ ⁻	7.43E-1	2.8
NO ₃ ⁻	2.64	13.41
PO ₄ ⁻	1.62E-1	NR
SO ₄ ⁻	1.81E-1	1.42
OH ⁻	1.09E-1	NR
CO ₃ ⁻	2.74E-1	1.35
Radionuclides	μCi/L	
Alpha total	2.9	NR
Total beta	2.88E4	NR
Cs ¹³⁷	2.49E4	NR
Si ^{89/90}	5.71	NR
Pu ^{239/240}	4.24	NR
Am ²⁴¹	2.49E-1	NR
Organics	grams/L	
TOC	2.7	NR
Physical properties		
Water (weight %)	72	
pH	9.64	

Table 2-11. Supernatant Physical Properties Comparison (1974/1990).

Sample	Water weight %	pH	Specific gravity
T-5921, 1974	75.28	10.18	1.238
R6038, 1990	72	9.64	NR

Table 2-12. Supernatant Inorganic Analytical Results Comparison in Moles/L (1974/1990).

Inorganic analytes	T-5921 1974	R-6038 1990
Al	1.53E-3	NR
Na	5.29	4.22
Ba	NR	6.53E-5
Cr	NR	6.59E-3
Fe	NR	7.84E-5
K	NR	1.23E-2
Si	NR	9.29E-3
OH ⁻	0.723	1.09E-1
NO ₂	1.82	7.43E-1
NO ₃ ⁻	1.51	2.64
PO ₄ ⁻³	6.15E-2	1.62E-1
F ⁻	2.16E-2	<7.47E-2
Cl ⁻	NR	6.09E-2
CO ₃ ⁻²	0.674	2.74E-1
SO ₄ ⁻²	NR	0.181

NOTE: Figure 2-7 reflects this data.

Table 2-13. Supernatant Radionuclides Analytical Results Comparison in $\mu\text{Ci/L}$ (1974/1990).

Radionuclides	T-5921/1974	R-6038/1990
Alpha total	NR	2.9
Total beta	NR	2.88E+4
Cs ¹³⁷	8.95E+4	2.49E+4
Cs ¹³⁴	1.94E2	NR
Sr ^{89/90}	NR	5.71
Am ²⁴¹	NR	2.49E-1
Pu ^{239/240}	NR	4.24
U ²³⁸	NR	0.606 g/L

- Lower soluble CO_3^{2-} concentration in 1990 was possibly because of the reduction of carbonate to carbon dioxide, or the incorporation of carbonates into a less soluble matrix.
- Lower cesium concentration in 1990 can be attributed to cesium decay to barium or possible analytical variance.

All analytes reported correspond to water soluble compounds present in the supernatant liquid. Unfortunately, the data available is not sufficient to establish the chemistry that took place in tank BX-107 residues between 1974 and 1990. Figure 2-7 depicts the tank BX-107 1974 and 1990 supernatant analytical results for select inorganic analytes.

2.4.3.4 Comparison of Sludge Analytical Data. As mentioned previously in this section, a core sample was obtained from tank BX-107 in 1979. Three segments and two core composites were evaluated. Tables 2-14, 2-15, 2-16, and 2-17 are general summaries of the data highlighting major properties and components found. Plutonium and uranium results were converted to $\mu\text{Ci/g}$ using their respective natural activities values.

TOC was reported at 7.3E-4% by weight in the water-insoluble portion of the core sample. Again, the following observations can be made.

- Material bulk density is between 1.42 and 1.54 g/cm^3 .

Supernatant Inorganic Analysis
Results Comparison

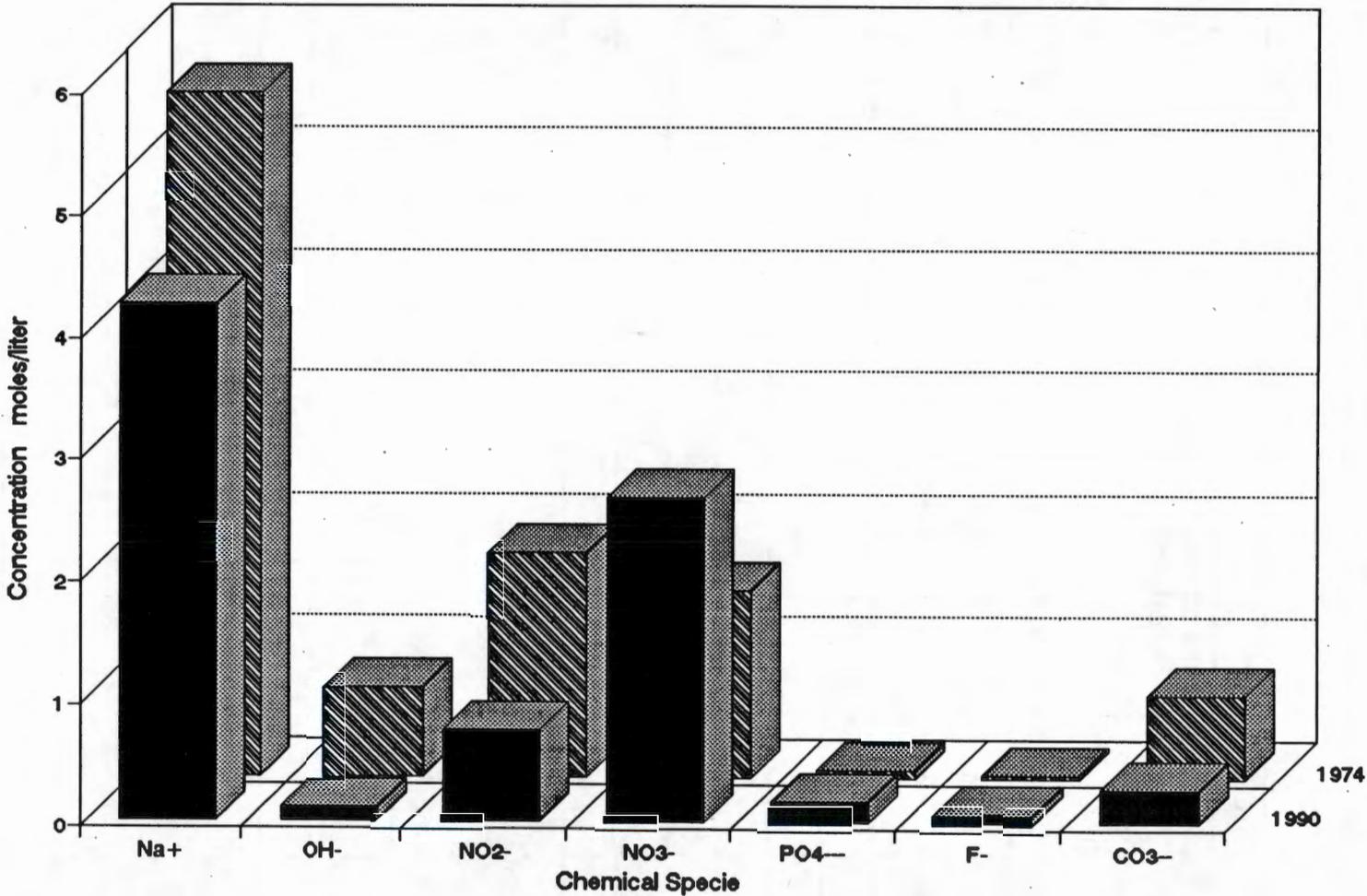


Figure 2-7. Supernatant Inorganic Analysis Results Comparison.

Table 2-14. 1979 Core Physical Properties Range.

Water content	52 - 55 %
Bulk density	1.42 - 1.54 g/cm ³
Particle density*	1.83 - 2.73 g/cm ³

*No discussion was found regarding this quantity, but it is assumed at this point that it refers to the density of mechanically packed particles, which, when divided by the density of water equals specific gravity.

Table 2-15. Major Metallic Elements Reported for the 1979 Core Sample in Weight %.

Analyte	Water soluble	Water insoluble	Ratio w.insoluble/w. soluble
Al	NR	1.64	N/A
Bi	<3.4E-2	1.61	47.35
Fe	NR	1.43	N/A
Hg	NR	0.131 ^a	N/A
Na	6.48	11	1.7
Ni	<1.6E-3	0.504 ^b	315
Si	1.8E-2	0.127	7

^aPossibly due to unit reporting error.

^bPossibly attributed to method contamination (nickel crucibles).

Table 2-16. Major Anions Reported for the 1979 Core Sample in Weight%.

Analyte	Water soluble	Water insoluble	Ratio w.insoluble/w. soluble
CO ₃ ⁻²	0.1	NR	N/A
F ⁻	8.7E-2	0.1	1.15
NO ₃ ⁻	14	1.3	0.09
OH ⁻	< 5E-2	NR	N/A
PO ₄ ⁻³	1.5	7.2	4.8
SO ₄ ⁻²	0.81	0.12	0.15

NOTE: NO₃⁻ and SO₄⁻² results clearly indicate that a serial leaching process was performed: i.e., the sample was probably (1) washed with water; (2) centrifuged; (3) solid-residue treated with acid; (4) centrifuged; (5) solids dissolved/fused in KOH/HCl.

Table 2-17. Major Radionuclides Reported for the 1979 Core Sample in $\mu\text{Ci/g}$.

Radionuclide	Water soluble	Water insoluble	Ratio w.insoluble/w.soluble
Cs ¹³⁷	2.74	8.37	3.05
Pu ^{239/240}	5.22E-6	0.1	19680
Sr ⁹⁰	2.3E-3	1.42	167
U ²³⁸	3.88E-7	5.64E-5	145

- Under then prevailing equilibrium state of tank BX-107 residues and conditions of the extraction procedure, aluminum, bismuth, iron, mercury, nickel, and silica compounds exhibited low water solubilities. The sodium compounds exhibited high water solubility, an indication that the sodium compounds are present as highly-soluble salts.
- Fluoride compounds were mostly water insoluble with about 47% solubility.
- Nitrate compounds were present in a highly soluble form, with about 8.5% left in the solid matrix.
- Phosphate compounds exhibited about 17% water solubility.
- Sulfate compounds exhibited about 87% water solubility.
- Cesium compounds exhibited about 25% water solubility.
- The bulk of strontium, plutonium, and uranium compounds were water insoluble.

Figures 2-8 and 2-9 are graphic representations of inorganic and radionuclides data reported in Tables 2-15, 2-16, and 2-17. A ratio number annotates analytes for which both water-soluble and water-insoluble concentrations were reported.

Note that the water-soluble data reported represents a single extraction stage under the physical conditions specified in the water extraction procedure. However, no data referring to solvent/solid extraction ratio were recovered.

2.4.4 Common Denominator in Tank BX-107 Waste

2.4.4.1 Major Analytes. The analytical data from 1974, 1979, and 1990, coupled with TRAC and process knowledge predicted compounds and analytes, provide a baseline to

Water Soluble and Water Insoluble Electrolytes Comparison - 1979 Core

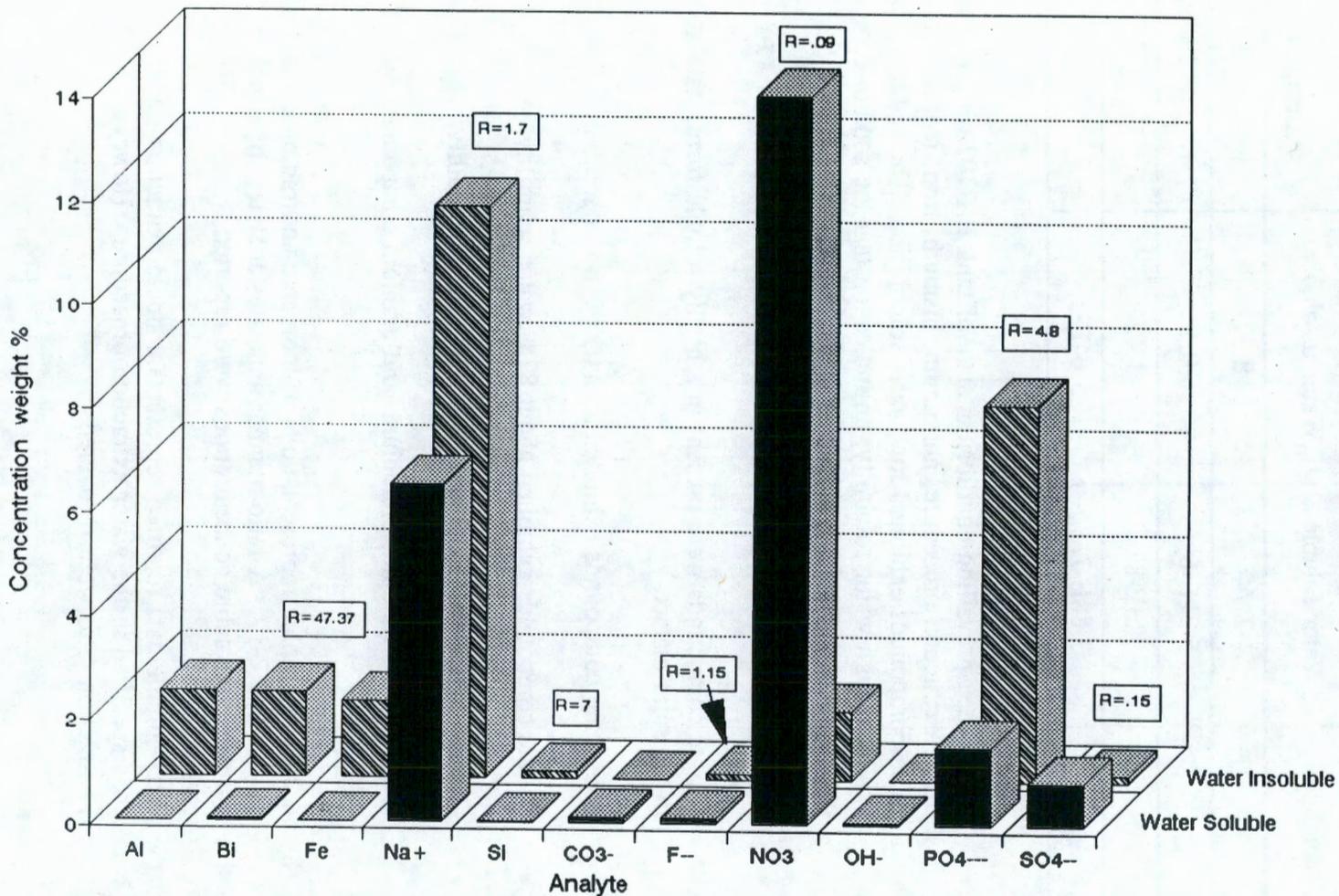
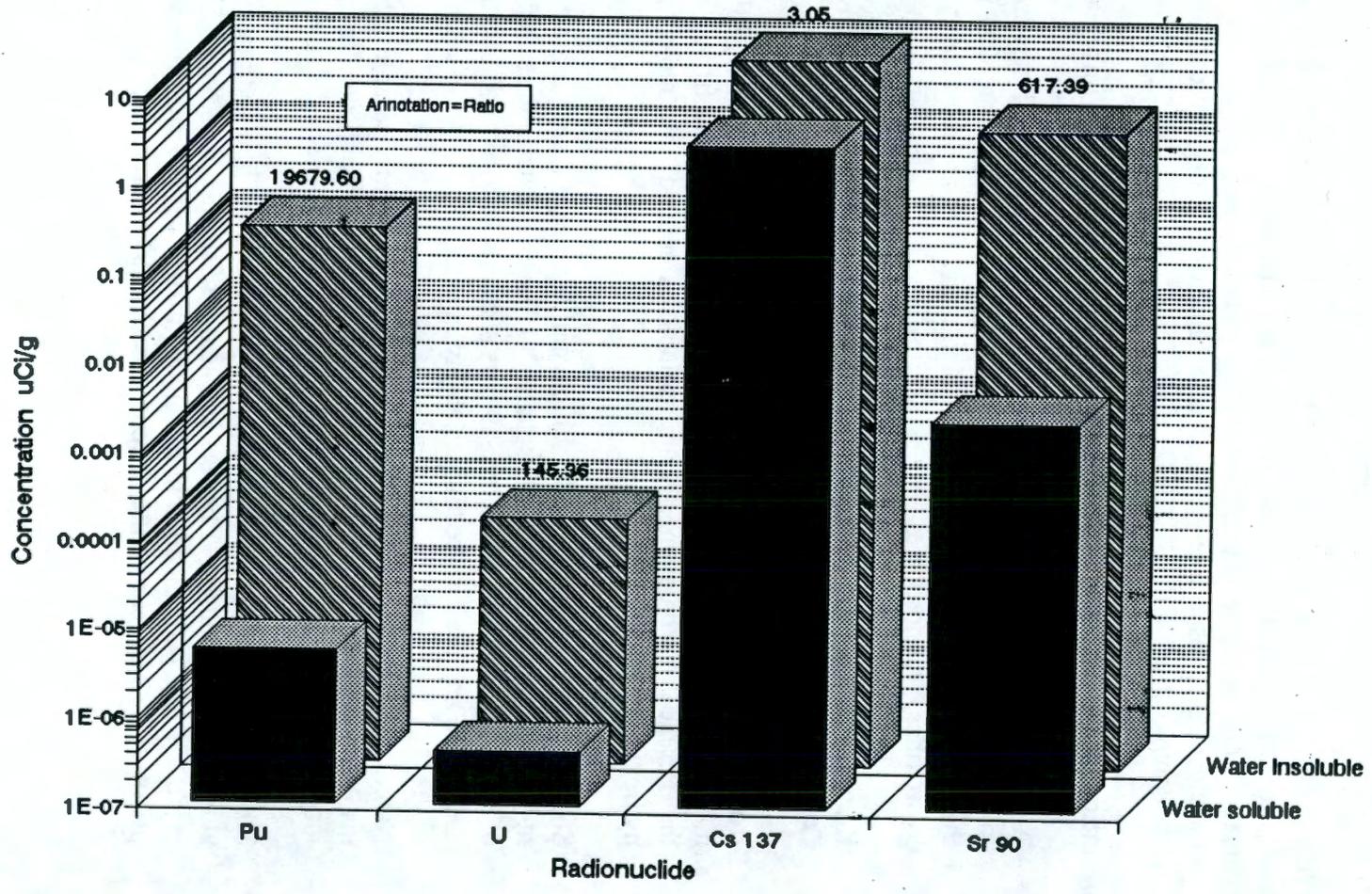


Figure 2-8. Water Soluble and Water Insoluble Electrolytes Comparison - 1979 Core.

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Figure 2-9. Water Soluble and Water Insoluble Radionuclides Comparison - 1979 Core.



identify the major matrix constituents of tank BX-107 waste. An attempt to assess the accuracy of TRAC or process knowledge predicted concentrations is beyond the scope of this report.

Table 2-18 lists TRAC, process history, and 1979 analytical data (the sum of soluble and insoluble concentrations reported) for select inorganic analytes and radionuclides*. TRAC inventory estimates were multiplied by 1985 waste quantities in tank BX-107 to yield the units of moles/L and $\mu\text{Ci/L}$, respectively. TOC data suggests that organic compounds present are water soluble.

2.4.4.2 Speciation. Literature and 1979 core data provide the following information.

- Aluminum compounds can range from soluble aluminum sulfates and nitrates to insoluble hydroxides and phosphates. However, 1979 core data indicate that the bulk of aluminum species are insoluble and thus are most likely oxides, hydroxides, or phosphates.
- Known bismuth compounds, mostly hydroxides and phosphates, are insoluble. Data from 1979 verifies this fact.
- Iron compounds range from soluble ferrous and ferric fluorides, sulfates, and nitrates to insoluble phosphates and hydroxides. Core data from 1979 indicate the presence of iron as mostly an insoluble compound, thus mostly as hydroxide or phosphate.
- Sodium compounds are mostly soluble and can range from nitrates, carbonates, hydroxides, and sulfates to phosphates. 1979 core data indicate high solubility of sodium compounds. Table 2-19 lists some sodium compounds, their density and color, and their respective solubility in water.
- Nickel compounds can range from fluorides and slightly soluble sulfates to insoluble hydroxides and phosphates. 1979 core data indicate that nickel is mostly insoluble and thus a hydroxide or phosphate.
- Silicon is probably present as silica (SiO_2) embedded in a sodium aluminosilicate matrix. Fluorosilicic acid ($\text{H}_2\text{SiF}_6 \cdot 2\text{H}_2\text{O}$ or $(\text{NH}_4)_2\text{SiF}_6 \cdot x\text{H}_2\text{O}$) crystals could be embedded in the sludge matrix but are most likely depleted at pH of >7 . This conjecture is confirmed by 1979 core data.

*Section 5.9 will have a sorted reproduction of this table including the most recent (1992) core sampling data.

Table 2-18. Process Knowledge, TRAC, and 1979 Core Data Comparison.

Inorganic analytes moles/L (wet-basis)	Process knowledge	TRAC	1979 Core
SO ₄ ⁻²	0.09	NR	0.14
PO ₄ ⁻³	0.22	0.22	0.133
NO ₃ ⁻	1.95	0.73	3.58
Na ⁺	4.08	2.94	6.94
OH ⁻	0.20	0.44	0.043
Al ⁺³	0.25	0.073	0.88
Bi ⁺³	0.08	0.05	0.11
CO ₃ ⁻	0.14	0.59	0.024
Cr ⁺³	0.002	0.0007	0.002
F ⁻	0.13	0.07	0.076
Fe ⁺³	0.02	0.17	0.37
NH ₄ ⁺	0.073	NR	NR
NO ₂ ⁻	0.61	0.059	NR
Ni ⁺⁺	NR	NR	0.124*
Si ⁺⁴	0.023	0.015	0.066
Zr ⁺⁴	0.0002	0.015	NR
Ba ⁺²	NR	2.2E-6	1.96E-4
Radionuclides μ Ci/g			
Pu ^{239/240}	67E-8	2.76E-2	0.103
U ²³⁸	6.27E-4	4.02E-4	5.67E-5
Zr ⁹³	NR	1E-3	NR
Am ²⁴¹	NR	1.51E-3	NR
Cs ¹³⁷	NR	NR	11.11
Ba ¹³⁷	NR	10.05	NR
Sr ⁹⁰	NR	25.13	14.2
Tc ⁹⁹	NR	2.51E-3	NR

*Probably due to method contamination because this result was not confirmed by the 1992 core data.

Table 2-19. Physical Characteristics of Select Sodium Salts (Lange 1965).

Compound	Density g/cc	Color	Solubility in water
NaNO ₃	2.26	Colorless to white	1:1.1
Na ₂ CO ₃ ·10H ₂ O	1.46	Transparent	1:3
NaOH	2.13 dry	white	1:1
Na ₂ SO ₄ ·10H ₂ O	1.46		1:1.5
Na ₂ HPO ₄ ·7H ₂ O	1.7	Hygroscopic powder	1:8
NaHPO ₄ ·2H ₂ O	1.9	Colorless crystals	1:1
Na ₃ PO ₄ ·12H ₂ O	1.6	Colorless to white crystals	1:1.35

- Cesium compounds can range from carbonates, hydroxides, and nitrates to sulfates and are mostly soluble. 1979 data indicates a 1/3 solubility ratio. Thus, some cesium compounds could be attached to mineral lattices.
- 1979 data indicates that plutonium is mostly in water insoluble forms.
- Strontium compounds can range from soluble fluorides, hydroxides, and nitrates to insoluble phosphates. 1979 core data indicates that strontium is in insoluble forms.
- Uranium compounds also vary in solubility. Nitrates and sulfates are soluble while phosphates, tetrafluorides, and hydroxides are insoluble. 1979 core data indicate the presence of mostly insoluble uranium.

In summation, the following can be speculated.

- Aluminum is most likely present as hydroxide or phosphate (insoluble).
- Bismuth is most likely present as hydroxide or phosphate (insoluble).
- Iron is most likely present as hydroxide or phosphate (insoluble).
- Sodium is most likely present as a sodium nitrate (soluble).
- Nickel is most likely present as hydroxide or phosphate (insoluble).
- Silicon is most likely present as fluorosilicic acid and silica/aluminosilicates.

- Cesium is most likely present as phosphates, hydroxides and salts (within silicate matrix).
- Plutonium is most likely present as a phosphate/hydroxide (insoluble).
- Strontium is most likely present as phosphate.
- Uranium is most likely present as fluoride, hydroxide, or phosphate (usually an oxide embedded in a phosphate matrix).

Table 2-20 was developed based on those observations and conjectures.

Equilibrium state is determined by many thermodynamic factors that are beyond the scope of this study. Appendix A contains an excerpt list of chemicals used during B Plant operations in the bismuth-phosphate process.

Data presented at this point provide a preliminary assessment of the conditions in the tank and will provide guidance and context in interpreting the most recent analytical results. Further discussions include the latest analytical results obtained for tank BX-107 and are presented in Section 6.0, Data Interpretation.

Table 2-20. Major Postulated Equilibrium Compounds in Tank BX-107 Residues Based on 1979 Core Data.

Major compounds	Other equilibrium species
$\text{Al}(\text{OH})_3 \cdot x\text{H}_2\text{O}$, AlPO_4	$\text{Al}(\text{OH})_3 \cdot x\text{SiO}_2$, plus other aluminum salts and oxides
BiPO_4	$\text{Bi}(\text{OH})_3$ plus other bismuth salts
$\text{Fe}(\text{OH})_2 \cdot x\text{H}_2\text{O}$, $\text{Fe}(\text{SO}_4)$	FeF_3 , $\text{Fe}_3(\text{PO}_4)_2$, other ferrous and ferric salts, other sulfates and hydroxides
Na_4SiO_4 , Metal silicate	$\text{H}_2\text{SiF}_6 \cdot 2\text{H}_2\text{O}$, $(\text{NH}_4)_2\text{SiF}_6$
NaNO_3	NaOH , other sodium salts
$\text{U}(\text{PO}_4)_2$, $(\text{UO}_2)_3(\text{PO}_4)_2$, $\text{UO}_2(\text{OH})$	Other uranium salts and hydroxides, UO_2 embedded in a phosphate matrix
Metal nitrates, carbonates and sulfates	Metal phosphate and hydroxides

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3.0 TANK CONTENTS SAMPLING OVERVIEW - 1992

3.1 DESCRIPTION OF SAMPLING EVENT

3.1.1 Sampling Schedule

The latest tank BX-107 sampling event occurred during May and June of 1992 in an effort to meet the Tri-Party Agreement sampling Milestone M-10-06 to obtain 24 cores from 12 tanks. Factors that contributed to the sampling decision are discussed in the *Waste Characterization Plan for the Hanford Site Single-Shell Tanks* (Winters et al. 1990).

3.1.2 Sampling Equipment

The push-mode sampling method, which is capable of accommodating liquids, sludge, and soft salt cakes, was used to sample the contents of tank BX-107. Figure 3-1 shows a typical sketch of the sampler. The sampler and the drill string assembly are mounted on a rotating platform located on a mobile sampling truck, as shown in Figure 3-2. During the sampling event, the stainless steel sampler is lowered into the material via a hydraulically-powered drill string. The sampler is capable of collecting 0.5-m (19-in.) long, ~0.02-m (7/8-in.) dia. cylindrical segments. A spring-loaded piston is used to create the negative pressure necessary to fill the sampler. A drill bit is used at the lower end of the drill string to pass through hardened material encountered during penetration. Drill ram hydrostatic fluid pressure is continuously monitored during the sampling operation. Once loaded, a rotary valve at the lower end of the sampler is shut and the sampler is pulled upward to the appropriate position. Then, using remotely controlled mechanical equipment, the sampler is unlatched and loaded into a stainless-steel lined lead-shielded cask for transportation to a laboratory hot cell. A normal paraffin hydrocarbon (NPH) liquid is added during sampler discharge from the drill string to prevent unwanted material from penetrating the sampler space.

The document *Tank Farm Operating Procedure* (Ross et al. 1992) provides a detailed overview of the proper core-sampling procedure as well as a list of additional reference documents. An animated video that depicts the operation of the push-mode sampler is also available in the Westinghouse Tank Waste Remediation System (TWRS) archives.

3.1.3 Sampling Event

Detailed sampling protocols, including volatile organics and radiation emissions monitoring prior to sampling are developed for each tank waste sampling event to alleviate possible safety concerns.

Figure 3-1. Push-Mode Sampler.

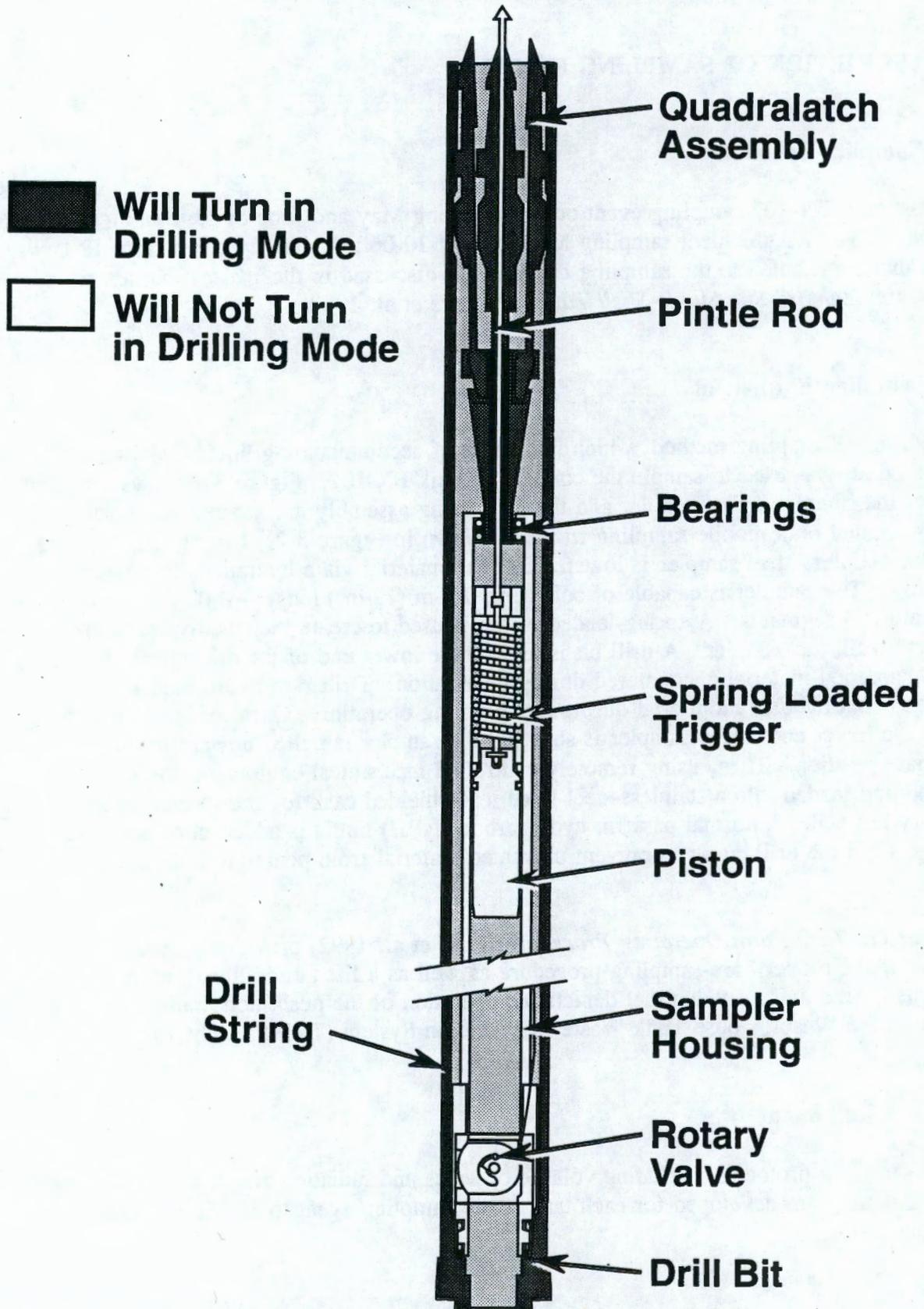
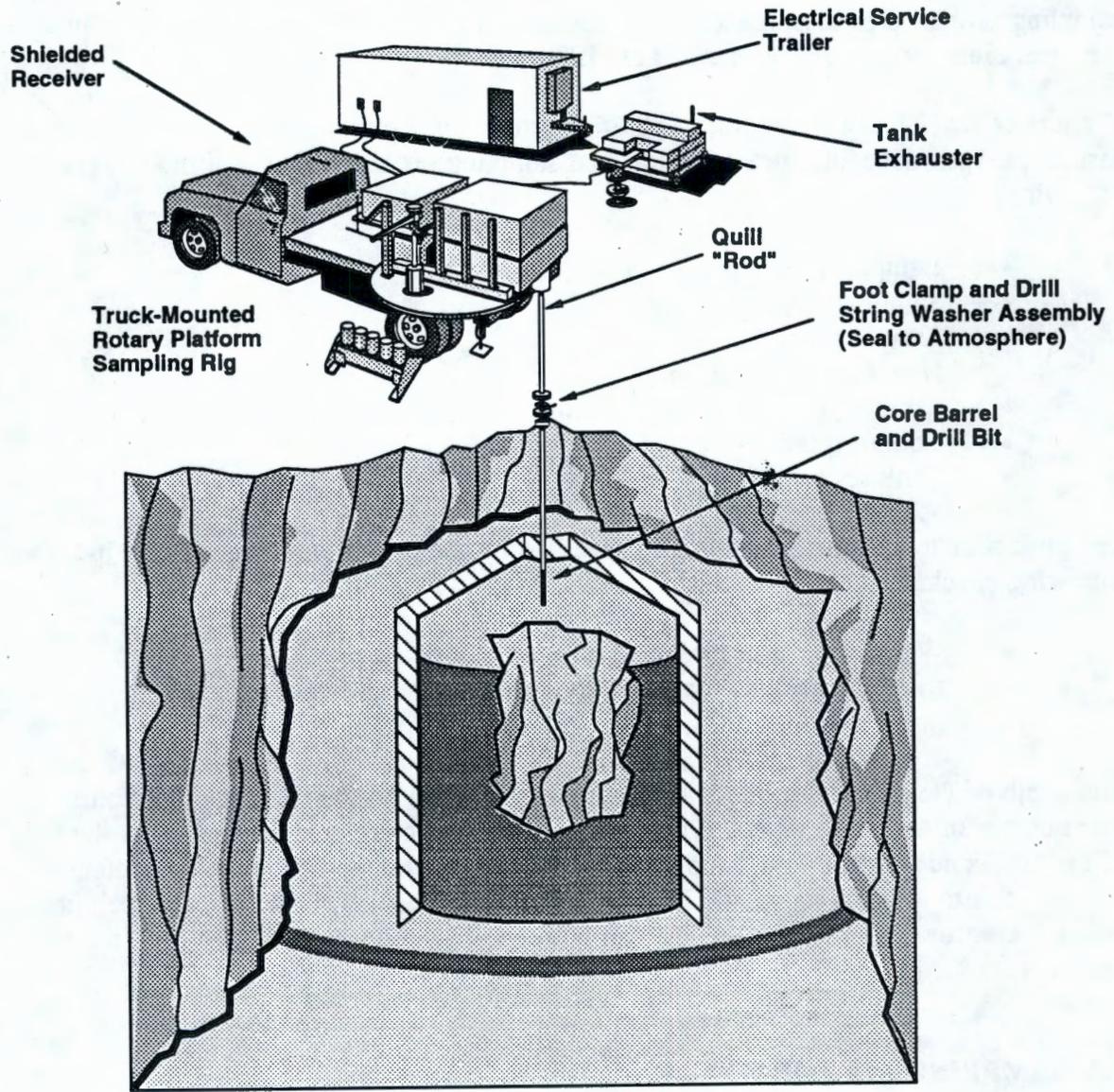


Figure 3-2. Mobile Sampling Truck.

Waste Characterization



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Core sampling of SSTs is performed in accordance with approved WHC procedures. Operating personnel are trained and certified to perform core sampling. During core sampling, a core sampling data sheet is completed to document the operation and a chain-of-custody record accompanies each sample from the field to the laboratory. Any sampling problems or discrepancies are recorded in a numbered field notebook maintained by the Operations supervisor (Winters et al. 1990).

Winters et al. (1990) defines responsibilities, safety and contamination controls, quality assurance, recordkeeping, prerequisites, and sampling procedures, which include the following.

- Equipment set up
- Sampling
- Sample recovery
- New sampler insertion
- Sample preparation for shipping
- Equipment clean up
- Tank restoration.

The tank riser to be sampled is usually specified by the person in charge (PIC), based on the following checks.

- Waste depth measurements
- Riser dimensions, contents, availability, and previous uses
- Sufficient open and level ground to permit using the sampling equipment.

The depth of the material in the tank is determined based on the latest level measurements. The number of samples that could possibly be taken are calculated starting from the bottom of the tank, and thus the first sample taken vertically at the top of the tank's content may not be a full 0.5 m (19 in.) segment. Following these calculations, drill rods are specified. Additional information on this subject can be found in the documents specified in the previous subsection.

3.2 SAMPLING PERFORMANCE

3.2.1 Hydraulic Ram Pressure Data

Hydraulic ram pressure data analysis (see Appendix A) postulates that most of the material encountered along the riser 7 (core 40) vertical axis is similar in consistency, whereas material encountered along riser 3 (core 41) is heterogeneously layered and exhibits higher resistance to sampler penetration at approximately 1.27 m (50 in.) from the waste surface. In general, however, resistance to sampler penetration, though slightly higher along riser 3 (core 41), averaged about 703,100 kgs/m² (1,000 psi) for both risers.

3.2.2 Sample Recovery Data

Two cores were sampled from tank BX-107 risers 7 and 3. Each core consisted of 7 segments. Rotary valve mechanical failures were reported for segments 1 and 3 of the first core sample (core 40) and for segments 1, 4, and 5 of the second core (core 41). Those results are summarized and averaged in Table 3-1.

The mean was calculated excluding the first segment recovery. Recovery values were generically calculated by dividing the total volume of the recovered sample, based on actual dimensional measurements data, by the total volumetric capacity of the sampler:

$$\text{Percent Recovery} = [\text{Liquid Volume} + \text{Solid Volume} / \text{Expected Volume}] * 100$$

The following factors were attributed to the variability in sample recovery (Propson 1993):

- Tank contents level
- Equipment capabilities such as bit design, seals, and closure valve
- Hydrostatic fluid properties and behavior
- Waste stratification and heterogeneity
- Transition layers (created as a result of the current sampling procedure and the limitations of the sampler used).

Table 3-1. Push-Mode Sample Percent Recovery (1992).

Segment	1	2	3	4	5	6	7	Mean
Core 40	0	100	0	100	100	100	66	78
Core 41	0	100	50	0	5*	100	100	59

*Hot cell chemist observation reported 2.4%.

3.3 SAMPLE TRANSFER TO LABORATORY HOT CELL

3.3.1 Sample Packaging for Transportation

Sample loaded casks were sealed and transported in four shipments to the 222-S Laboratory between June 10 and 12, 1992, 28 days from the beginning of the sampling operation, and 9 days following its completion. Table 3-2 summarizes sample handling statistics.

Table 3-2. Sampling to Shipment Time Span in Days (1992) (± 1 day).

Segment No.	1	2	3	4	5	6	7
Core 40	28	28	26	26	26	23	3
Core 41	14	13	13	9	9	9	9

Cask radiation survey data, as indicated in the chain-of-custody records, were below U.S. Department of Transportation (DOT), U.S. Environmental Protection Agency (EPA), and DOE, regulated safety transportation requirements. Document number TO-080-090, *Ship Core Sample*, contains detailed procedural information regarding this subject.

3.3.2 Chain-of-Custody Records

Chain-of-custody records are completed for each of the segments sampled. They contain information which uniquely identifies each sample (see Appendix A).

No discrepancies were found in core 40 and 41, segments 1 through 7 chain-of-custody records. Copies of those records are archived with the sampling cognizant engineer and can be found within the tank BX-107 cores 40 and 41 data package document, WHC-SD-WM-DP-028. Table 3-3 summarizes the dose rate through the drill string, in millirem per hour, during sampling.

Tank BX-107 core samples were transported to the 222-S Laboratory hot cell in a specialized truck without incident.

Table 3-3. Dose Rate Through Sampler Drill String in mR/hr (1992).

Segment No.	1	2	3	4	5	6	7
Core 40	6	140	3	100	35	25	25
Core 41	50	NR	50	2	NR	2	33

4.0 SAMPLE HANDLING SCHEME - 1992

4.1 SAMPLE PREPARATION PROCESS DESCRIPTION

Tank BX-107 core samples 40 and 41 were received by the 222-S Laboratory from June 26 to September 24, 1992. The sample casks were logged in, surveyed for radiological control, and verified for seal integrity. The core samples usually remain in the transportation cask at room temperature until their scheduled extrusion phase. Daily routine inspections are performed.

4.1.1 Sample Extrusion

Core samples are extruded in the 1E-2 hot cell at the 222-S Laboratory. Each segment sampler contained within the cask liner is remotely loaded into the hot cell through the air lock system. The liner is then remotely opened and the sampler is withdrawn. Any liquids recovered from the liner are measured for volume and retained for analysis if an adequate volume is obtained.

The sampler is remotely loaded onto the mechanical extruder. The segment is removed from the sampler by driving an internal piston the length of the sampler body and extruding the waste into a receiving tray. The sampler is removed and later decontaminated for future use. Any drainable liquid in the sample flows into a pre-weighed receiver at one end of the tray. Samples for volatile organics tests are taken immediately after extrusion. The recovered material is weighed, measured, and photographed, and any observations are recorded and maintained in a permanent record. Each sample is then transferred to a sealed container for storage in the hot cell until all segments from the entire core have been extruded (Winters 1990). During this operation, which lasts approximately 2 hours, the samples are exposed to high air flow resulting in moisture loss and possible species oxidation. Efforts are made to minimize exposure of the sample to hot cell atmosphere. No preservatives are added to the collected sample aliquots. The weights of the extruded segments are provided in Table 4-1.

Hot cell volume estimates and density calculations were also reported but are biased and inaccurate because of procedural error, and thus will not be reported at this point. Free liquid volumes were reported as insignificant.

Segment extrusion description sheets are enclosed in the physical properties summary section of the tank BX-107 cores 40 and 41 data package. They provide information such as segments descriptions, free water contents, and negatives of segment photographs.

Unfortunately, the photographs (plus negatives) taken during tank BX-107 core segments extrusion were misplaced and cannot be found. No video records of the extrusion process are available either.

Table 4-1. Weights of Extruded Segments in Grams (1992).

Core 40, segment 1	0
Core 40, segment 2	204.31
Core 40, segment 3	0
Core 40, segment 4	216.57
Core 40, segment 5	218.04
Core 40, segment 6	215.81
Core 40, segment 7	132.45
Core 41, segment 1	0
Core 41, segment 2	186.17
Core 41, segment 3	185.23
Core 41, segment 4	0
Core 41, segment 5	5.53
Core 41, segment 6	248.04
Core 41, segment 7	250.2

Table 4-2 summarizes the results of the hot cell chemist's physical assessments. The equivalent to 100% segment recovery, 187 ml, was used as a basis to estimate percent recovery. Core 41, segment 5 was approximately 4.5 ml, and thus was completely used for VOAs, percent water, and DSC studies.

Segments free liquid (liquid collected by the cask liner during extrusion plus additional free liquid in the sampler) was reported below 25 ml for all segments. Those liquids were re-combined and homogenized with their parent segments.

Request for special analysis forms accompanied the appropriate sample to the analytical laboratory hot cell. Those forms contain information relative to sample origin, ID, sample weight, radiation levels, and analysis requested. Copies of those forms are included in Addendum 1 of the tank BX-107 cores 40 and 41 data package.

No notes relative to NPH contamination were found. Moisture loss from the sample during extrusion and homogenization were not addressed by the administering chemist.

During the extrusion process for the core segments of tank BX-107, it was discovered that only about 60% of the expected material was obtained (Kristofzski 1992). Thus, material from the archive samples was used to build the core composites. No resampling activities were performed.

4.1.2 Sample Breakdown and Waste Description

After the entire core has been extruded, all segment portions (liquid and solid) are transferred to the 1E-1 hot cell where they are prepared for analysis. If more than 25 ml of segment is free liquid it will be saved, composited, and analyzed separately from the solids. Liquid volumes less than 25 ml are returned to the solids before homogenization.

Prior to homogenization a small solid aliquot is removed from the segments for particle size analysis. Samples for volatile organic analysis are taken immediately. Penetrometer measurements are made for every segment (Winters et al. 1990).

Samples were appropriately labeled for tracking purposes. A list of sample identifications can be found in cores 40 and 41 data package narrative section.

Summary lists depicting sample breakdown, homogenized segments sampling, and core composite sampling requested analysis versus sample amount can also be found in that same section.

Table 4-2. Visual Assessments as Reported the by Hot Cell Chemist (1992).

Core	Segment	Color	Texture	Consistency	Air pockets	Free liquids
40	2	Grey	Light	Granular creamy	Yes	NR
40	4	Grey	Light	Granular bottom 2 in. dry	Yes	NR
40	5	Bottom 2/3 grey	Bottom 2/3 light	Bottom 2/3 granular top 1/3 sloppy	Yes	NR
40	6	NR	NR	Top 1/3 sloppy bottom 2/3 juicy	NR	Little
40	7	Bottom 2/3 white	Crystal	Adhesive	NR	NR
41	2	Grey	Lighter	NR drier	Yes	NR
41	3	Grey	Shiny	Slick	NR	NR
41	6	Grey	Light	NR moist	NR	NR
41	7	Grey/green	Light	NR moist	NR	NR

4.1.3 Sample Homogenization

Subsequently, core segments are individually homogenized in a holding jar using a hand-held rotor stator. It should be noted that core 40 segments 1 and 3 were empty. Core 41 segments 1 and 4 were also empty (due to sampler valve failure).

4.1.4 Sample Compositing

Tank BX-107 blend plans indicate that equal amounts were used from each recovered segment to build core composites (18 to 19 g from each segment for core 40 and approximately 21 to 22 g for core 41). Two composites were formed from each of the tank BX-107 cores sampled in 1992. The sample breakdown scheme is depicted in Figure 4-1.

Sample aliquots were collected from the homogenized segments rather than from the extrusion tray, according to characterization change notices WHC-SOW-91-0006 dated July 23, 1992 and August 13, 1992 (Kristofzski 1993). Those notices also state that because of the insufficient amount of sample left from core 40, segment 2, the second composite of that core will use whatever material is left in segment 2.

4.1.5 Sample Archiving

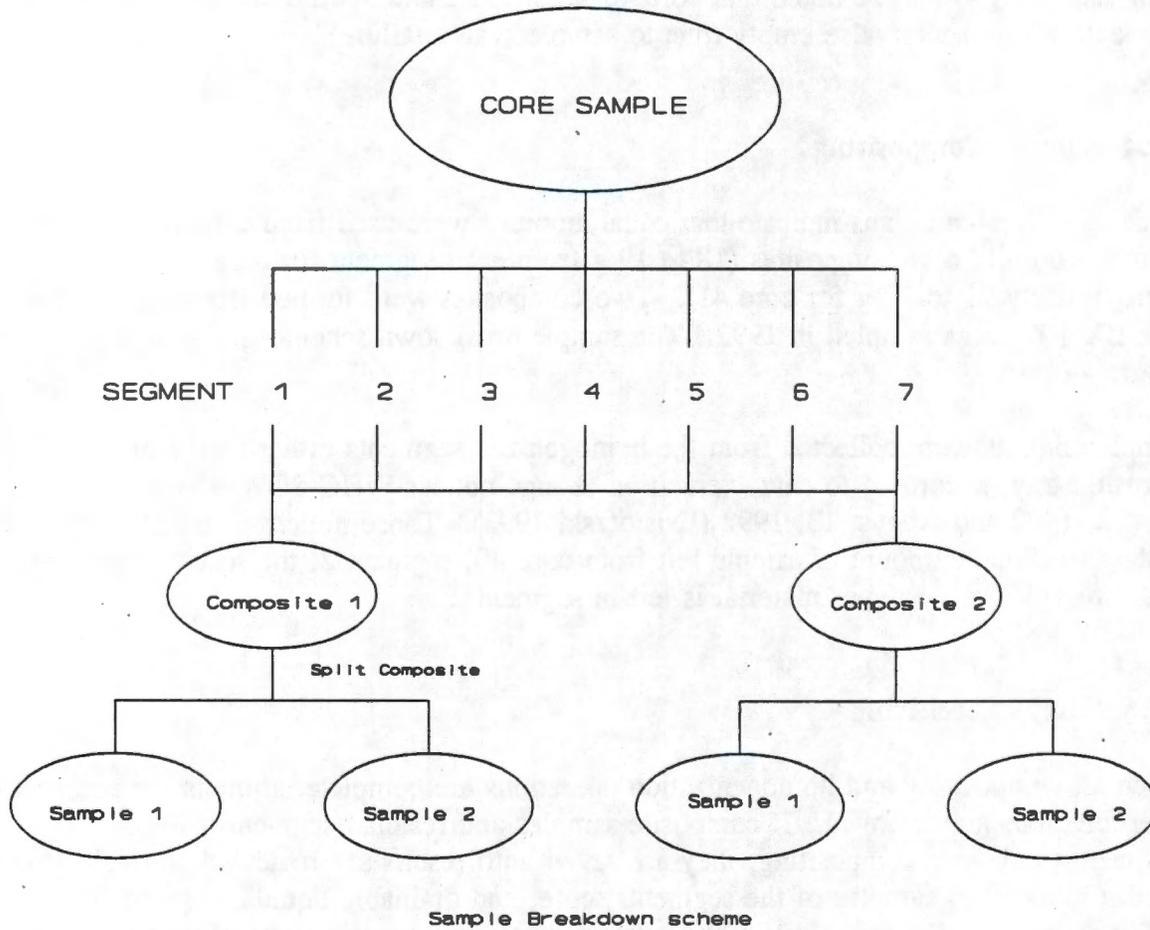
When all compositing and homogenization operations are complete, aliquots for analytical determinations are removed. If composite samples and residual segment materials are sufficient to allow recompositing, they are saved until results are reviewed and validated. Small (30 to 50 g) samples of the segments, core, and drainable liquid are saved for additional testing (Winters et al. 1990). Other portions are taken and archived appropriately for approximately 1 year. All activities records are permanently maintained by the HAS.

4.2 SAMPLES TRANSFER SCHEME

Sub-samples of the composites and of the core segments were shipped to the PNL 325-B hot cell for analytical purposes. Those samples included (1) two core composites from core 40; (2) two core composites from core 41; (3) segments 2, 4, 5, 6, and 7 sub-samples of core 40; and (4) segments 2, 3, 5, 6, and 7 sub-samples of core 41.

Inter-laboratory sample chain-of-custody records and sample receipt form copies can be found in Kristofzski (1993). Sample chain-of-custody records provide various sample information including shipping and receiving points, personnel, and sample identification. Sample receipt records also provide administrative and technical information relative to recordkeeping and sample condition. No discrepancies were observed in these records.

Figure 4-1. Sample Breakdown Scheme.



Core 40 (segments 7 and 5) and core 41 (segments 2, 3, 5, 6, and 7) were delivered on July 30, 1993. Core 40 (segments 2, 4, 6 and composites 1 and 2) and core 41 (composites 1 and 2) were transferred from the 222-S Laboratory to PNL 325 hot cell on September 24, 1993. In addition, sample receipt description forms were also completed. Information compiled in those forms includes observational comments, size and condition of the sample container, sample gross weight, and physical description.

Other composites and segments aliquots slotted for water extraction, acid digestion, fusion, TGA, DSC, and particle size analysis remained in the 222-S Laboratory.

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5.0 ANALYTICAL RESULTS AND WASTE INVENTORY - 1992

5.1 ANALYTICAL SCOPE OF WORK

Addendum 4, Appendix B1 of the tank BX-107 data package (Kristofzski 1993) contains test instructions relative to the samples forwarded to the PNL 325 Laboratory. They state that the solid core composites - a total of 4 - from both core samples 40 and 41 are to be analyzed for semi-volatile organics (semi-VOA), extractable halides (EOX), percent solids, and plutonium/uranium isotopes. Segments 5 and 7 of core 40, as well as segments 2, 3, 5, 6, and 7 of core 41, are to undergo volatile organics testing, while segments 2, 4, and 6 of core 40 are to be used for rheology and other physical testing such as percent solids, settling velocity, and density. Sample aliquots were assigned unique laboratory IDs. Appendix C contains tabulated segment identification number information and laboratory procedure numbers corresponding to the analytical tests applied. Table 5-1 presents this information in a structured format. A general analytical scheme flowchart for baseline tanks such as tank BX-107 can be found in Appendix I of Winters et al. (1990).

Test instruction records also provide information and general guidelines related to the preparation, analysis, and quality control procedures used. They include laboratory location, aliquots quantities, and duplicate/spike/blank requirements.

5.2 SAMPLE PREPARATION FOR ANALYSIS

Sample were prepared for analysis in PNL-325 and 222-S Laboratory hot cells. Preparation, as mentioned in Section 4.0, included necessary sample splitting (for duplicates), homogenization, and extraction procedures. Properly labeled samples were then transferred accompanied by inter-laboratory chain-of-custody records to the appropriate laboratory section where required pre-analysis or method preparations were performed. Copies of those records are included in Addendum 4 of the tank BX-107 data package.

At this point, appropriate work was performed and bench sheets, as well as preparation data sheets were completed for each of the tests required. These records compile information relative to aliquots weights, any method deviation, laboratory method number, and other notes relative to test conditions, sample shortage, and the appropriate analysts' signatures. Samples were thus readied to undergo the preconceived analytical scheme.

5.3 PHYSICAL TESTS

5.3.1 Methods

As indicated in Table 5-1, cores 40 and 41 composites were evaluated for percent solids content. Segments 2, 4, and 6 of core 40 underwent physical and rheological tests such as

Table 5-1. Analysis Scheme for Cores 40 and 41.

VOAs (PNL)	Core 40, segments 5 and 7 Core 41 segments 2, 3, 5, 6, 7
Density, % solids, rheology (PNL)	Core 40, segments 2, 4, 6
SVOAs, EOX (PNL), TOC (222.5)	Core 40, composites 1 and 2 Core 41, composites 1 and 2
Solids, metals, anions, radionuclides (222-S)	
TGA/DSC (222-S)	All core segments and composites

percent solids, settling behavior, density, and viscosity. Particle size analysis was performed on all recovered core segments (in the 222-S Laboratory).

Density for solid samples was calculated based on the net weight and volume following centrifugation for 1 hour at over 1,000 times gravity. Volume and weight percent centrifuged solids and supernate were also calculated.

Settling rates were conducted in pre-weighed, volume graduated, centrifuge tubes. Two dilutions, 1:1 and 3:1, were prepared for each of the segments (2, 4, and 6 of core 40).

Weight-percent solids was conducted in a drying oven at 105 °C for 24 hours following overnight air-drying at room temperature. Undissolved solids weight percent was calculated by subtracting the percentage of dissolved solids in the liquid supernate part of the sample from the total solids weight percent.

Shear strength was measured using a specialized vane viscometer. The sample was placed in a water bath at 32 °C for a minimum of 1 hour. A shear vane was then placed into the sample and rotated at 0.3 rpm. The shear stress of the sample was recorded as a function of time. The shear strength was then calculated appropriately. The method was repeated at 95 °C.

Because segments 2, 4, and 6 of core 40 were nonpourable at room temperature, causing viscometer slippage, and because of the drying action that could occur at 95 °C, shear stress as a function of shear rate measurements were run in duplicates on one-to-one and three-to-one dilutions of those samples. Shear stress was recorded as a function of the shear

rate generated by the rotating viscometer conical spindle. The viscosity was then calculated by dividing the shear stress by the shear rate. The yield stress was also obtained from this measurement. Plots depicting shear stress and viscosity as a function of shear rate were generated. They are included in Addendum 4, Appendix C of the tank BX-107 cores 40 and 41 data package.

No penetrometer testing was performed on cores 40 and 41 samples. It was stated that the instrumentation available would not have provided useful data. All the segments were highly fluid state and would have been well below the measurable range of the instrument.

Particle size analysis was performed at the 222-S Laboratory using scattering laser technology following sonication suspension of the sample aliquots in a DI water matrix. Particle number and volume histograms were generated. Procedures relative to those methods are appropriately referenced in the narrative section of the data package (Kristofzski 1993).

Appropriate test instructions and data sheets were completed for the quantitative tests performed. They were included again, along with associated graphs, in Addendum 4, Appendix C, of the tank BX-107 cores 40 and 41 data package (Kristofzski 1993).

All tests except particle size analysis were performed in duplicate and abiding by the appropriate quality assurance measures.

Method performance, except for particle size analysis, is thus considered good and results are defensible. The particle size analysis results were potentially biased by the dissolution of some of the soluble salt crystals of the sample in the water dispersant. An assessment of that bias was not reported.

5.3.2 Results

Table 5-2 summarizes weight percent solids for cores 40 and 41 composites. Table 5-3 summarizes physical and rheological test results for segments 2, 4 and 6 of core 40.

Those samples were described as being light tan in color and having various amounts of free liquids. Copies of the settling rate curves are included in Appendix A.

Significant decrease in viscosity was observed as the temperature of the samples increased. They exhibited a behavior generally observed with materials similar to greases and lubricants (Kristofzski 1993).

Table 5-4 summarizes particle sizes analysis results for the recovered segments of cores 40 and 41. Percent solids (or sample) in the dispersant (water) ranged from 1.5E-3 to 7.1E-3.

Table 5-2. Average Weight Percent Solids-Cores 40 and 41 Composites (PNL).

	Composite 1	Composite 2
Core 40	43.75	44.675
Core 41	45.835	NR

Table 5-3. Physical And Rheological Results for Core 40 Segments, 2, 4, and 6 (PNL).

Property	Sample type	Units	Segment 2	Segment 4	Segment 6
Settled solids	Segment aliquot	Volume %	100	100	97.8
Centrifuged solids	Segment aliquot	Volume %	71.7	76	66
		Weight %	77.2	80.6	70.4
Density	Segment aliquot	g/ml	1.47	1.45	1.4
	Centrifuged supernate	g/ml	1.18	1.17	1.21
	Centrifuged solids	g/ml	1.58	1.54	1.49
Solids	Segment aliquot	Weight %	44.8	45.6	42
Undissolved solids	Segment aliquots	Weight %	38	39.6	32.6
Shear strength	Segment aliquot	Dynes/cm ²	3,000	8,800	4,800
Viscosity	Diluted aliquots	Centipoise	2	2	2

Table 5-4. Particle Size Distribution (in μm) for Cores 40 and 41 Segments (222-S Laboratory).

Sample	Distribution	Particle Size Range						
		0.5 - 1 μm	1.5 - 1.5 μm	1.5 - 2 μm	2 - 5 μm	5 - 10 μm	10 - 20 μm	20 - 150 μm
Core 41 Seg. 7	Number %	58.97	18.98	6.54	13.2	2.05	0.21	0.06
	Volume %	0.53	0.79	0.74	11.26	11.05	12.17	63.46
Core 41 Seg. 6	Number %	69.11	15.23	5.19	9.31	1.09	0.06	0.01
	Volume %	2.91	2.97	2.78	34.18	26.49	13.6	17.06
Core 41 Seg. 5	Number %	59.9	21.01	7.19	10.44	1.23	0.15	0.08
	Volume %	0.23	0.38	0.36	3.4	2.98	3.54	89.11
Core 41 Seg. 3	Number %	64.97	20.65	5.99	7.44	0.85	0.08	0.01
	Volume %	1.94	2.85	2.27	18.15	15.93	13.89	44.96
Core 41 Seg. 2	Number %	77.07	15.44	3.15	3.76	0.45	0.09	0.03
	Volume %	1.53	1.42	0.8	6.1	5.8	12.51	71.85
Core 40 Seg. 7	Number %	67.8	16.27	5.23	9.41	1.22	0.06	0.01
	Volume %	2.17	2.41	2.13	26.63	23.21	10.3	33.15
Core 40 Seg. 6	Number %	66.45	17.3	5.63	9.33	1.16	0.13	0.01
	Volume %	2.23	2.69	2.4	26.65	23.97	23.15	18.89
Core 40 Seg. 5	Number %	58.94	19.69	7.48	12.48	1.32	0.06	0.02
	Volume %	1.1	1.71	1.78	19.56	14.18	6.7	54.97
Core 40 Seg. 4	Number %	68.64	16.77	5.04	8.48	0.99	0.07	0.01
	Volume %	2.51	2.84	2.34	27.07	22.2	14.23	28.81
Core 40 Seg. 2	Number %	74.05	15.11	3.93	5.88	0.9	0.1	.02
	Volume %	1.38	1.3	0.93	9.55	10.59	10.46	65.78

5.4 RADIOCHEMICAL ANALYSIS RESULTS

5.4.1 Methods

KOH/HCl fusion aliquots of composites 1 and 2 of cores 40 and 41 underwent radiochemical analysis for plutonium and uranium concentrations. Uranium was determined by a laser fluorimetry technique. Plutonium was determined by chemical separation followed by alpha counting and alpha energy analysis (AEA). Uranium and plutonium isotopes were selectively analyzed using ionization mass spectroscopy at PNL.

The total beta (TB) counting procedures and gamma energy analysis (GEA) were used to detect beta and gamma emitting isotopes, respectively.

At the PNL Laboratory, results were completed 3 to 5 months following sample receipt. The field blank uranium concentration was less than the method detectable activity (MDA) of 2 $\mu\text{g/g}$, approximately 1,000 times below samples concentrations. The field and hot cell blanks concentrations were below the instrument detection limit (IDL).

Plutonium field blank concentration was over 200 times less than cores 40 and 41 composite samples for AEA analysis. The plutonium process blank read $4\text{E-}7 \mu\text{Ci/g}$, which is approximately three orders of magnitude lower than the samples and indicates that there was little contamination of the samples during preparation (PNL).

Most concentrations reported were about 2 to 6,000 times above IDLs. Standard recoveries were all acceptable. Uncertainties were calculated for all the reported results.

At the 222-S Laboratory, total alpha corresponds to the sum of plutonium and americium for all core composite samples, except core 40, composite 2. The high ratio of the AT to the plutonium and americium results indicate:

- The potential presence of undetermined alpha emitters
- Beta "cross talk"
- Incorrect plutonium and americium recoveries (incomplete exchange with spike).

The ratio of total beta to the sum of strontium/yttrium and Cs^{137} shows good results agreement. Cesium-137 beta activity was based on results reported from gamma spectroscopy.

There are also indications that I^{129} was lost during acidification of the fused sample prior to analysis. Carbon-14 analysis may be biased because of standard failure.

GEA analysis spikes percent recoveries values were acceptable (75% to 125%) for most radionuclides analyzed except for americium. Technetium and plutonium spikes were about 70%.

Appropriate method quality assurance procedures were applied and instruction/analytical data sheets were completed. Copies of those data sheets are included in the tank BX-107 cores 40 and 41 data package.

5.4.2 Results

Plutonium and uranium isotopic analysis were reported in tank BX-107 cores 40 and 41 data package, Addendum 4, Appendix D3. Tables 5-5 and 5-6 summarize the results.

U^{238} and Pu^{239} are the dominant isotopes present with lesser quantities of U^{235} and Pu^{240} , and nominal quantities of the remaining identifiable isotopes. Plutonium result errors were reported at approximately 7% for the samples and 48% for the blanks. Blank results were not subtracted from sample results. Dilution factors ranged from approximately 8 to 100,000 times prior to performing actual mass spectroscopy analysis.

Table 5-7 summarizes average radiochemical results reported in Addendum 2 of the Tank BX-107 cores 40 and 41 data packages.

Field and Hot Cell Blanks Total Beta (TB) results were $1.44E-4$ and $7.64E-4$ $\mu\text{Ci/g}$, respectively.

5.5 THERMAL-GRAVIMETRIC ANALYSIS RESULTS

5.5.1 Methods

Thermal-gravimetric analysis (TGA) and differential scanning calorimetry (DSC) were performed on cores 40 and 41 segments and composites. TGA consists of heating approximately an aliquot at a controlled rate during which sample weight variation is recorded. TGA scans usually determine water loss between room temperature and 120 °C, as well as chemical changes at elevated temperatures.

DSC is performed by heating a sample and reference pan at a controlled rate. The difference in temperature between the sample and reference is measured. The temperature differences represent physical or chemical changes occurring in the sample. Exotherms represent heat generated by the sample (e.g., combustion); endotherms result when heat is absorbed by the sample (e.g., melting, evaporation, or sublimation) (Winters et al. 1990).

Table 5-5. Uranium and Plutonium Results for Cores 40 and 41 Composites (PNL).

Core	Composite	Uranium $\mu\text{g/g}$	Plutonium $\mu\text{Ci/g}$
Field blank		<2	5.22E-5
40	1	1.81E+3	8.395E-2
40	2	1.705E+3	9.72E-2
41	1	2.26E+3	1.335E-1
41	2	2.225E+3	1.32E-1

Table 5-6. Uranium and Plutonium Isotopes Distribution for Cores 40 and 41 Composites (PNL).

Isotope	Mass percent range core 40	Mass percent range core 41
U^{234}	0.0051 - 0.0068	0.0055 - 0.0077
U^{235}	0.6753 - 0.7003	0.6738 - 0.6834
U^{236}	0.0055 - 0.103	0.0054 - 0.0077
U^{238}	99.2843 - 99.3133	99.3034 - 99.3154
Pu^{238}	0.0036 - 0.0043	0.004 - 0.0042
Pu^{239}	96.8142 - 96.9950	97.0820 - 97.1357
Pu^{240}	2.9457 - 2.9916	2.8367 - 2.8514
Pu^{241}	0.0347 - 0.1301	0.0193 - 0.0442
Pu^{242}	0.0062 - 0.0598	0.0035 - 0.0271

Table 5-7. Radiochemical Analysis Summary for Cores 40 and 41 Composites (222-S).

	Units	Core 40 comp. 1	Core 40 comp. 2	Core 41 comp. 1	Core 41 comp. 2
U^{238}	$\mu\text{g/g}$	1.82E+3	2.02E+3	2.82E+3	2.41E+4*
U^{238}	$\mu\text{Ci/g}$	6.09E-4	6.79E-4	9.48E-4	(8.09E-4)
AT	$\mu\text{Ci/g}$	8.68E-2	7.23E-2	1.81E-1	1.08E-1
TB	$\mu\text{Ci/g}$	5.03E+1	3.58E+1	5.24E+1	4.85E+1
Cs^{137}	$\mu\text{Ci/g}$	1.34E+1	1.13E+1	2.02E+1	2.47E+1
Sr^{90}	$\mu\text{Ci/g}$	9.97E00	8.25E00	1.19E+1	9.35E00
Am^{241}	$\mu\text{Ci/g}$	1.02E-2	1.40E-2	1.45E-2	1.33E-2
$Pu^{239/240}$	$\mu\text{Ci/g}$	4.40E-2	7.96E-2	5.56E-2	4.95E-2
Tc^{99}	$\mu\text{Ci/g}$	3.81E-2	2.90E-2	4.57E-2	3.49E-2
H^3	$\mu\text{Ci/g}$	3.96E-4	6.31E-2	3.93E-4	8.89E-4
C^{14}	$\mu\text{Ci/g}$	2.20E-4	3.02E-4	3.19E-4	NR

*Could be due to data transcription error.

Additional information on those methods can also be found in the reference cited in the previous section of this report.

Analysis was performed 2 months following samples receipt into the 222-S Laboratory hot cell.

Batch and result sheets were completed appropriately and can be found, along with data and calibration curves, in the tank BX-107 cores 40 and 41 data package.

The TGA results ranged from 34% to 54%, and all RPDs were less than 20%, except for core 41, segment 2. Gravimetric percent water measurements that use a larger sample size (thus more representative) were also obtained.

Both TGA and DSC scans were performed in duplicate on undigested aliquots of all recovered core segments and composites. DSC calibration frequency was determined by the administering chemist and was based on the instrument performance relative to laboratory standards. All RPDs were less than 20%.

5.5.2 Results

Reported average results are summarized in Table 5-8. No exotherms were detected in any of cores 40 and 41 segments and composites. TGA and DSC scans spanned from 25 to 450 °C.

5.6 METALLIC ELEMENTS ANALYSIS RESULTS

5.6.1 Methods

Metals were determined following three different sample treatments: (1) water extraction; (2) acid digestion (based on EPA 3050); and (3) KOH fusion.

Analyses was performed using inductively coupled plasma spectroscopy (ICP). For analytes with low ICP sensitivity, atomic absorption spectroscopy (AAS) was used. Hydride reduction method (HYAA) was used for arsenic and selenium, and cold vapor technique (CVAA) was used for mercury.

Appropriate analytical data sheets were completed and enclosed in the tank BX-107 cores 40 and 41 data package.

An acid digestion procedure was applied prior to cold vapor/hydride generation techniques and AA analysis for arsenic and selenium.

Table 5-8. TGA and % Water Results for Cores 40 and 41 Segments and Composites.

Sample	TGA Water %	% Water loss
Core 40, segment 2	44.35	54.92
Core 40, segment 4	51.3	50.8
Core 40, segment 5	52.85	NR
Core 40, segment 6	46.1	56.25
Core 40, segment 7	49.6	43.65
Core 40, composite 1	56.45	57.75
Core 40, composite 2	63.5	57.1
Core 41, segment 2	44.35	NR
Core 41, segment 3	53.3	56.85
Core 41, segment 5	37.9	27.9
Core 41,, segment 6	41.2	49.44
Core 41, segment 7	34.6	49.52
Core 41, composite 1	64.5	56.15
Core 41, composite 2	52.5	55.95

ICP analysis following acid digestion exhibited many analytical difficulties for cores 40 and 41 composites.

- Standard % recovery was not adequate for potassium, bismuth, selenium, silica, silver, sodium, calcium, magnesium, and boron (see data package, addendum 1).
- Sample and duplicate readings differed between 60 to 200%, for most analytes, for 3 of the 4 composites.
- Spike % recovery was out of specifications for aluminum, calcium, iron, sodium, phosphorus, silica, sulfur, zirconia and other elements in both core 40 and 41 composites 1. Spike % recovery was not reported for the other 2 composites.

Those inadequacies were explained as follows (Kristofzski 1993):

- Poor recoveries of iron, manganese, and calcium accompany high preparation blanks values.
- Silver recoveries are commonly low due to AgCl precipitation following addition of HCl.
- If an analytical result is below DL, the CLP report format will return a 200% RPD for duplicates and 100% for serial dilutions.
- Spike failures for major elements are frequently caused by a high element concentration in the sample; when the added spike concentration is insignificant compared to the concentration of the element present in the sample, a failure generally occurs.

ICP analysis following water extraction showed good standard % recovery values. Sample and duplicate reading, however, showed a large concentration span for elements such as zirconium, barium, calcium, sodium, and zinc.

Spike percent recovery results were better than with acid digestion, but still showed anomalies relative to chromium, sodium, phosphorus, sulfur, silica, calcium, and bismuth. No spike % recovery was reported for core 41 composite 2.

ICP analysis following sample fusion showed good standard percent recoveries (between 75 to 125%). However, sample and duplicate readings showed a large span for (1) cadmium, lead, selenium, and titanium in composite 1 of core 40; (2) cadmium, silver, and zirconium in composite 2 of core 40; and (3) silver in composite 2 of core 40. No data were reported for composite 1 of core 41.

Spike % recovery was acceptable in general, but was not reported for composite 2 of core 41. On few occasions, the sample concentration was lower than that of the preparation blank.

The low fusion concentration results exhibited by calcium are attributed to possible spectral interferences or highly insoluble precipitate/matrix formation. High nickel concentration results following fusion are attributed to sample contamination resulting from the use of nickel-impregnated crucibles.

5.6.2 Results

Table 5-9 reports metallic analytes results, in $\mu\text{g/g}$, for cores 40 and 41 composite samples.

The preparation blanks concentrations, which mostly read below instrument detection limit (IDL), were not subtracted from actual sample readings.

5.7 ANIONS ANALYSIS RESULTS

5.7.1 Methods

Water soluble ions such as fluoride, chloride, nitrate, nitrite, phosphate, and sulfate were analyzed by ion chromatography (IC). In addition, nitrite was also evaluated using WHC spectrophotometric procedure. Cyanide (CN^-) was evaluated using a micro-distillation procedure. The pH was determined using a standard pH meter. Carbonate was determined as total inorganic carbon (TIC), before the total organic carbon (TOC) analysis, by coulometry measurements of the CO_2 evolved following sample acidification.

An overview of those methods can be found in Sasaki et al. (1990), Sections 5 and 10. Further method details can be retrieved from PNL and 222-S Laboratories manuals, or the tank BX-107 cores 40 and 41 data package.

Appropriate QA/QC measures were applied. Analytical data sheets, including IC chromatograms, were included in the tank BX-107 cores 40 and 41 data package.

Standard and spike percent recovery values for those methods were all within the appropriate specifications, namely 75% to 125%. Preparation blanks concentrations were acceptable compared to the sample values recorded.

Instrument detection limit was also adequate, and generated high sample reading to detection limit ratios. Poor duplicate results were noticed in general for TIC. CN^- concentration were less than the method detection limit (MDL).

Table 5-9. Metallic Elements Results for Cores 40 and 41 Composites in $\mu\text{g/g}^*$ (222-S).

Elements	Acid/water digestion detection limit (IDL)	Fusion digest detection limit (IDL)	Preparation type	Core 40 composite 1	Core 40 composite 2	Core 41 composite 1	Core 41 composite 2
Al	3.4	17	Water	105	80.4	71.6	279
			Acid	11900	12300	16200	16600
			Fusion	13900	12000	17300	14100
Sb	35.6	178	Water	37.3	37.3	36.3	37.2
			Acid	34.1	34.9	35.2	33.8
			Fusion	183*	183*	182*	183*
As	3.1	15.5	Water	3.06	3.06	3.16	3.05
			Acid	2.8	2.86	3.03	2.77
			HYAA	0.43	0.393	0.361	0.536
			Fusion	15	15	14.9	15
Ba	0.3	1.5	Water	0.379	0.531	0.306	0.553
			Acid	6.75	6.38	10.3	9.31
			Fusion	6.07	8.07	11.8	10.6
Be	0.3	1.5	Water	0.306	0.306	0.306	0.305
			Acid	0.28	0.286	0.288	0.277
			Fusion	1.5	1.5	1.49	1.5
Cd	0.7	3.5	Water	0.713	0.713	0.713	0.711
			Acid	1.86	1.93	2.74	2.56
			Fusion	3.68	3.51	4.35	5.26
Ca	2.4	12	Water	752	714	147	121
			Acid	2140	547	1170	296
			Fusion	360	306	369	549
Cr	1.4	7	Water	157	133	156	159
			Acid	854	877	1070	1030
			Fusion	972	838	1080	983
Co	1.2	6	Water	1.02	1.02	1.22	1.09
			Acid	2.08	1.69	2.32	0.951
			Fusion	4.99	5	4.98	12.8
Cu	0.6	3	Water	0.726	0.776	0.611	0.87
			Acid	62.7	72.5	18.7	17
			Fusion	67.3	71.6	26.5	41.2
Fe	2	10	Water	54.7	42	58.4	318
			Acid	9450	9430	12900	12300
			Fusion	11000	8870	13300	11100

Table 5-9. Metallic Elements Results for Cores 40 and 41 Composites in $\mu\text{g/g}^*$ (222-S).

Elements	Acid/water digestion detection limit (IDL)	Fusion digest detection limit (IDL)	Preparation type	Core 40 composite 1	Core 40 composite 2	Core 41 composite 1	Core 41 composite 2
Pb	8.5	42.5	Water	7.23	7.23	8.66	7.77
			Acid	54.3	50.5	77.1	69.3
			Fusion	45	65.2	99.5	88.2
Mg	0.6	3	Water	16.5	34.4	6.51	8.41
			Acid	170	149	189	126
			Fusion	111	89.8	131	116
Mn	0.3	1.5	Water	1.08	1.25	0.306	1.3
			Acid	35.4	33.3	47.2	43.1
			Fusion	62.8	54.4	73.4	67.6
Ni	1.8	9	Water	1.43	1.43	1.83	1.42
			Acid	10.9	10.4	14	13.6
			Fusion	7200 ^b	1990 ^b	2860 ^b	10700 ^b
K	13.3	66.5	Water	167	137	156	130
			Acid	264	199	306	282
			Fusion	NA	NA	NA	NA
Se	13.1	65.5	Water	8.46	8.46	13.3	68.2 ^b
			Acid	312 ^b	295 ^b	369 ^b	7.66 ^b
			HYAA	1.5	1.915	2.02	0.623
			Fusion	41.4 ^b	41.5 ^b	41.3 ^b	330 ^b
Ag	1	5	Water	1.02	1.02	1.02	1.02
			Acid	0.932	0.952	0.961	0.923
			Fusion	4.99	7.02	6.29	5.46
Na	8.7	43.5	Water	53300	67000	80100	72000
			Acid	100000	102000	103000	100000
			Fusion	104000	101000	102000	95000
Zn	2.5	12.5	Water	25.6	23.8	2.55	5.12
			Acid	56.1	35.5	81	65.5
			Fusion	75.3	48.1	88.2	122
Bi	8.5	42.5	Water	106	80.2	113	635
			Acid	21200	22100	27100	26200
			Fusion	21900	20600	28100	18600
B	1.9	9.5	Water	27.9	28.7	20.7	22.8
			Acid	49.8	62.3	50.8	25.3
			Fusion	18.4	18.8	6.65	16.2

Table 5-9. Metallic Elements Results for Cores 40 and 41 Composites in $\mu\text{g/g}^*$ (222-S).

Elements	Acid/water digestion detection limit (IDL)	Fusion digest detection limit (IDL)	Preparation type	Core 40 composite 1	Core 40 composite 2	Core 41 composite 1	Core 41 composite 2
Ce	8.6	43	Water	9.99	9.99	8.76	9.96
			Acid	151	169	191	203
			Fusion	92.1	178	201	229
La	2.1	10.5	Water	1.63	1.63	2.14	1.63
			Acid	1.49	1.52	1.54	1.48
			Fusion	7.98	7.99	7.97	7.99
P	8	40	Water	4750	5000	4600	4810
			Acid	19700	21300	23900	27200
			Fusion	24200	20800	27400	21500
Si	2.3	11.5	Water	414	364	136	262
			Acid	1540	1720	1740	1660
			Fusion	6600	6000	7990	6540
Sr	0.6	3	Water	1.7	1.66	1.46	5
			Acid	154	157	185	170
			Fusion	171	160	188	155
S	6	30	Water	4560	4630	4390	4150
			Acid	4760	5000	4470	4030
			Fusion	4830	4650	4150	4130
Sn	1.6	8	Water	2.34	2.34	1.63	2.34
			Acid	3.28	6.3	7.19	11.2
			Fusion	11.5	11.5	11.5	11.5
Ti	0.5	2.5	Water	0.408	0.408	0.51	0.406
			Acid	3.18	3.59	4.59	4.06
			Fusion	3.25	5.8	7.27	9.05
Zr	1.1	5	Water	1.54	1.11	1.21	6.09
			Acid	94.6	92.2	87.9	110
			Fusion	189	66.2	121	166
Hg	NR	NR	CVAA	0.611	0.375	0.631	0.642

*Biased due to interference. Fusion is not suitable for most trace metals because of larger dilution required.

^bNot valid results due to method contamination (nickel crucibles).

*Wet basis.

5.7.2 Results

Table 5-10 summarizes average anion results in $\mu\text{g/g}$.

5.8 ORGANIC ANALYSIS RESULTS

5.8.1 Methods

Total organic carbon (TOC), total extractable organic halides (TOX/EOX), semi-volatile and volatile organic analysis (SVOA, VOA) were applied to tank BX-107 cores 40 and 41 composite samples. VOA were also performed on core 41, segments 2, 3, 5, 6, and 7, and core 41, segments 5 and 7.

TOC is a chemical oxidation method to determine the concentration of water soluble and water insoluble organics in the sample. Unusually high concentrations in the water soluble fraction could be an indication of the presence of organic complexing agents such as EDTA, HEDTA, acetate and citrate. Organic halides are measured as follows.

1. Sample pH is adjusted to near neutral prior to extraction.
2. Organic halides present in the sample are extracted using ultrasonic mixing into an ethyl acetate solution.
3. Extract is injected into a total organic halides (TOX) analyzer where the sample is combusted and the halides, except fluoride, are measured coulometrically.

Semi-volatile organic compounds are analyzed by gas chromatography/mass spectroscopy (GC/MS) EPA method SW-846-8270 following a methylene chloride sonication extraction procedure.

Volatile organics are analyzed using EPA SW-846-5040, which consists of a thermal desorption followed by a purge and trap methodology.

For VOA, samples were received on October 27, 1992, after four months in the 222-S Laboratory hot cell. Analysis was performed two days from sample receipt by PNL laboratory personnel.

Analytical data sheets were completed appropriately and were included, along with chromatograms, into the tank BX-107 cores 40 and 41 data package.

The NPH contamination cleanup procedure using methanol was performed on sample aliquots. For VOA, the dilution factor was approximately 625.

Table 5-10. Anion Results for Cores 40 and 41 Composites in $\mu\text{g/g}^*$ (222-S).

Specie	Method	IDL	Prep.BLK	Core 40 comp. 1	Core 40 comp. 2	Core 41 comp. 1	Core 41 comp. 2
F ⁻	IC	1.00E1	<1.00E1	8.51E3	8.42E3	8.88E3	1.09E4
Cl ⁻	IC	2.36E1	2.36E1	1.16E3	1.18E3	1.06E3	1.18E3
NO ₃	IC	1.86E3	1.86E3	1.42E5	1.5E5	1.28E5	1.30E5
NO ₂	IC	1.00E2	<100	9.68E3	9.16E3	1.25E4	1.20E3*
NO ₂	Spec.	5.11E3	<5.11E3	1.32E4	1.03E4	1.33E4	1.25E4
PO ₄ ⁻³	IC	1.00E2	<100	1.44E4	1.5E4	1.34E4	1.42E4
SO ₄ ⁻²	IC	1.00E2	<100	1.41E4	1.42E4	1.30E4	1.23E4
pH	pH meter	NA	NA	9.63	9.68	9.82	9.72
NH ₃	Dist.	4.50E3	<4.5E3	<4.5E3	<5.5E2	<4.5E3	<4.5E3
CN	Dist.	0.1-2	0.02-<2	3.11	1.43	2.99	2.43
Carbon	TIC	5.00E2	3.4E-1	1.15E3	1.15E3	1.24E3	1.10E3
CO ₃ ⁻	Calculated from TIC	NA	NA	5745	5733	6218	5500

*This could be attributed to a calculation error.

*Wet Basis.

Semi-VOAs were extracted on November 11, 1992, and analyzed on December 12, 1992. No dilutions were necessary for semi-VOAs. However, it is unclear whether the results reported were per kg of waste material. High quantification/detection limits were reported for both methods (VOA and SVOA) and attributed to method deficiencies.

TOX spike recovery was low but accepted by the administering chemist and attributed to matrix interferences.

5.8.2 Results

Table 5-11 summarizes TOC results in $\mu\text{g/g}$ for cores 40 and 41 composites.

No volatile EPA target compounds in concentration above the Contract Required Quantification Limits (CRQLs) were observed in the blank or core samples. However, kerosene constituents, such as decane, undecane, dodecane, and tridecane were observed. Table 5-12 summarizes averages of those results. Detailed GC/MS reports, including sample and duplicate results, can be found in the tank BX-107 cores 40 and 41 data package.

Water-soluble organic halides contents were $< 10 \mu\text{g/g}$ for sample and blanks, based on the total wet weight of the sample in core 40. Core 41 EOXS were below MDL.

Semi-volatile organic analysis showed traces, mostly below IDL, of several organic compounds. Those results were tabulated and included in Addendum 4, Appendix E of the data package. Tables 5-13 and 5-14 list average results for compounds detected in relatively considerable amounts.

5.9 OVERALL ANALYTICAL DATA SUMMARY (DATA PRESENTATION)

Table 5-15 compiles average species concentrations as reported for the 1979 and 1992 core samples in parallel with calculated TRAC and process history data. In addition, a tank contents bulk inventory, calculated based on the 1992 analytical data, is presented. Process History, TRAC, and 1979 core data were appropriately converted to μg per gram of wet residue.

5.10 DATA QUALITY - CORES 40 AND 41

5.10.1 Data Validation

Tank BX-107 cores 40 and 41 analytical results validation was performed by Los Alamos Technical Associates, according to standard procedures described in Kersner (1990),

Table 5-11. Total Organic Carbon Results for Cores 40 and 41 Composites in $\mu\text{g/g}$ (222-S).

Sample	Preparation blank	Total organic carbon (TOC)
Core 40, composite 1	<5.50E2	7.00E2
Core 40, composite 2	<5.50E2	<5.50E2
Core 41, composite 1	<5.50E2	<5.50E2
Core 41, composite 2	<5.50E2	8.97E2

Table 5-12. Volatile Organic Results for Cores 40 and 41 Segments in $\mu\text{g/g}$ (PNL).

Volatile organic compound	Core 40 seg.7	Core 40 seg.5	Core 41 seg.2	Core 41 seg.3	Core 41 Seg.5	Core 41 Seg.6	Core 41 Seg.7
Decane	2.2	2.05	15.5	2.25	6.95	NR	NR
Undecane	2.0	2.35	2.05	3.35	5.1	NR	NR
Dodecane	23.0	26.5	29.0	41.5	70.0	14.5	30.0
Tridecane	10.25	11.55	20.5	31.5	58.3	15.5	40.0
Tetradecane	2.15	2.05	4.4	9.95	19.0	5.9	12.5

NOTE: The presence of those compounds is attributed to sample contamination with NPH.

Table 5-13. Semi-Volatile Organics Results for Core 40 Composites in $\mu\text{g/g}$ (PNL).

Compound	Composite 1	Composite 2
Undecane	10.05	11.5
Dodecane	550	640
Tridecane	1,600	1,750
Tetradecane	1,200	1,250
Pentadecane	38.5	45.5
Tributylphosphate	8.2	9.4

NOTE: The presence of those compounds (except tributylphosphate) is attributed to sample contamination with NPH.

Table 5-14. Semi-Volatile Organics Results for Core 41 Composites in $\mu\text{g/g}$ (PNL).

Compound	Composite 1	Composite 2
Undecane	14.5	13
Dodecane	640	570
Tridecane	1,450	1,350
Tetradecane	1,150	1,050
Pentadecane	53	49
Tributylphosphate	6.85	3.75

NOTE: The presence of those compounds (except tributylphosphate) is attributed to sample contamination with NPH.

Section 2.0 to 2.2. Quality assurance procedures, described in Winters et al. (1990), Appendix D, were also applied.

The main objective of the data validation effort is to assess the usability and defensibility of the data generated. As mentioned in the narrative section of the tank BX-107 cores 40 and 41 data package, this was accomplished through a detailed examination of the data package to recreate the analytical process and verify that proper and acceptable analytical techniques were applied. The data package was checked for correct submission of required deliverables, correct data transcriptions from the raw data to the summary forms, and the proper calculation of various parameters.

Data summary tables including data qualifiers depicting data usability status (unusable, estimate) were consecutively generated. The following lists major factors used in the data qualification process.

- Chain-of-custody records
- Holding times
- Instrument calibration
- Calibration verifications
- Analytical blanks
- Preparation blanks
- Interference check sample
- Laboratory control sample
- Duplicate analysis
- Matrix spike or post digestion spike
- Retention time
- Contract required detection limit standard

Table 5-15. Overall Data Summary and Inventory Estimates (Wet Basis).

Group	Mol. wt.	Process history	TRAC	1979 core total	1992 cores 40 and 41 average total	Tank inventory based on 1992 core data
Metals		$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	Kg
Be	9	NR	NR	NR	1.5	2.82
B	11	NR	NR	NR	47.05	88.47
Na	23	64717	46571	110000	102000	191800
Mg	24	4655	NR	NR	158.5	298
Al	27	444	1359	16400	14300	26890
Si	28	NR	290	1270	6783	12755
P	31	NR	NR	NR	23480	44152
S	32	NR	NR	NR	4440	8349
K	39	NR	NR	NR	263	495
Ca	40	NR	5846	NR	1040	1956
Ti	48	NR	NR	NR	6.34	11.93
V	51	NR	NR	NR	9.14	17.19
Cr	52	72	25	74	968	1820
Mn	55	NR	188	52.7	64.56	121.39
Fe	56	772	6720	14300	11100	20872
Ni	59	NR	NR	NA	12.22	22.97
Co	59	NR	230	NR	7	13.16
Cu	64	NR	NR	NR	51.6	97.03
Zn	65	NR	NR	NR	83.3	156.64
As	75	NR	NR	NR	0.43	0.81
Se	79	NR	NR	NR	1.52	2.86
Sr	89	NR	9	NR	169	318
Zr	91	126	941	NR	136	256
Ag	108	NR	NR	NR	6.25	11.75
Cd	112	NR	NR	15.3	4.25	7.99
Sn	119	NR	NR	NR	11.5	21.62

Table 5-15. Overall Data Summary and Inventory Estimates (Wet Basis).

Group	Mol. wt.	Process history	TRAC	1979 core total	1992 cores 40 and 41 average total	Tank inventory based on 1992 core data
Sb	122	NR	NR	NR	183	344
Ba	137	NR	NR	NR	9	16.92
La	139	NR	NR	438	7.99	15.02
Ce	140	NR	41	NR	175	329
Hg	201	NR	NR	NA	0.6	1.13
Pb	207	NR	2017	20	76	143
Bi	209	11531	7351	16100	22300	41933
Total metals inventory excluding phosphorus and sulfur (including uranium)				165020	162240	305076
Radionuclides		$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	KCi
AT		NR	NR	NR	0.0963	0.1811
Am 241		NR	1.51E-3		0.013	0.0244
Pu 239/240		6.7E-7	2.76E-2	0.1027	0.0572	0.1076
U 238		6.27E-4	4.02E-4	5.64E-5	7.467E-4	0.0014
TB		NR	NR	NR	46.7	87.81
Cs 137		NR	9.7437	11.11	17.4	32.72
Tc 99		NR	2.51E-3	NR	0.0369	0.0693
Sr 90		NR	25.13	14.2	9.87	18.56
C 14		NR	9.744E-5	NR	2.61E-4	4.91E-4
H 3		NR	NR	NR	3.77E-4	7.09E-4
Total radionuclides inventory				25.41	46.8	~ 88
Anions		$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	Kgs
OH-	17	2345	5159	NR	NR	NR
NH ₄ ⁺	17	856	NR	NR	<4.5E-3	NR
CN ⁻	26	NR	NR	NR	2.49	4.6171
F-	19	1703	957	1000	9190	17281

Table 5-15. Overall Data Summary and Inventory Estimates (Wet Basis).

Group	Mol. wt.	Process history	TRAC	1979 core total	1992 cores 40 and 41 average total	Tank inventory based on 1992 core data
Cl ⁻	35	0	0	NR	1140	2144
NO ₂ ⁻	46	19352	1872	NR	8120	15269
NO ₃ ⁻	62	83379	31385	153000	137000	257613
CO ₃ ⁻²	60	5793	24290	1000	5799	10911
PO ₄ ⁻³	95	14414	14414	87000	12611	136537
SO ₄ ⁻²	96	5959	NR	9300	13400	25197
Total anions inventory (water soluble)				173000	247072	464409
Total metals and anions inventory				338020	351092	660190
pH		>9.5	NR	NR	9.71	NA
Total organic carbon				7.3	675	1252
Inorganic chelators		µg/g	µg/g	µg/g	µg/g	Kgs
EDTA		NR	2.9231	NR	NR	NR
HEDTA		NR	4.8719	NR	NR	NR
Physical properties						Units
Total solids (dry basis)				45	44.13	%
Water				NR	56	%
Undissolved solids				NR	36.73	%
Bulk density				1.45	1.44	g/cm ³
Particle density				2.25	NR	g/cm ³
Supernatant density				1.238	1.19	g/cm ³
Particle size distribution ± 20 %						Number
0.5 - 2 microns					90	%
2 - 5 microns					9	%
5 -150 microns					1	%

NOTE: Appendix A contains a detailed summary including calculated statistical values and inventory of the 1992 cores 40 and 41 data.

-
- Serial dilution
 - Surrogate recoveries.

A validation summary for cores 40 and 41 analytical results can be found in Addendum 1 of the data package.

Following those steps, most reported data was qualified as an estimate (UJ or J), with some being classified as unreliable (R). The following lists some of the flaws, according to the data validation report.

- Typical ICP analysis problems, such as elemental and matrix interferences, were encountered. Preparation blank contaminations were reported. Some analysis also lacked calibration records.
- Total alpha results were qualified as unreliable because of the lack of a matrix spike (was not required when analysis was performed).
- Americium-241 data was classified unreliable because no energy counts were found for tracer recoveries below the program goal of 50%.
- 222-S Laboratory plutonium analysis reports were missing some documentation, thus results were classified as estimates.
- Iodine results were classified unreliable because of poor carrier recovery.
- Carbon-14 results were classified as estimates because duplicate RPDs exceeded acceptance criteria.
- 222-S Laboratory uranium analysis results were also classified as estimates because of poor precision and missing documentation for internal standard traceability.

In general, for most analyses the analytical chemist was not required to perform additional reruns if the original quality control failure remains after the first rerun.

Discrepancies in the data package results included incorrect reports of exotherms for segment 6 of core 40. DSC scans were also missing for core 41 segments.

5.10.2 Field and Hot Cell Blanks

Field and 222-S Laboratory hot cell blanks* were omitted from the initial shipment to the PNL Laboratory. Those blanks were prepared and analyzed separately from the tank BX-107 cores 40 and 41 samples for volatile and semi-volatile organic compounds.

Acetone was the only volatile compound observed above the CRQL. Phthalates were also detected in the semi-volatile analysis. However, it was determined that the presence of those compounds was because of contaminations during method preparation. Details of those analyses can be found in Addendum 6 of the tank BX-107 cores 40 and 41 data package (Kristofzski 1993).

5.10.3 TCLP Extraction

Core 40, composite 1, underwent TCLP extraction in the 222-S Laboratory. Toxic Characteristics Leaching Procedure (TCLP) as defined by EPA, is a 20:1 extraction procedure using a sodium acetate buffer. The 222-S Laboratory procedure, however, was scaled down by a factor of 10 (for radioactive samples). The pH was also adjusted to 2 to test the loss of metals by absorption and/or precipitation in the sample container caused by matrices with pH greater than 5 cm (2 in.) (Kristofzski 1993). Table 5-16 is a summary of the TCLP results prior to acidification.

Detailed results can be found in the data summary section of the tank BX-107 data package. Because of the historical affinity of hydroxy-carboxylic acids towards metallic ions, by selectively forming stable chelates (Raphael 1989), and because only a selected number of elements were analyzed thus minimizing the relevancy in understanding the waste matrix, those results will not be assessed in this report.

Table 5-16. TCLP Results for Core 40, Composite 1.

Contaminant metal	Dangerous waste number	Concentration mg/L
Ag	D011	<0.01
Ba	D005	0.102
Co	D006	<0.07
Cr	D007	7.11
Pb	D008	0.71
As	D004	2.04
Se	D010	1.63
Hg	D009	0.0258

*Hot cell blank consists of deionized water samples contacted with the extraction tray.

6.0 ANALYTICAL DATA INTERPRETATION THROUGH 1992

This section interprets up-to-date analytical data (presented in Section 5.0) relative to tank BX-107 contents.

6.1 RESIDUES PHYSICAL PROFILE

Physical properties represent a key assessment and design factor. These characteristics have been compiled in tabulated formats. Tables 6-1, 6-2, and 6-3 detail the physical data reported.

Most physical data assessments were performed on segments 2, 4, and 6 of core 40. Settling rate curves are included in Appendix A. Actual viscosity values were not reported. Table 6-4 summarizes that information. In general, 1992 and 1979 core data agree with the values reported for the physical properties.

Particle size analysis was performed on 1992 core segments. Number and volume distribution agreed, in general, between core 40 and core 41 segments (refer to Table 5-4). Section 6.7 will address particle size spatial distribution on segment and core levels. Table 6-5 is an overall number and volume based summary of particle size distribution.

Note that the wide % distribution range exhibited in the volume distribution for particles between 2 to 150 μm is mostly related to the size of the sample used (0.1 grams or less) whereby larger particles can be easily lost or broken into smaller particles. Data reveals that, in general, 99% of the particles are between 0.5 to 5 μm while approximately 77% of the volume is occupied by particles from 5 to 150 μm . Table 6-6 is an overall summary of the data.

6.2 RADIOCHEMICAL CHARACTERISTICS

6.2.1 1992 Data Interpretation

A table that compares analytical ranges on a core composite level is included in Appendix A. Table 6-7 presents analytical ranges on a core composite level basis. Table 6-8 summarizes the average and range of radionuclides concentrations.

Figure 6-2 is a graphical representations of the average data presented in Table 6-8. It should be noted that AT is higher than the sum reported for plutonium and americium.

Table 6-1. Residue Volumetric Breakdown in Gallon (June 1993).

Sludge (including interstitial liquid)	Interstitial liquid	Supernatant liquid	Ratio sludge/interstitial/supernatant
3.44E5	0.29E5	0.01E5	315/29/1

Table 6-2. Physical Measurements Summary (1992).

Temperature	Pressure	Residues level (sidewall bottom)
July 1993	NR	April 1993
66 °F	atmospheric	~ (120.7 in.)
21 °C	atmospheric	~ 3.07 m

Table 6-3 Sludge Macro-Physical Characteristics (1992).

Color	Grey mostly with white and green streaks
Texture	Light mostly with shiny and crystalline streaks
Consistency	Creamy granular with some adhesive and cohesive characteristics

Table 6-4. Tank Contents Physical Characteristics Summary Based on Core 40 Segments (1992).

Physical characteristic	Average value	Units
Total settled solids	99.3	Volume %
Settling time (to account for 90% of the sample by volume)	2	Hours
Approximate time asymptotic settling reached	10	Hours
Total centrifuged solids (1,000 x gravity for 1 hour)	71.2	Volume %
	76	Weight %
Bulk density	1.44	g/cm ³
Particle density (1979) (density of dry particles)	2.25	g/cm ³
Density of centrifuged supernate	1.19	g/cm ³
Solids (dry)	44.13	Weight %
Solids range	44 - 46	Weight %
Undissolved solids (dry)	36.73	Weight %
Shear strength	5533	Dynes/cm ²

NOTE: The difference between settled and centrifuged solids can provide an estimate of the very light particles, soluble or colloidal chelate type, which are present in tank BX-107 residues.

NOTE: The percent water content is above the postulated safety limit of 20%.

Table 6-5. Particle Size Summary in Percent (1992).

Particle size intervals (microns)	Number distribution range	Number distribution mean	Volume distribution range	Volume distribution mean
0.5 - 1	58.97 - 77.07	66.59	0.23 - 2.91	1.65
1 - 1.5	15.11 - 21.01	17.65	0.38 - 2.97	1.94
1.5 - 2	3.15 - 7.48	5.54	0.36 - 2.78	1.65
2 - 5	3.76 - 13.2	8.97	3.4 - 34.18	21.32
5 - 10	0.45 - 2.05	1.13	2.98 - 26.49	15.64
10 - 20	0.06 - 0.21	0.1	3.54 - 23.15	12.06
20 - 150	0.01 - 0.08	0.03	17.06 - 89.11	48.8

NOTE: Figure 6-1 is a reflection of the data in Table 6-6.

Figure 6-1. Particle Size Distribution Summary.

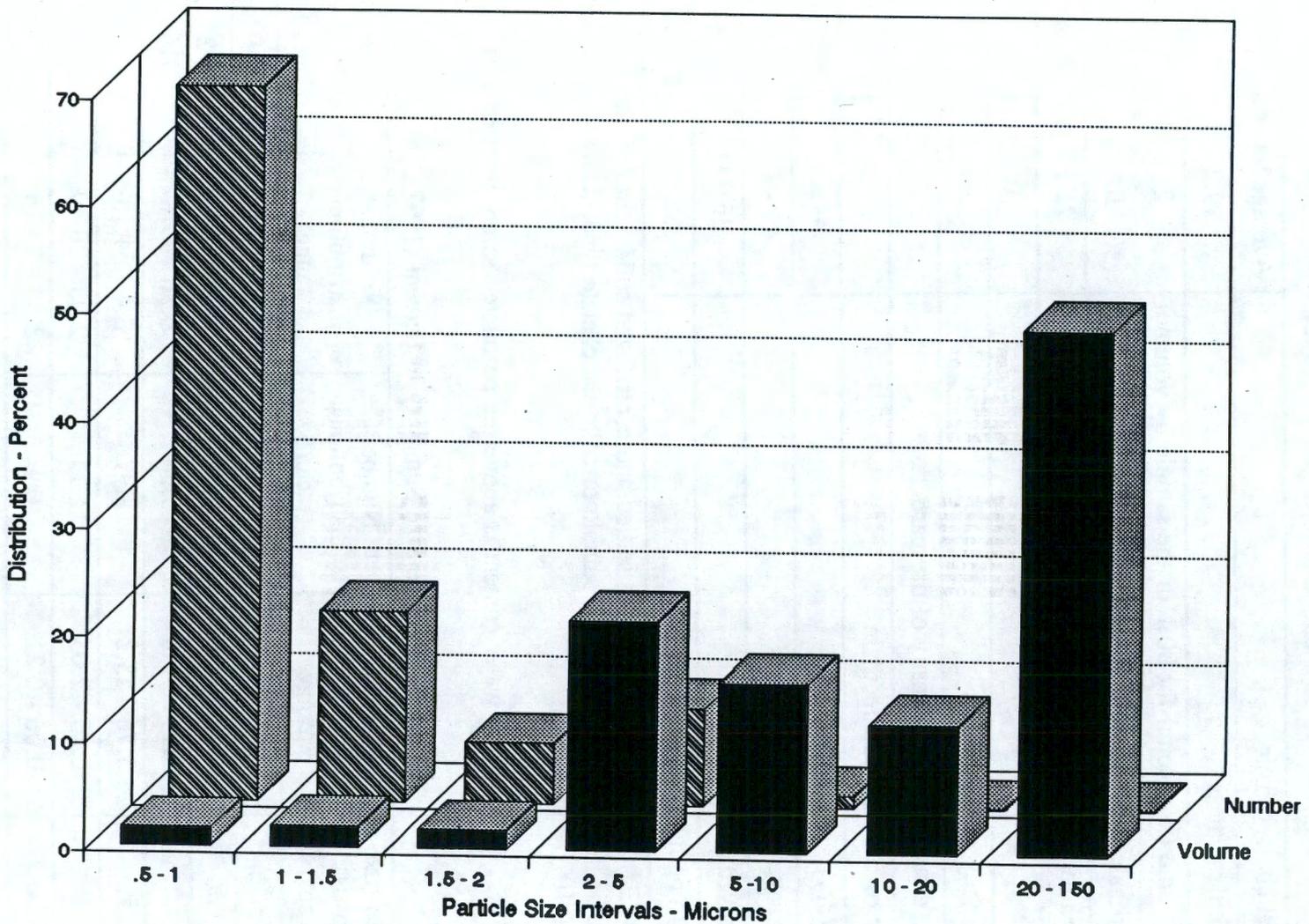


Table 6-6. Overall Summary of Particle Size Distribution in Percent (1992).

Particle size interval microns	Approximate average number distribution ($\pm 2\%$)	Approximate number distribution range ($\pm 20\%$)	Approximate average volume distribution ($\pm 2\%$)	Approximate volume distribution range ($\pm 20\%$)
0.5 - 2	90	77 - 100	5	1 - 9
2 - 5	9	3 - 13	21	3 - 34
5 - 150	1	0.5 - 5	76	24 - 100

Table 6-7. Radionuclides Analytical Ranges for Cores 40 and 41 Composites (1992).

Radionuclide	Core 40 analytical range ($\mu\text{Ci/g}$)	Core 41 analytical range ($\mu\text{Ci/g}$)
U^{238}	6.1E-4 - 6.8E-4	9.5E-4
U^{238} $\mu\text{g/g}$	1.56E+3 - 2.18E+3	2.82E+3 - 2.29E+4*
$\text{Pu}^{239/240}$	3.49E-2 - 7.17E-2	4.49E-2 - 5.64E-2
Am^{241}	8.05E-3 - 1.38E-2	1.28E-2 - 1.62E-2
AT	7.17E-2 - 9.47E-2	1.01E-1 - 1.23E-1
TB	3.28E+1 - 5.03E+1	4.58E+1 - 5.36E+1
Cs^{137}	1.09E+1 - 1.35E+1	2.00E+1 - 2.51E+1
Sr^{90}	7.71 - 10.2	8.74 - 12
Tc^{99}	2.83E-2 - 4.06E-2	3.24E-2 - 4.92E-2
C^{14}	1.33E-4 - 4.21E-4	2.24E-4 - 3.49E-4
H^3	3.89E-4 - 7.99E-4	3.66E-4 - 1.08E-3

*The upper range, an analytical value, reported for U^{238} in $\mu\text{g/g}$ is most likely a reporting error, with the actual value being $2.29 \text{ E}+ 3$.

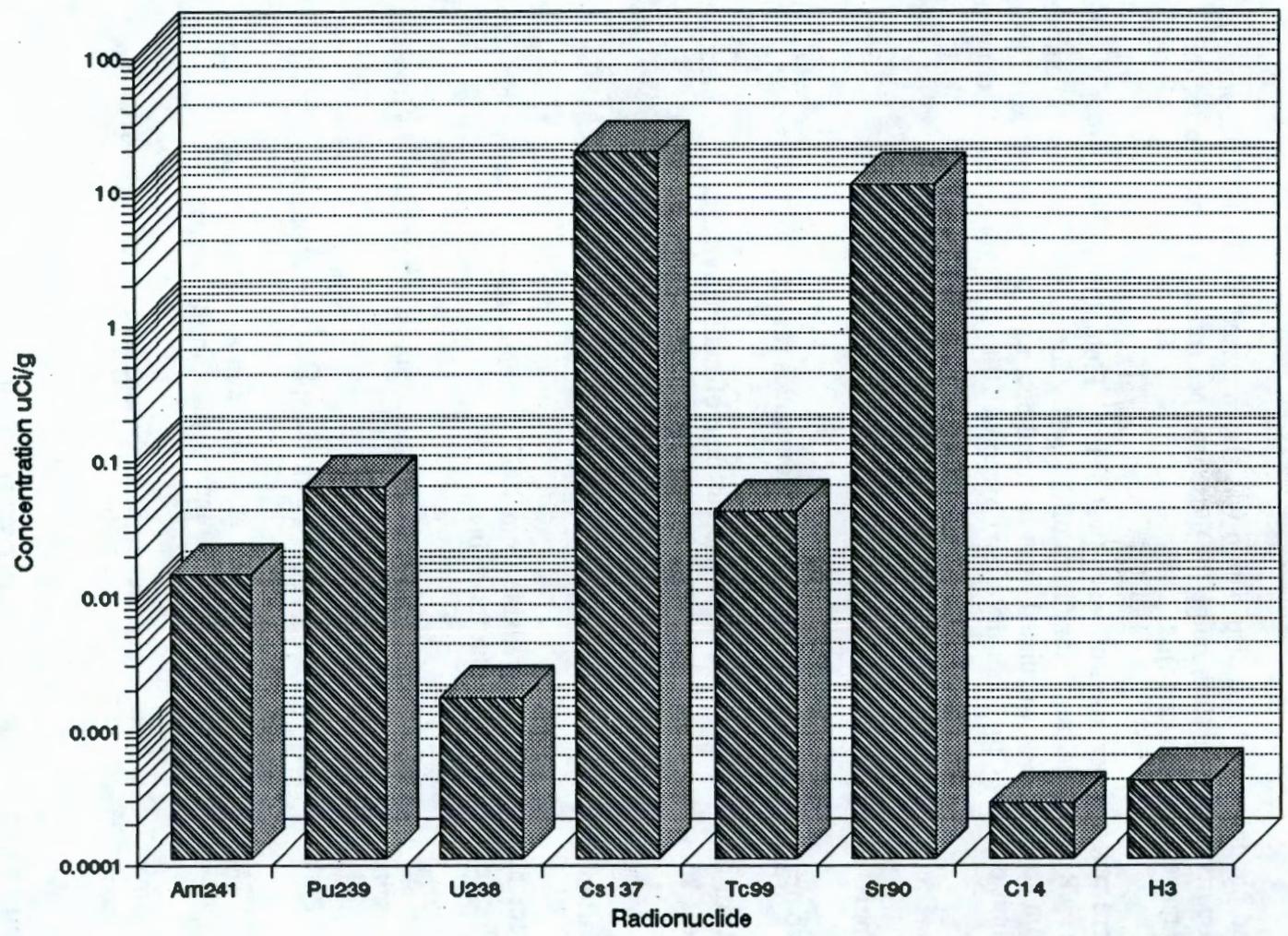
Table 6-8. Radionuclides Profile Summary (1992).

Radionuclides	Cumulative average ($\mu\text{Ci/g}$)	Analytical range ($\mu\text{Ci/g}$)
U^{238}	7.467E-4	N/A
U^{238} $\mu\text{g/g}$	2E+3	2.29E+4 - 2.82E+3
$\text{Pu}^{239/240}$	5.72E-2	3.49E-2 - 8.34E-2
Am^{241}	1.30E-2	8.05E-3 - 1.62E-2
AT	9.63E-2	1.01E-1 - 9.47E-2
TB	4.67E+1	3.28E+1 - 5.36E+1
Cs^{137}	1.74E+1	1.09E+1 - 2.51E+1
Sr^{90}	9.87	7.71 - 12
Tc^{99}	3.69E-2	2.83E-2 - 4.92E-2
C^{14}	2.61E-4	1.33E-4 - 4.21E-4
H^3	3.77E-4	3.66E-4 - 1.08E-3

953322.1383

WHC-EP-0739

Figure 6-2. Radionuclides Profile - 1992 Data.



TB results were also higher than the sum of cesium and strontium concentration. Those issues were addressed in Section 5.4.1. Other radioactive elements usually contribute to the alpha and beta spectrums with varying degrees of activities.

Uranium and plutonium isotopic distribution was also reported by the PNL laboratory. Table 6-9 reports a concentration range for those isotopes (refer to Table 5-6 for the distribution range values reported).

All results are reported per gram of waste (wet basis). Plutonium concentration slightly exceeded the TRU disposal regulatory limit of $0.1 \mu\text{Ci/g}$ for core 41 samples. Total cesium plus strontium concentration calculated from the total beta reported value exceeded the postulated limit of $100 \mu\text{Ci/g}$ (on a dry basis) by about 10 to 20% for cores 40, composite 1, and core 41.

6.2.2 Comparison with 1979 Core Data

Table 6-10 compares 1979 and 1992 average radionuclides results.

Data, except for uranium, corresponds to the outcome of radioactive decay. No assessment against process knowledge or TRAC estimates will be made at this point.

6.2.3 Speciation of Radionuclides

No soluble radionuclides data is available from 1992. Chemical knowledge combined with 1979 data interpretation indicates the following.

- Cesium is most likely present as a combination of phosphates, hydroxides, and salts.
- Plutonium is most likely present as a phosphate (oxyphosphate).
- Uranium is most likely present as an oxide in a phosphate matrix. Some uranium fluorides could be also incorporated in that matrix.
- Strontium is most likely present as a phosphate or an oxide-hydroxide combination.

6.3 INTERPRETATION OF TGA AND DSC RESULTS

As discussed in Section 5.5, no DSC exotherms were reported for any of cores 40 and 41 segments. However, major endotherms were detected at approximately 100°C and are

Table 6-9. Isotopic Distribution of Plutonium and Uranium. (from PNL Data 1992)

Isotope	Units	Concentration range
U ²³⁴	μg/g	0.102 - 0.154
U ²³⁵	μg/g	13.476 - 14.006
U ²³⁶	μg/g	0.108 - 2.06
U ²³⁸	μg/g	1986.07 - 1986.3
Pu ²³⁸	μCi/g	2.059E-6 - 2.46E-6
Pu ²³⁹	μCi/g	0.055 - 0.56
Pu ²⁴⁰	μCi/g	0.0016 - 0.0017
Pu ²⁴¹	μCi/g	1.104E-5 - 7.442E-5
Pu ²⁴²	μCi/g	2.002E-6 - 1.55E-5

Table 6-10. Radionuclides Data Comparison in μCi/g, 1979 and 1992.

Radioactive element	1979 Core data*	1992 Core data
U ²³⁸	5.64E-5	7.467E-4
Pu ^{239/240}	1.027E-1	5.72E-2
Am ²⁴¹	NR	1.30E-2
Cs ¹³⁷	11.11	17.4
Sr ⁹⁰	14.2	9.87

*Not compensated for radioactive decay.

attributed to free and hydrate water loss by forced evaporation from the sample. Other small endotherms were also noticed at higher temperatures (200 to 280 °C), and are attributed to bound water loss (possible from complexed aluminosilicates). These facts indicate that no excess activation energy is building within tank BX-107 residues (thus no potentially reactive components).

6.4 METALS AND ELECTROLYTES PROFILE

6.4.1 Interpretation of 1992 Data

In interpreting metallic element and anion data, the focus will be on key analytes, as well as analytes reported in major concentrations.

In general, fusion digestion showed higher concentration values than acid digestion for all elements except for groups IA and IIA metals, like calcium and magnesium. Potassium was not reported for the fusion digestion because of method contamination.

In this study, water extracted and fusion digested concentrations are compared for major analytes, except for the elements mentioned in the previous paragraph (acid digestion values are used instead). Water extract results are labeled "water soluble," while fusion digestion concentrations are labeled "total matrix concentration" or "totals." Anion concentrations were reported following a water extraction scheme, and thus are also labeled "water soluble."

Table 6-11 is a bulk summary. Section 6.7 contains tabulated information relative to core level comparison. It is important to note that metallic ions were evaluated by ICP, and thus the total values reported do not necessarily represent ionic bonds. The valence reported represent the most thermodynamically stable state. Figure 6-3 is a graphical representation of this data. Note that total phosphates and sulfates were based on phosphorus and sulfur total matrix concentrations. Table 6-12 compares those concentrations on a mole/L basis. Metallic ions in that table are listed by descending electropositivity (equivalent to corrosion potential).

Soluble cations balance (based on normality) in Table 6-12 can range from activated metal ions (in oxide or salt form, like cesium salts) to ammonia. Insoluble anions, on the other hand, are mostly attributed to unaccounted for hydroxides. Thus tank BX-107 inorganic residues consist of a thermodynamic equilibrium state of salts and hydroxides.* Section 6.4.3 will further elaborate on this subject.

*A charge balance was reported in the BX-107 Cores 40 and 41 data package. ICP Acid results were used to evaluate cations and Ion chromatography results to estimate anions in g/meq. SiO_3^- and TOC (as acetate) were used as contributors to the anionic charge. A copy is enclosed in Appendix A.

Table 6-11. Water Soluble and Total Contents Average
and Range Summary in $\mu\text{g/g}$. (1992)

Electrolytes	Water soluble	Water soluble analytical range	Totals	Totals analytical range
Al^{+3}	134	92 - 175	14300	12950 - 15700
Ca^{+2}	434	134 - 733	1040	733 - 1344
Cr^{+3}	152	145 - 158	968	965 - 1032
Fe^{+2}	118	48 - 188	11100	9935 - 12200
K^{+}	147	143 - 152	263	232 - 294
Na^{+}	68100	60000 - 70000	102000	98500 - 102500
Bi^{+3}	234	93 - 3.74	22300	21250 - 23350
Ce^{+3}	9.7	9 - 10	175	135 - 215
P^{+5}	4790	4700 - 4800	23480	22500 - 24450
Si^{+4}	294	199 - 389	6783	6300 - 7265
Sr^{+3}	2.46	1.6 - 3.2	169	166 - 172
S^{+6}	4430	4200 - 4500	4440	4140 - 4740
Zr^{+4}	2.49	1.3 - 3.7	136	128 - 144
F^{-}	9190	8285 - 9890	NA	NA
Cl^{-}	1140	1170 - 1120	NA	NA
NO_3^{-}	137000	129000 - 146000	NA	NA
NO_2^{-}	8120	9420 - 12250	NA	NA
PO_4^{-3}	14200	13800 - 14700	71955*	NA
SO_4^{-2}	13400	12650 - 14150	~13400	NA
CO_3^{-2}	1160	1150 - 1170	NA	NA
pH	9.71	9.66 - 9.77	NA	NA

*Based on total phosphorus, thus assuming that all phosphorus is present as phosphate.

Figure 6-3. Water Soluble and Totals Comparison for 1992 Inorganic Data.

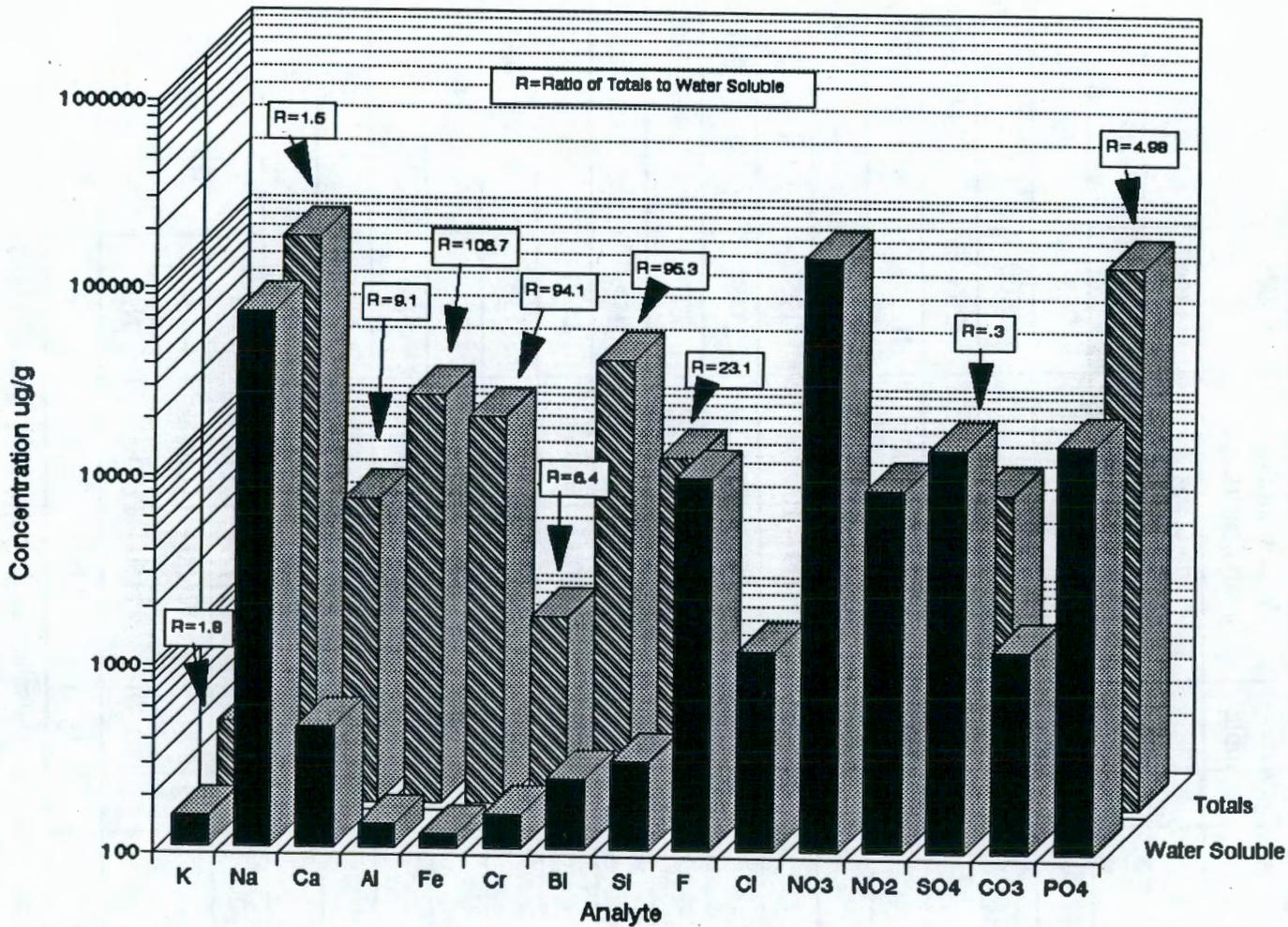


Table 6-12. Comparison of Water Soluble and Water Insoluble Electrolytes Concentrations in moles/liter. (1992 data)

Electrolyte	Water soluble matrix	Insoluble matrix
K ⁺	0.00543	0.00971
Ca ⁺⁺	0.01562	0.03744
Na ⁺	4.26365	6.38609
Al ⁺³	0.00715	0.76267
Fe ⁺²	0.00303	0.28543
Cr ⁺³	0.00421	0.02681
Bi ⁺³	0.00161	0.15365
Si ⁺⁴	0.01512	0.34884
F ⁻	0.69651	NR
Cl ⁻	0.04560	NR
NO ₂ ⁻	0.25419	NR
NO ₃ ⁻	3.18194	NR
SO ₄ ⁻²	0.25419	(~ 2-5% of soluble value)
CO ₃ ⁻²	0.13918	NR
PO ₄ ⁻³	0.21524	(calc.) 1.09068
Total	9.04948	9.10131
Total cations	4.31583	8.01063
Total anions	4.73365	~ 1.09068
Balance, based on normality	0.13356 Cations	6.59525 Anions

Note that 1979 data reported water soluble mercury at 0.00925 moles/L, thus much higher than the 1992 data reported by cold vapor atomic absorption.

6.4.2 Comparison with 1979 Core Data

Core data from 1979 for metallic elements and electrolytes was appropriately converted to $\mu\text{g/g}$. Table 6-13 reflects a comparative summary. Because of the unavailability of another analytical baseline data, we can postulate that data profile is similar in general.

Table 6-14 compares 1979 and 1992 ratios for water insoluble (or totals) over water soluble concentrations for major select analytes. The following observations can be made.

- Higher ratio of water insoluble bismuth in 1992 is most likely because of analytical error or sample variability. At this point, it is premature to make a scientific conclusion for lack of additional baseline data.
- Water insoluble silica was higher in 1992, which possibly indicates a shift in medium thermodynamic conditions or a kinetic equilibrium shift favoring the formation of a less soluble phase (such as the crystallization of fluorosilicic acid).

6.4.3 Inorganic Speciation

It is beyond the scope of the current version of the report to accurately speciate tank BX-107 contents because of the scarcity of information available about chemical reactions under alkaline and high-ionic strength media in a radiation field. However, thermodynamic equilibrium data is an excellent tool for predicting potential equilibrium species. Gibbs and activation energy values, as well as reaction rate data, provide insights on the time dependent change of chemical species. At this point we will focus on thermodynamic factors related to the Gibbs and Helmholtz free energies, which are directly related to the reaction potential equilibrium and solubility constants of ideal solid and liquid solutions. Tables 6-15 and 6-16 list those most major inorganic compounds expected to dominate tank BX-107 residues. Data presented were based on physical and chemical thermodynamic reasoning coupled with a serial stoichiometric molar balance derived from the 1992 analytical data. Figures 6-4 and 6-5 reflect the data presented in those tables.

Table 6-13. Comparison of 1979 and 1992 Data for Metallic Elements and Anions in $\mu\text{g/g}$.

Analyte	Average 1992 cores	1979 Core
Al^{+3}	14300	14590
Ca^{+2}	3960	NR
Cr^{+3}	968	74
Fe^{+2}	11100	15719
K^{+}	263	NR
Na^{+}	100000	114836
Bi^{+3}	22300	7614
Si^{+4}	6783	12,700
F^{-}	9190	1954
Cl^{-}	1140	NR
NO_3^{-}	137000	159783
NO_2^{-}	8120	NR
PO_4^{-3}	71,955*	90861
SO_4^{-2}	13400	9737
CO_3^{-}	1160	1029

*Based on total phosphorus.

Table 6-14. Water Insoluble to Water Soluble Ratios Comparison for 1979 and 1992 Data.

Analyte	1979 Ratio	1992 Ratio
Na^{+}	1.7	1.5
Bi^{+3}	47.35	95.3
Si^{+4}	7.0	23
F^{-}	1.15	NR
SO_4^{-2}	0.15	0.3
PO_4^{-3}	4.8	4.98

Table 6-15. Postulated Major Equilibrium Water Soluble Compound Based on 1992 Data.

Major potential soluble compounds	Maximum concentration moles/L	Other potential equilibrium species plus notes
KCl	0.00543	Other potassium salts and hydroxides
CaCl ₂	0.01562	Other calcium salts
NaNO ₃	3.18194	Other sodium salts and hydroxides
NaNO ₂	0.25419	Oxidizing to nitrate
[Si(OH) ₆] ²⁻	0.01512	Ammoniumsilicofluoride, silica, H ₂ SiF ₆ .XH ₂ O, aluminofluorosilicates, fluorophosphates
FeSO ₄ .7H ₂ O	0.201	Other ferrous and ferric salts, oxides and hydroxides
Cr ₂ (CO ₃) ₃	0.00421	Other chromium salts, oxides, and hydroxides
BiPO ₄	0.00161	Other bismuth salts and hydroxides
Na ₂ SO ₄	0.19797	Other sodium salts and hydroxides
Na ₂ CO ₃	0.02324	Other sodium salts and hydroxides
Na ₃ PO ₄	0.128367	Other sodium phosphates, other sodium salts and hydroxides
AlF ₃	0.00715	In equilibrium with other aluminum salts and oxides
F ⁻	0.58434	Unaccounted for
PO ₄ ⁻	0.085263	Unaccounted for

Table 6-16. Postulated Major Equilibrium Compounds in Sludge Based on 1992 Data.

Major potential compound	Expected concentration moles/L	Other potential equilibrium species plus notes
BiPO_4	0.15364	Other metal phosphates, bismuth hydroxides
$\text{FeO} \cdot x \text{H}_2\text{O}$	0.28543	Other iron salts and oxides, ferrous and ferric sulfates
Ca(OH)_2	0.03744	Other calcium salts
K_3PO_4	0.00971	Other potassium salts, hydroxides
CrPO_4	0.02681	Other chromium salts, hydroxides
$\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$	0.46944	Other sodium salts, hydroxides
NaNO_3	5.91665	Other sodium salts and hydroxides, NaNO_2
$y\text{SiO}_2 \cdot x\text{H}_2\text{O}$	0.34884	Silica, aluminofluorosilicates, $\text{H}_2\text{SiF}_6 \cdot x\text{H}_2\text{O}$
Al(OH)_3	0.76267	Other aluminum salts, aluminum silicate hydrates. Again postulated based on process knowledge and most stable thermodynamic phase, because no hydroxide data is available

NOTE: Appendix A contains a copy of material balance estimates based on the oxide model as reported in the tank BX-107 data package.

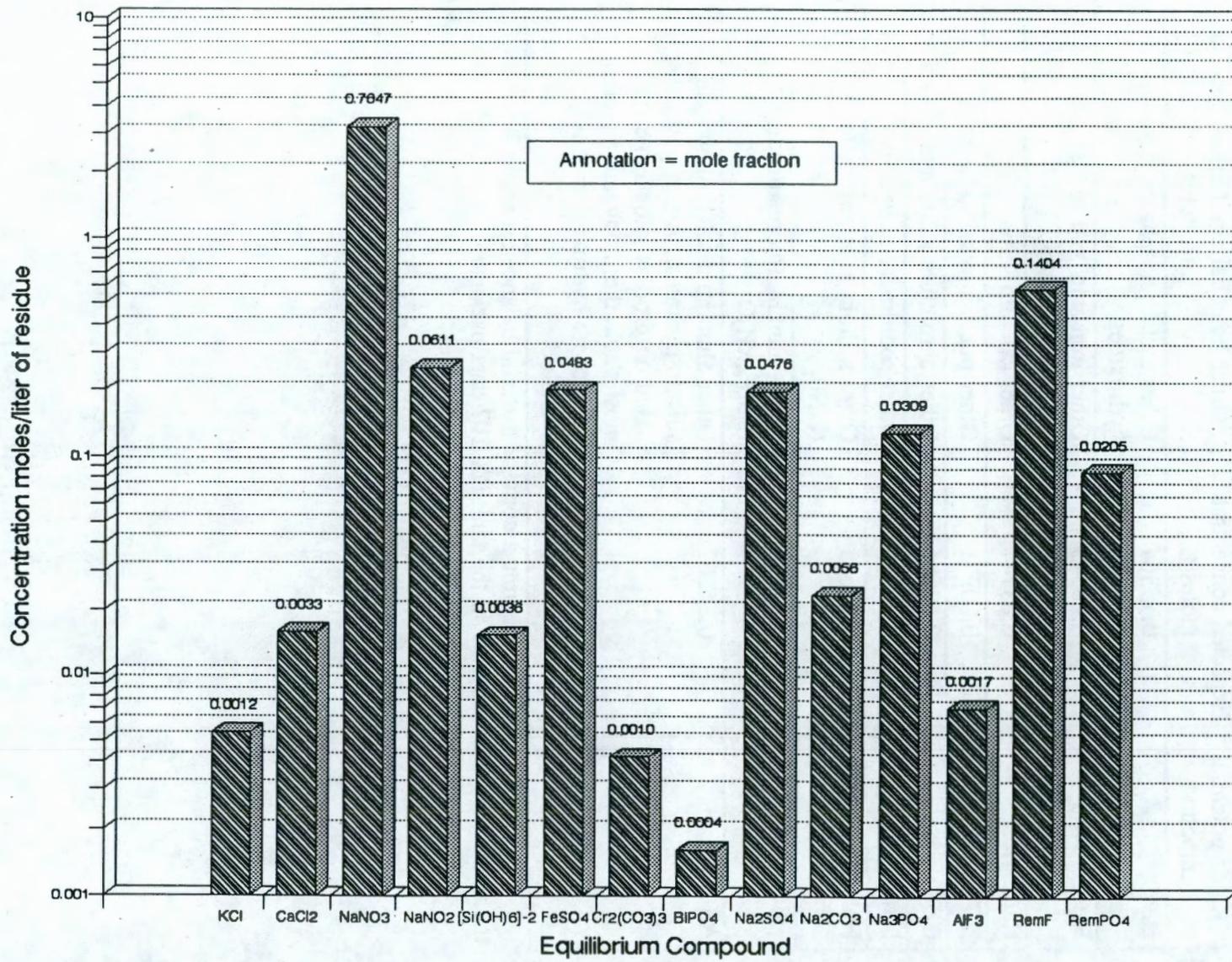


Figure 6-4. Postulated Water Soluble Chemical Profile.

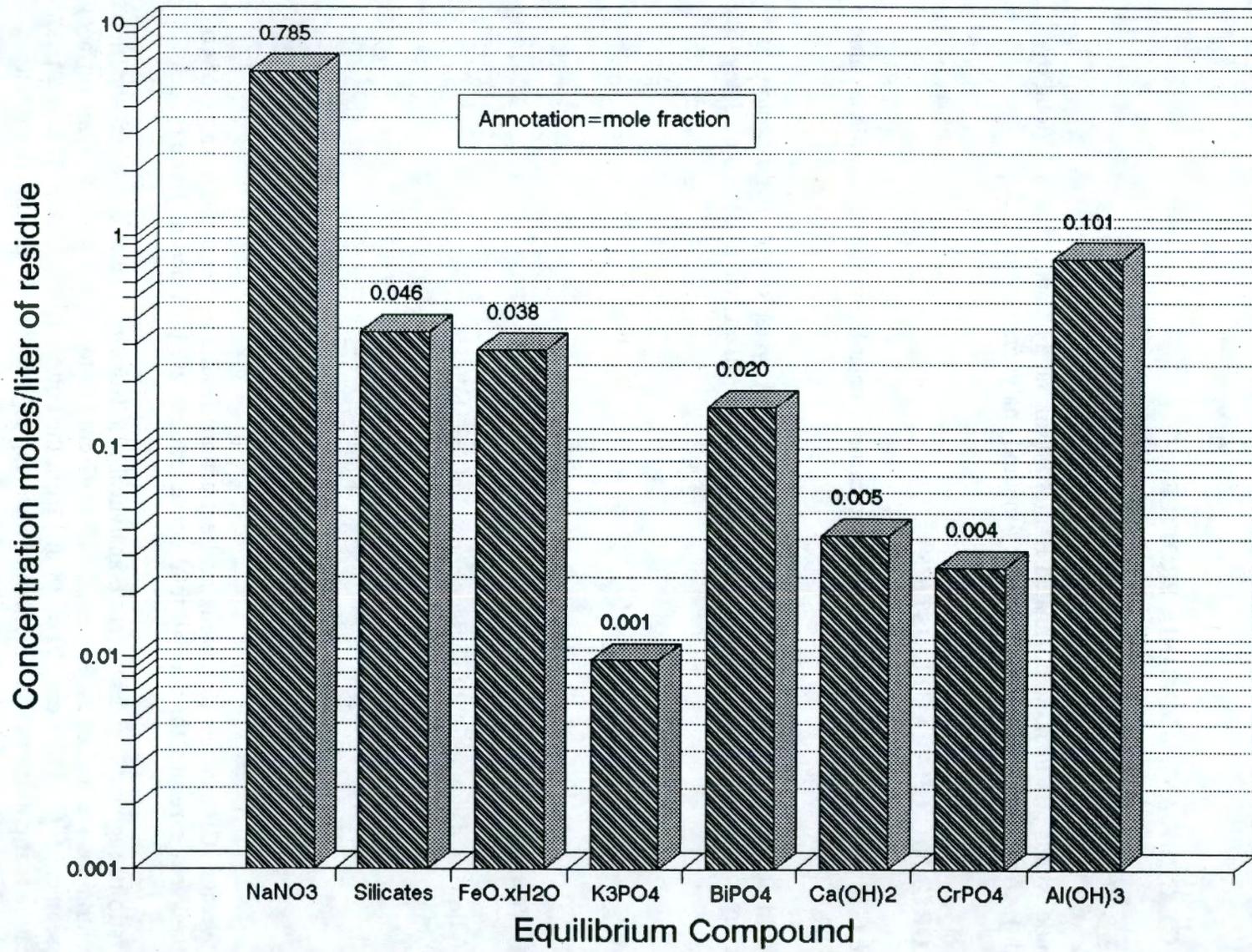


Figure 6-5. Postulated Sludge Equilibrium Profile.

It should be noted that the tank BX-107 wastes are not as simple as the above-postulated chemical formulas. Metal to metal bonds are possibly very abundant; metal oxides are most likely chained to hydroxides and salts.

6.5 CYANIDE AND AMMONIA PROFILE

Table 6-17 summarizes results reported for those key safety compounds.

Cyanide is thus less than the postulated Hanford Tank safety standard of 3% (total by weight). Ammonia results were biased by method analytical procedure.

6.6 ORGANIC CHARACTERISTICS

Table 6-18 and 6-19 summarize total organic carbon, volatile, and semi-volatile organic average results.

It should be noted that total organic carbon accounts for water soluble hydrocarbons and other organic molecules, such as hydrocarboxylic acids (like citric acid) and chelates (like EDTA).

6.7 WASTE SPATIAL MODEL

6.7.1 Vertical Layering

The following data are used to assess horizontal variability.

- Core 41, segments 2 and 6 homogenization test results
- Core 40, segments 2, 4, and 6 physical tests
- Water % data for all core segments
- Particle size analysis data
- Sampling resistance and recovery data.

The average ICP analysis (following acid extraction) results for segments 2 and 6 of core 40, and the average results for core 40 total concentrations are provided in Table 6-20.

The ratio of segment 2 and segment 6 analytical results indicates that potassium and zinc concentrations are higher in segment 2 (48 to 97 cm [19 to 38 in.] from residues surface) than segment 6 (51 to 97 cm [20 to 38 in.] from the bottom of the tank). This is a possible indication that potassium and zinc (both processing high ionization potential) are not associated with a heavy anion (possibly chlorides or fluorides).

Table 6-17. Average CN and NH₃ Results in $\mu\text{g/g}$. (1992)

Analyte	Mean	Analytical range	Statistical range
CN ⁻	2.49	1.06 - 3.14	0 - 7.37
NH ₃ /NH ₄	<4.5E3	N/A	N/A

Table 6-18. Average Total Organic Carbon. (1992)

Total organic carbon (following water extraction)	6.75E2 $\mu\text{g/g}$
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Table 6-19. Average Volatile and Semi-volatile Organics in $\mu\text{g/g}$. (1992)

Organic	Volatile	Semi-Volatile
Decane	2.85	N/A
Undecane	2.84	10.78
Dodecane	3.09	595
Tridecane	2.20	1675
Tetradecane	6.23	1225
Pentadecane	N/A	42
Tributylphosphate	N/A	17.6
Totals	17.21	3565

NOTE: The presence of those compounds, except tributylphosphate, is attributed to NPH contamination.

Table 6-20. Core 41 Segments 2 and 6 and Composite Analytical
Average Comparison in $\mu\text{g/g}$.

Element	Segment 2	Segment 6	Ratio seg. 2/seg. 6	Core 41 composites
Al	16250	17000	0.96	12950
Sb	35.25	34.3	1.03	183
As	6.51	2.985	2.18	14.95
Ba	8.965	8.51	1.05	11.2
Be	0.289	0.2885	1	0.28
Cd	3.785	2.375	1.59	1.9
Ca	368.5	281.5	1.30	1344
Cr	1205	1025	1.18	905
Co	1.39	1.47	0.95	2
Cu	22.15	18.3	1.21	69.45
Fe	17250	12100	1.42	9935
Pb	95.7	65.5	1.46	55.1
Mg	161.5	73.55	2.20	160
Mn	49.55	40.05	1.23	58.6
Ni	16.4	12.45	1.32	10.65
K	563	102.85	5.47	232
Ag	1.022	0.9625	0.9	0.9
Na	97100	99050	0.98	102500
V	5.5	NR	N/A	NR
Zn	121	43.95	2.75	61.7
Bi	31150	27950	1.11	21250
B	28.7	25.95	1.1	56.05
Ce	259.5	191	1.36	135
La	1.54	2.02	0.76	1.5
P	26600	25400	1.05	22500
Si	2240	1490	1.50	6300
Sr	219	172.5	1.27	166
S	4310	4020	1.07	4740
Sn	9.95	8.51	1.17	5
Ti	5.755	4.205	1.37	3.3
Zr	145.5	93.9	1.55	128

In general, the data indicate a close resemblance between the two segments. Their profile matches closely with the core 41 composite profile.

Physical tests (including TGA and water % data) can also be an indication of the spatial variability. They were presented in Tables 5-3 and 5-9. However, because of the sampling and analytical time span, those results are slightly biased (especially because of water loss by evaporation and segment recovery values). In general, they correlated well. It should be noted that the lower density number for segment 6 of core 41 is because of the lower % solids in that sample.

Table 5-4 presented particle size analysis data for cores 40 and 41 segments. The focus will be on number distribution at this point. Figure 6-6 is a plot of this data relative to cores 41. It shows that particle size distributions along riser 7 is consistently homogeneous. The same conclusion is reached by examining the number distribution data for core 40 and the particle size volume distribution for both cores 40 and 41.

Segment recovery and sampling resistance data is presented and assessed in Appendix A of this report. However, it is mostly inconclusive at this point and is not an indication of possible vertical layering of tank BX-107 residues. In general, it can be postulated that material along riser 7 (core 40) is vertically consistent, while material along riser 3 (core 41) exhibited higher resistance to sample penetration approximately 50 in. from the waste surface. No penetration resistance data is available for core 41, segments 5, 6, and 7. Thus, at this point, and with the amount of data on hand, one can postulate that tank BX-107 residues are consistently layered.

6.7.2 Horizontal Layering

Because a discussion on horizontal layering can be misleading, assessing the horizontal variability between the material along risers 7 and 3 is the focus of this assessment.

As shown in the previous section, segment level data from both risers showed matching results, and pointed to the presence of mostly homogeneous material (on a macro-molecular scale).

The following analytical data is evaluated.

- Radiochemical analysis results
- Metallic elements and electrolytes results

Tables 6-21 and 6-22 summarize average radiochemical, metals, and electrolytes analysis results respectively for cores 40 and 41.

Figure 6-6. Particle Size Number Distribution Core 41.

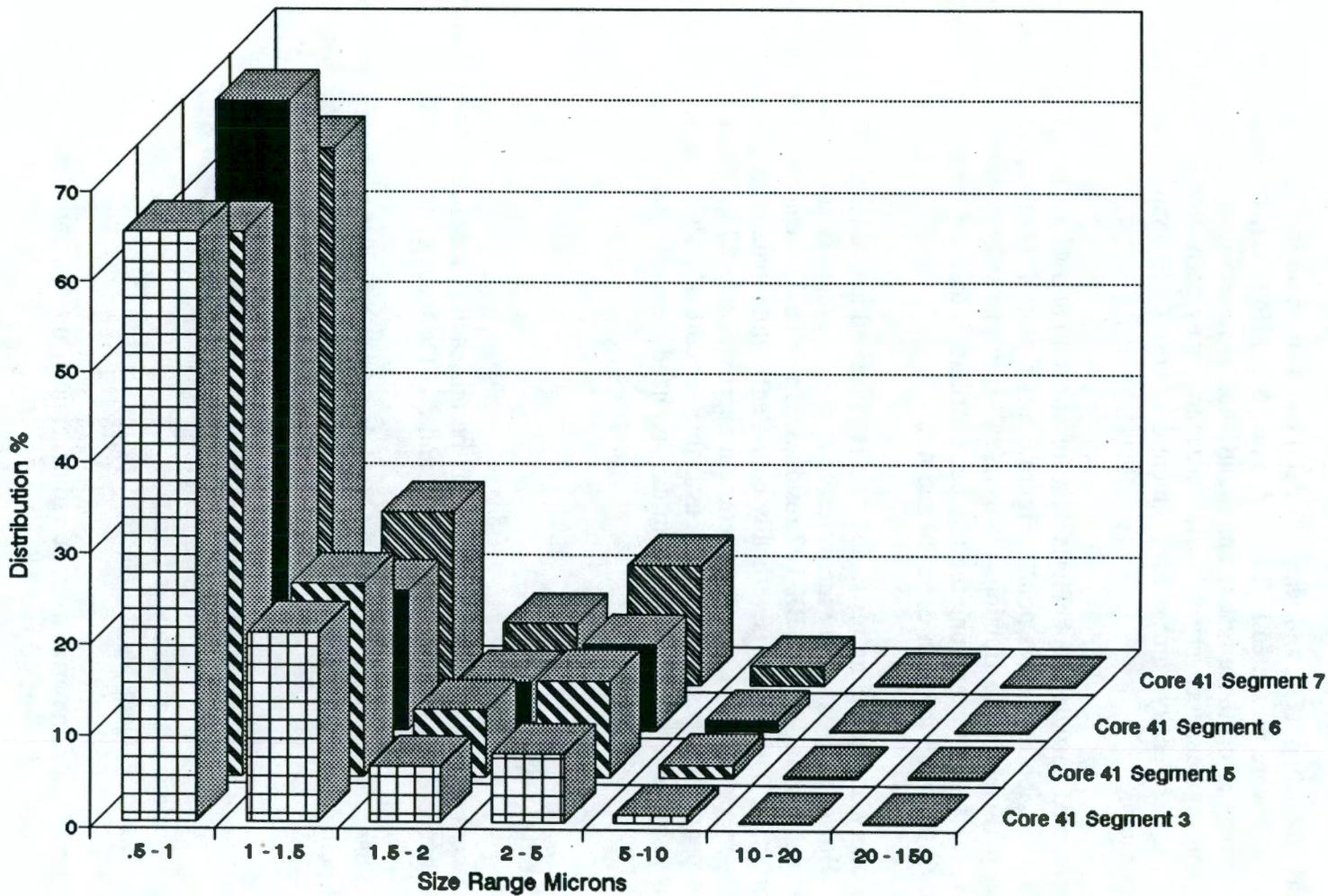


Table 6-21. Core 40 and 41 Average Radionuclides Results in $\mu\text{Ci/g}$.

Radionuclide	Core 40	Core 41	Ratio (core40/41)
AT	0.06955	0.1445	2.07
TB	43.05	50.45	1.17
Cs ¹³⁷	12.35	22.45	1.82
Sr ⁹⁰	9.11	10.625	1.17
Am ²⁴¹	0.0121	0.0139	1.15
Pu ^{239/240}	0.0618	0.05255	0.85
Tc ⁹⁹	0.03355	0.0403	1.20

Table 6-22. Cores 40 and 41 Average Inorganic Profile in $\mu\text{g/g}$.

Electrolyte	Core 40	Core 41	Ratio (core 41/40)
Al	12950	15700	1.21
Sb	183*	183*	1
As	15	14.95	1
Ba	7.07	11.2	1.58
Ca	1344	733	0.55
Cr	905	1032	1.14
Cu	69.45	33.85	0.49
Fe	9935	12200	1.23
Pb	55.1	93.9	1.7
Mg	160	124	0.78
Mn	58.6	70.5	1.2
Ni	10.65	13.8	1.3
K	232	294	1.27
Se	41.45	37.15	0.9
Na	102500	98500	0.96
Zn	61.7	105	1.7
Bi	21250	23350	1.1
B	56.05	38.05	0.68
Ce	135	215	1.59
P	22500	24450	1.09
Si	6300	7265	1.15
Sr	166	172	1.04
S	4740	4140	0.87
Zr	128	144	1.13
Hg	0.493	0.64	1.30
F	8285	9890	1.19
Cl	1170	1120	0.96
NO ₃ ⁻	146000	129000	0.88
NO ₂ ⁻	9420	12250	1.3
PO ₄ ⁻³	14700	13800	0.94
SO ₄ ⁻²	14150	14150	0.89
CO ₃ ⁻²	1150	1150	1.02
pH	9.66	9.77	1.01

*Matrix interference.

NOTE: Metal results were from total fusion results while anions came from IC analysis results.

Thus, based on the data presented in the two previous subsections, we can conclude that tank BX-107 residue constituent are evenly distributed vertically along risers 3 and 7, and horizontally between those two risers. No evidence of layering was found.

6.8 TWRS PROGRAM ELEMENTS INFORMATION

TWRS elements (i.e., Tank Safety Screening, Waste Tank Operations and Maintenance, the HWVP Program, the Retrieval Program, Waste Tank Safety, and the Pretreatment Program) need various waste characterization information. This section addresses the specific needs of those elements as they relate to tank BX-107.

6.8.1 Tank Safety Screening

The safety screening element is aimed at rapid classification of the Hanford Site high-level waste tanks into safety categories (Watch List and non Watch List) based on ferrocyanide, flammable gas, organics, and radionuclides (criticality issues) contents. Table 6-23 presents the tank safety screening summary, which shows that no tank safety screening criteria was exceeded.

6.8.2 Waste Tank Operations and Maintenance

Many of the safety issues are of concern to the Waste Tank Operations and Maintenance element of TWRS. While temperature and waste level are continuously monitored, vapor pressure is maintained at atmospheric. Table 6-23 contains some of the analytes of interest to this TWRS element. The concentration of other parameters of interest, such as pH, temperature, pressure, and corrosion electrolytes, can be found in Table 5-15.

6.8.3 HWVP Program

The current waste disposal plan focuses on the transformation of a pretreated, volume reduced, tank waste into high- and low-level glass capsules suitable for disposal into a geologic repository. Thus, current and future characterization efforts are essential to the success of this program. The concentration and ranges of key analytes, such as phosphates, fluoride, chloride, TOC, mercury, lead, chromium, and radionuclides can be found in Tables 5-15, 6-8, and 6-11.

6.8.4 Retrieval Program

In its current technology development efforts, the Retrieval Program also needs a multitude of tank characterization data. Historically, physical data has been used the most in

Table 6-23. Tank Safety Screening Summary.

Primary decision variable	Primary decision criterion (Babad 1993)	1992 Core data results
Energetics	≤ 125 cal/g	No exotherms
Total organic carbon	$< 3\%$ dry basis	0.11 - 0.18% dry basis
Water %	17 wt%	56 wt%
Gas composition	25% LFL for all flammable gases	Not determined
Total alpha (criticality)	< 1 g/L	≈ 0.003 g/L

developing design specifications for waste retrieval equipment and waste simulation experiments. Again, Table 5-15 (1992 core data) contains information in this regard. Detailed information relative to physical data can also be found in Sections 5.3 and 6.1.

6.8.5 Waste Tank Safety Program

The majority of the Waste Tank Safety Program characterization needs are the major driver in the characterization effort. Volatile, semi-volatile, and plutonium and uranium isotopic profiles are of interest. Analytical data from 1992 indicates that no major volatile compounds were found. Tributylphosphate concentration averaged approximately 18 $\mu\text{g/g}$. Plutonium and uranium isotopic analysis are reported in Tables 5-5 and 6-9. Table 5-15 (1992 core data) contains inventory data.

6.8.6 Pretreatment Program

The average characteristics of the tank wastes are a key decision factor for pretreating tank wastes. Table 5-15, 1992 analysis and inventory data serves as excellent references in this area.

7.0 CONCLUSION AND RECOMMENDATIONS

No safety issues were revealed in the 1992 core data. The waste in tank BX-107 will continue to be appropriately managed through interim mitigation measures and continuous monitoring of tank temperature and waste level. In the future, the waste will be retrieved and appropriate pretreatment technologies, mostly involving waste volume reduction, will be applied. Pretreatment residues will then be vitrified into low- and high-level fractions and disposed of in a geologic repository.

Future characterization efforts should center around analytical speciation of the waste to develop a better understanding of the waste matrix.

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APPENDIX A

SUPPLEMENTAL DATA

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Figure A-1. 1990 Stabilization Record.

Page 1 of 2STABILIZATION EVALUATION
JET PUMPED TANKSTANK: 107-BX

Reason Jet Pumping Halted:

- Meets 0.05 gpm Criteria, Date of Shutdown 9-14-90
 Major Failure of Jet Pump, Date of Shutdown _____

Additional Comments: The surface is light brown, not marked sludge fine material with a pool of liquid located north of man #4.
 Evaluation: (See continuation page for calculations and additional comments)

Photograph review completed - Date of most recent photograph 9/11/90
 Tank history review completed.
 Tank temperature profile reviews completed.
 Starting Liquid Level 130.2" Date 6/20/90

Final Liquid Level Measured After Equilibration of Interstitial Liquid:
 Dip Tubes 98.6 Date 9/24/90 LOW N/A Date N/A FIC/Manual 120.7" Date 9/18/90

Total Net Jet Pump Production 23,113 gallons
 Supernatant volume 600 Date 9/24/90

Calculated Solid Characteristics:

Porosity	<u>13.2 %</u>	Permeability/viscosity (K/u)	<u>N/A</u>
Capillary Height	<u>24"</u>	Est. Drainable Liquid Remain.	<u>29330 gallons</u>
Final Avg. Flowrate	<u>< 0.05 gpm</u>	Date	<u>9/24/90</u>

Disposition of Tank:

- Tank Interim Stabilized at 29,330 gallons of Drainable Liquid Remaining
23,330 thousand gallons of Pumpable Liquid Remaining.
 Tank Not Interim Stabilized; Jet Pumping Restarted

Evaluation Performed by: VC Boyle Date 9/24/90
 Checked by: R.K. Lambour Date 9-25-90

APPROVED BY:

Manager, Single-Shell Tanks	<u>M.D. Diamond</u>	Date	<u>9-26-90</u>
Manager, Tank Farm Programs	<u>R.G. Wesselsfelder</u>	Date	<u>9-25-90</u>
Manager, Tank Farms	<u>T.B. Wesselsfelder</u>	Date	<u>9-26-90</u>

Distribution: SSTPE Tank History File, Defense Waste Safety, Tank Farm
 Surveillance Analysis & Support, Prepared by Signatures, Managers approval
 Signature

Table A-1. B Plant: Fuel Reprocessing (200 East Area) (1945-1952).

Compound name	Formula	Comments
Aluminum	Al	Fuel element constituent
Ammonium Nitrate	NH_4NO_3	
Ammonium Silicofluoride	$(\text{NH}_4)_2\text{SiF}_6$	
Ammonium Sulfate	$(\text{NH}_4)_2\text{SO}_4$	
Ammonium Sulfite	$(\text{NH}_4)_2\text{SO}_3$	
Ceric Nitrate	$\text{Ce}(\text{NO}_3)_4$	
Ferrous Sulfate	FeSO_4	
Hydrogen Fluoride	HF	
Hydrogen Peroxide	H_2O_2	
Hyflo-Super-Cel	Contains Silica	Product name
Lanthanum Nitrate	$\text{La}(\text{NO}_3)_3$	
Nitric Acid	HNO_3	
Oxalic Acid	$\text{HO}_2\text{CCO}_2\text{H} \cdot 2\text{H}_2\text{O}$	
Phosphoric Acid	H_3PO_4	
Potassium Hydroxide	KOH	
Potassium Permanganate	KMnO_4	
Silicon	Si	Fuel element constituent
Sodium Bismuthate	NaBiO_3	
Sodium Carbonate	Na_2CO_3	
Sodium Dichromate	$\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$	
Sodium Hydroxide	NaOH	
Sodium Nitrate	NaNO_3	
Sodium Nitrite	NaNO_2	
Sulfuric Acid	H_2SO_4	
Trichloroethane	CH_3CHCl_3	Equipment degreasing
Zirconyl Nitrate	$\text{ZrO}(\text{NO}_3)_2$	

Table A-2. B Plant: Strontium Recovery and Waste Fractionization
(200 East Area) (1965-1976). (2 sheets)

Compound name	Formula	Comments
Aluminum Nitrate Nonahydrate	$Al(NO_3)_3 \cdot 9H_2O$	
Ammonia (Anhydrous)	NH_3	
Calcium Carbonate	$CaCO_3$	
Carbon Dioxide	CO_2	
Chromium Nitrate	$Cr(NO_3)_3$	
Citric Acid	$C_3H_4OH(COOH)_3$	
Di2-Ethyl Hexyl Phosphoric Acid	$C_{16}H_{34}O_2POOH$	
Duolite ARC-359 (IX Resin)	Sulfonated Phenolic	Product name
Ferric Nitrate	$Fe(NO_3)_3 \cdot 6H_2O$	
Hydrogen Peroxide	H_2O_2	
Hydroxyacetic Acid	CH_3OCO_2H	
Lanthanum-Neodymium Nitrate	$La(NO_3)_3 \cdot Nd(NO_3)_2$	
Lead Nitrate	$Pb(NO_3)_2$	
Magnesium Nitrate	$Mg(NO_3)_2$	
Magnesium Carbonate	$MgCO_3$	
Nickel Nitrate	$Ni(NO_3)_2 \cdot 6H_2O$	
Nitric Acid	HNO_3	
Normal Paraffin Hydrocarbon	$C_{10}H_{22}$ to $C_{14}H_{30}$	
Oxalic Acid	$HO_2CCO_2H \cdot 2H_2O$	
Phosphotungstic Acid	$H_3[P(W_3O_{10})_4] \cdot 14H_2O$	
Potassium Permanganate	$KMnO_4$	
Silver Nitrate	$Ag(NO_3)_2$	
Sodium Acetate	$NaC_2H_3O_2 \cdot 3H_2O$	
Sodium Bisulfate	$NaHSO_4$	
Sodium Carbonate	Na_2CO_3	
Sodium Gluconate	$(CHOH)_4CO_2HCH_2ONa$	
Sodium Hydroxide	$NaOH$	

Table A-2. B Plant: Strontium Recovery and Waste Fractionization
(200 East Area) (1965-1976). (2 sheets)

Compound name	Formula	Comments
Sodium Nitrate	NaNO_3	
Sodium Nitrite	NaNO_2	
Sodium Persulfate	$\text{Na}_2\text{S}_2\text{O}_8$	
Sodium Phosphate	Na_3PO_4	
Sodium Sulfate	Na_2SO_4	
Sugar	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$	
Tartaric Acid	$\text{C}_2\text{H}_2(\text{OHCOOH})_2$	
Tetrasodium Ethylene Diamine-Tetra Acetate (EDTA)	$\text{N}_2\text{C}_2\text{H}_4(\text{C}_2\text{H}_2\text{O}_2\text{Na})_4$	
Tributyl Phosphate	$(\text{C}_4\text{H}_9)_3\text{PO}_4$	
Trichloroethane	CH_3CHCl_3	Equipment degreasing
Trisodium Hydroxyethyl Ethylene-Diamine Triacetate (HEDTA)	$\text{N}_2\text{C}_2\text{H}_4(\text{C}_2\text{H}_2\text{O}_2\text{Na})_3$ $(\text{C}_2\text{H}_4\text{OH})$	
Zeolite AW-500 (IX Resin)	$\text{Al}_2(\text{SiO}_5)_2(\text{OH})_2$	Product name

Table A-3. B Plant: Waste Encapsulation (200 East Area) (1974-1976).

Compound name	Formula	Comments
Acetone	$\text{CH}_3\text{C}_2\text{OH}_3$	
Boric Acid	H_3BO_3	
Calcium Chloride	CaCl_2	
Cesium Chloride	CsCl	
Hydrochloric Acid	HCl	
Hydrogen Peroxide	H_2O_2	
Nitric Acid	HNO_3	
Oxalic Acid	$\text{HO}_2\text{CCO}_2\text{H}\cdot 2\text{H}_2\text{O}$	
Phosphoric Acid	H_3PO_4	
Potassium Permanganate	KMnO_4	
Sodium Fluoride	NaF	
Sodium Hydroxide	NaOH	
Sodium Nitrate	NaNO_3	
Sodium Phosphate	Na_3PO_4	
Strontium Fluoride	SrF_2	
Trichloroethane	CH_3CCl_3	

A.1 Hydraulic Ram Pressure Data

As mentioned in the previous section, the drill string is powered by a hydraulic system. A major component of that system is a hydraulic vane pump whose function is to compensate for back-pressure surges during sampling operations. The response factor will not be discussed in this report. Due to the nature of the material being sampled, the hydraulic ram pressure will vary due to penetration opposite resistant as well as internal and external friction forces. If the hydraulic ram pressure remains (near) constant during the sampling operation, it is an indication that projected material resistances are being encountered. If, on the other hand, the ram pressure increases, it is an indication that the material has less resistance to penetration than projected, because the hydraulic vane pump output remains constant. If the ram pressure decreases, it is as a result of additional resistances to penetration. The vane pump will usually try to counteract those resistances by increasing its pumping capacity, thus hydraulic pressure, to within its maximum design or preset limitations. In summation, it can be mathematically stated that hydraulic ram pressure equals $\text{Function 1 \{Pump Capacity\} / \text{Function 2 \{Penetration Resistances\}}$. Thus, the hydraulic ram pressure is proportional to pump capacity, and inversely proportional to penetration resistances related to material physical properties and friction forces.

The hydraulic ram will cease operation as programmed or set if pressure, referring to the bottom ram hydraulic oil pressure, exceeds 70,310 kgs/m² (100 lb/in² [psi]).

A strip chart records the hydraulic ram pressure during sampling. Tables A-4 and A-5 compiled calculated data based on those strip chart readings. No definitive conclusions in regards to the relations between the hydraulic ram pressure and the hardness or the layering of the material in the tank will be made at this point. As additional tank push-mode sampling data is analyzed or becomes available, a formula containing all the major factors influencing the sampling performance could be developed.

Penetration velocity was calculated by dividing the penetration depth per segment length, namely 0.4826 m (19 in.), by the sampling time in seconds. The resistant force data source is a formula that correlates the hydraulic ram pressure reading to the ram down force, herein called resistant force. Tables A-6, A-7, and A-8 present a tabulated copy of those values. In general, cores 40 and 41 strip chart fingerprints had a 0 to 3 degrees negative slope as sampling time progressed, ending with a slight pressure surge as the sample valve was shut. An approximate average number was selected from each of the records available (error is estimated around 5%), and the corresponding calculated resistance force was presented. Core 41, segments 5, 6, and 7 charts were not found. Sampling velocity figures for the first segments also carry some error because the segment length was less than 48 cm (19 in.).

Table A-4. Core 40 Sampling Data (1992).

Segment number	1	2	3	4	5	6	7
Sampling date May 1992	13	13	14	14	14	18	18
Sampling time (sec)	90	83	75	45	74	78	105
Penetration velocity (in./sec)	0.211	0.229	0.253	0.422	0.257	0.244	0.181
Sample recovery %	0	100	0	100	100	100	66
Resistant force (PSI)	852	997	997	1141	997	1141	0 for 50 sec. 275 for 55 sec.

Table A-5. Core 41 Sampling Data (1992).

Segment number	1	2	3	4
Sampling date May 1992	28	29	29	June 3
Sampling time (seconds)	52	55	55	60
Penetration velocity (in./sec)	0.365	0.345	0.345	0.317
Sample recovery	0	100	50	0
Resistant force (PSI)	420 10 sec 997 12 sec 1141 30 sec	852 18 sec 1285 37 sec	852 5 sec 1285 30 sec >4027 1 sec 3450 10 sec	564 5 sec 1141 55 sec

Table A-6. Core Sampling Truck Downward Force

Strip Chart Reading	Bottom Ram Pressure (psi)	Ram Down Force (psi)
0	0	4027
0.25	7.5	3883
0.5	15	3738
0.75	22.5	3594
1	30	3450
1.25	37.5	3306
1.5	45	3161
1.75	52.5	3017
2	60	2873
2.25	67.5	2728
2.5	75	2584
2.75	82.5	2440
3	90	2295
3.25	97.5	2151
3.5	105	2007
3.75	112.5	1863
4	120	1718
4.25	127.5	1574
4.5	135	1430
4.75	142.5	1285
5	150	1141
5.25	157.5	997
5.5	165	852
5.75	172.5	708
6	180	564
6.25	187.5	420
6.5	195	275
6.75	202.5	131
7	210	-13

Table A-7. BX-107 Core 40 Time-Weighed Resistance Force (cont'd).

Velocity Inches/Second	Resistance (psi)	Events Seconds	TWRF (psi)
0.211	852		852
0.229	997		997
0.253	997		997
0.422	1141		1141
0.257	997		997
0.244	1141		1141
0.181	275	55	144

Table A-8. BX-107 Core 41 Time-Weighed Resistance Force.

Velocity (inches/seconds)	Resistance (psi)	Events (seconds)	TWRF (psi)
0.365	420	10	969
	997	12	
	1141	30	
0.345	852	18	1143
	1285	37	
0.345	852	5	1479
	1285	30	
	4027	1	
	3450	10	
0.317	564	5	1093
	1141	55	

Figures A-2 and A-3 are plots of the resistant force versus sampler velocity for cores 40 and 41, respectively. For segment 7 of core 40 and core 41, a time-weighted resistant force average was calculated as follows.

$$\text{Time-weighted resistant force} = \frac{1}{\text{Total segment sampling time}} * \sum_{1}^{n} \text{time} * \text{resistant force}$$

where n is the number of different events directly related to the change in pressure reading during segment sampling. Numerical analysis techniques could be applied to find the optimum penetration velocity, (e.g., for a specific waste stream or layer). However, this is beyond the scope of this report.

A.2 Chain-of-Custody Records

Shipment number is used to track sample handlers, carrier, and destination.

- Sample number, unique to each core segment, and used throughout the laboratory analytical process
- Sampling supervisor's signature for accountability purposes
- Tank number to designate the tank sampled
- Riser number which specifies the riser used for sampling a particular segment or core
- Segment number, segment 1 usually refers to the top segment sampled for a particular core
- Core number, usually a sequential number related to the fiscal year core sampling schedule of the designated tanks.
- Cask serial number that pinpoints the cask used for carrying the sample during transportation to the laboratory hot cell
- Radiation survey data, which reports radiation values on top, at the bottom, the sides, as well as any contamination that can be smeared, relative to the sample-loaded shipping cask

Figure A-2. Time-Weighted Resistant Force Versus Penetration Velocity for Core 40.

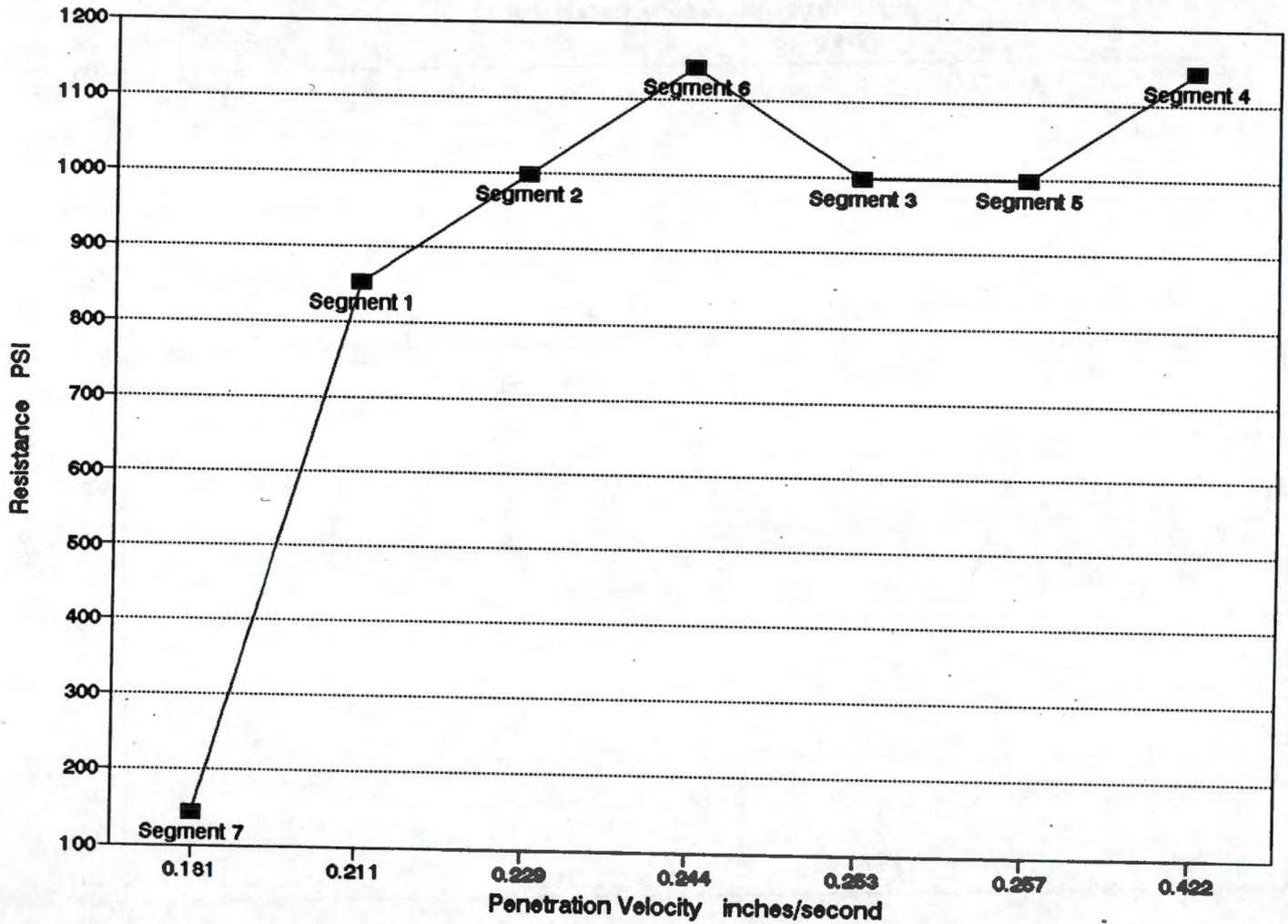
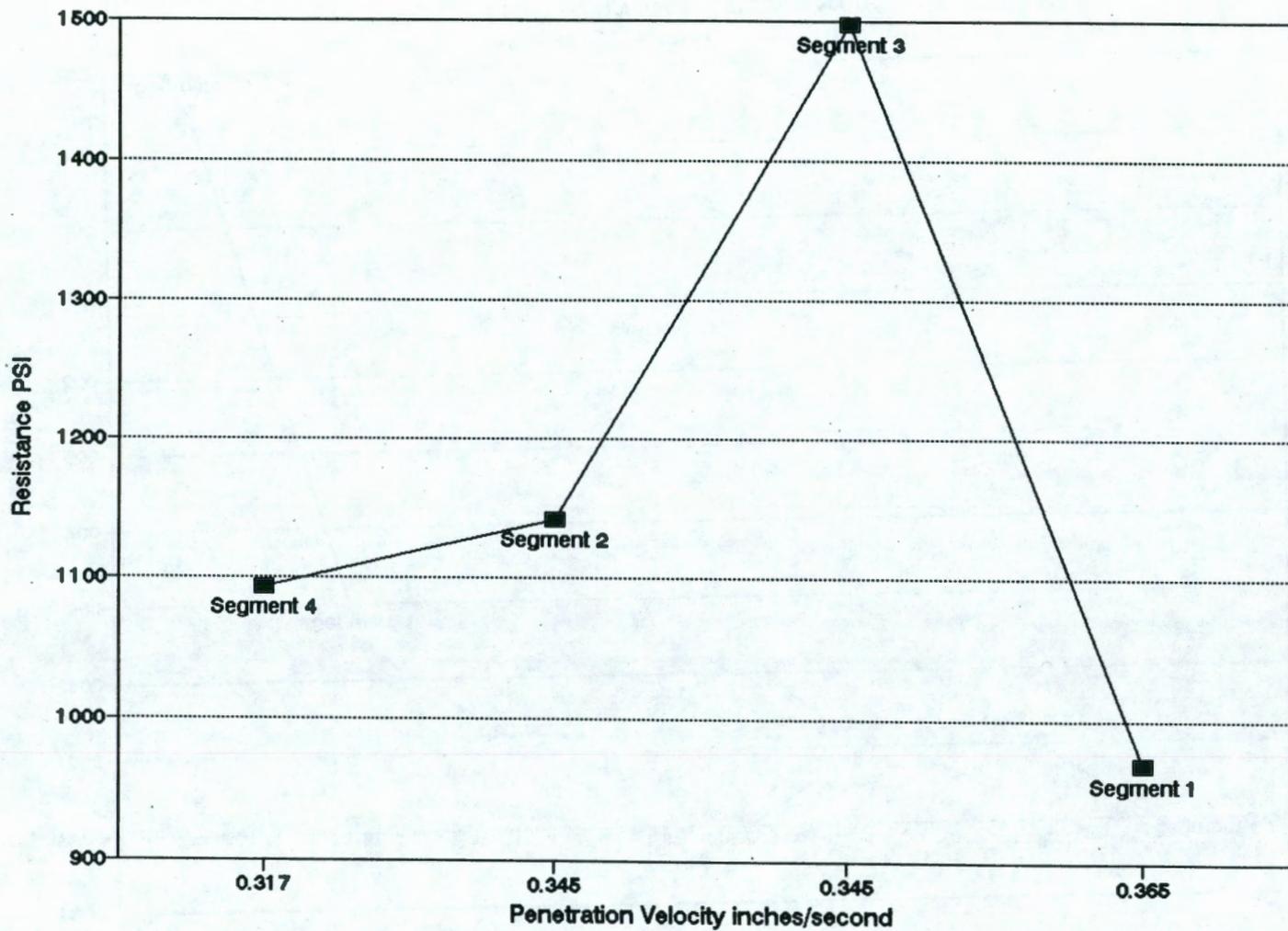


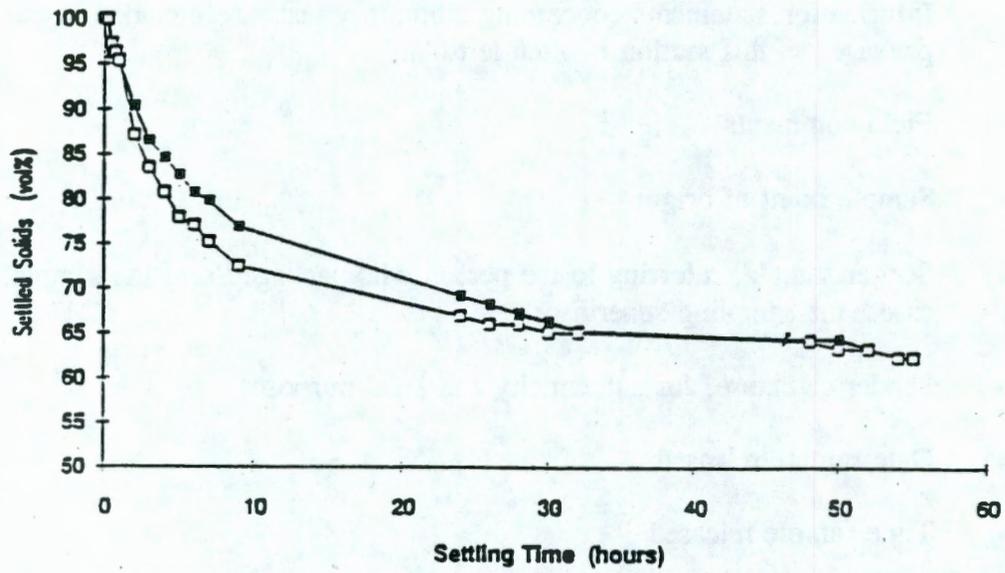
Figure A-3. Time-Weighted Resistant Force Versus Penetration Velocity for Core 41.



-
-
- Shipment description, (including the work package number, cask seal number, sampler number used, date and time sampler unsealed, expected liquid and solid contents, dose rate through drill string, and expected sample length)
 - Information statements concerning laboratory tests are included in the work package, so this section is often left blank
 - Field comments
 - Sample point of origin
 - Sender sample, referring to the person who packaged/sent the sample, in most cases, the sampling supervisor
 - Sender signature, for authenticity and legal purposes
 - Date sample released
 - Time sample released
 - Sample destination, usually a laboratory hot cell area
 - Seal check verification for shipment to laboratory
 - Sample recipient name
 - Sample recipient signature
 - Date sample received into the laboratory hot cell
 - Time sample received
 - Seal check verification upon arrival to laboratory
 - Seal data verification; a quality assurance step to verify sample tracking information
 - Laboratory comments section.

Figure A-4. Settling Rate Curves: Core 40, Segment 2.

Core 40 Segment 2 1:1 Dilution



Core 40 Segment 2 1:3 Dilution

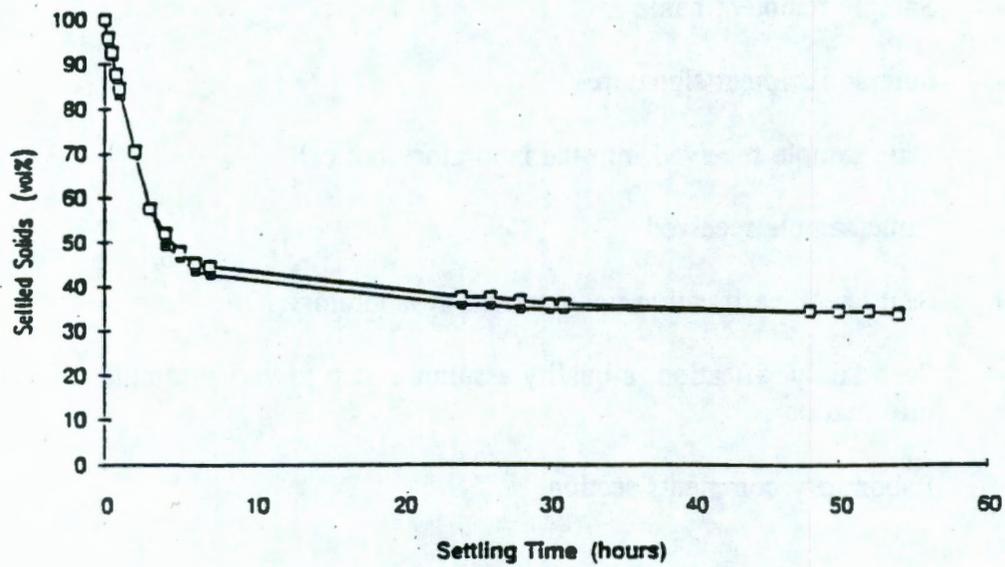
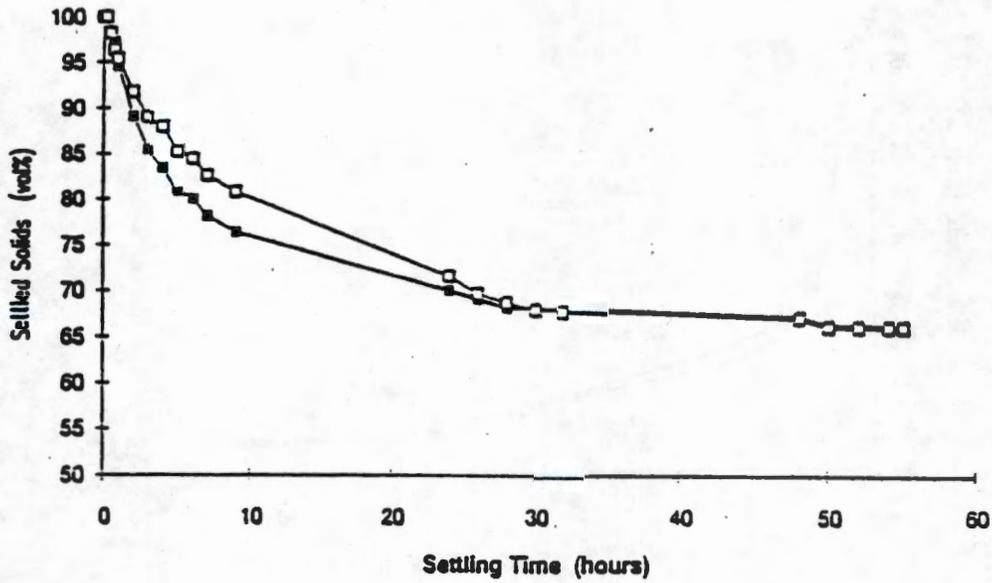


Figure A-5. Settling Rate Curves: Core 40, Segment 4.

Segment 4 1:1 Dilution



Segment 4 1:3 Dilution

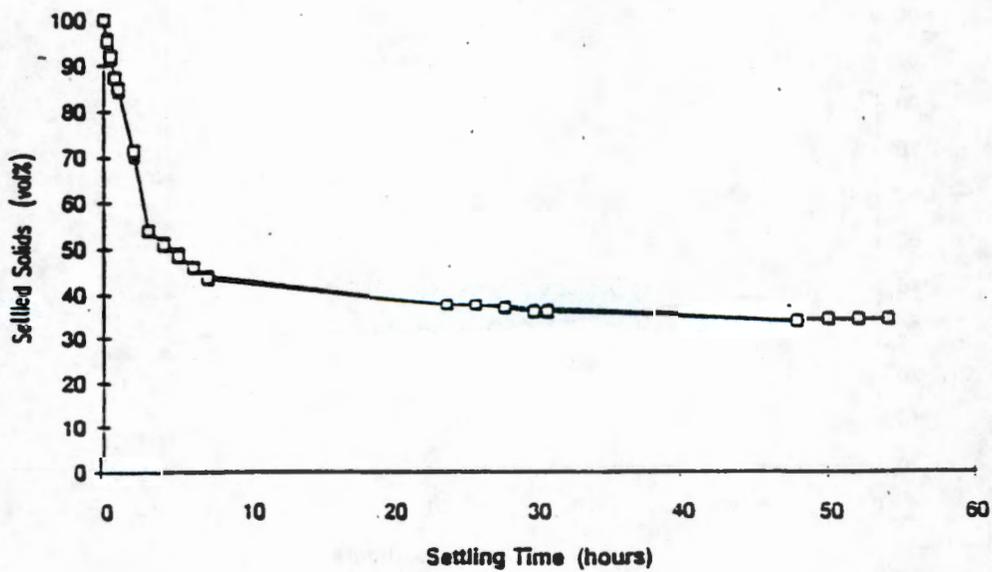
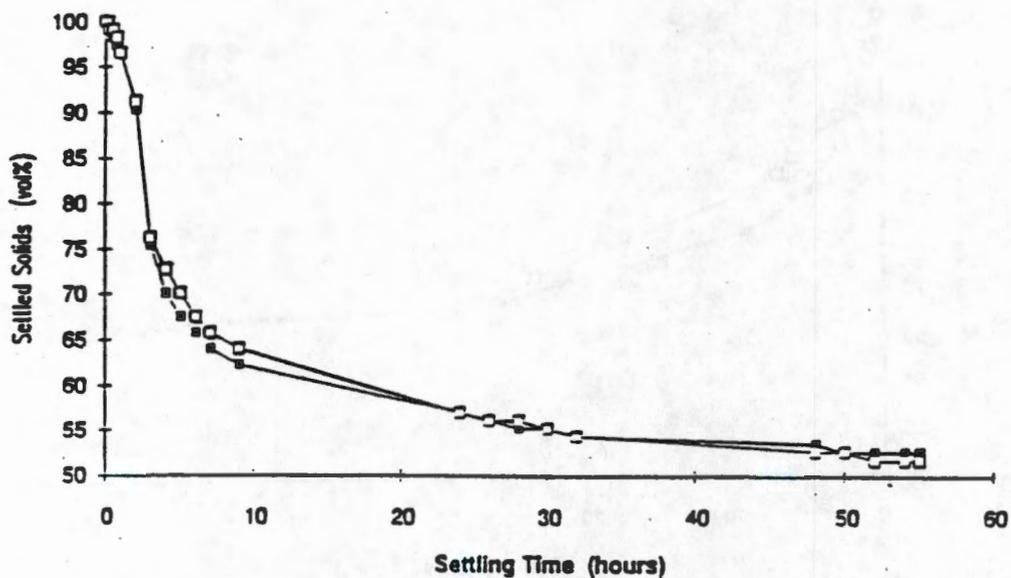


Figure A-6. Settling Rate Curves: Core 40, Segment 6.

Segment 6 1:1 Dilution



Segment 6 1:3 Dilution

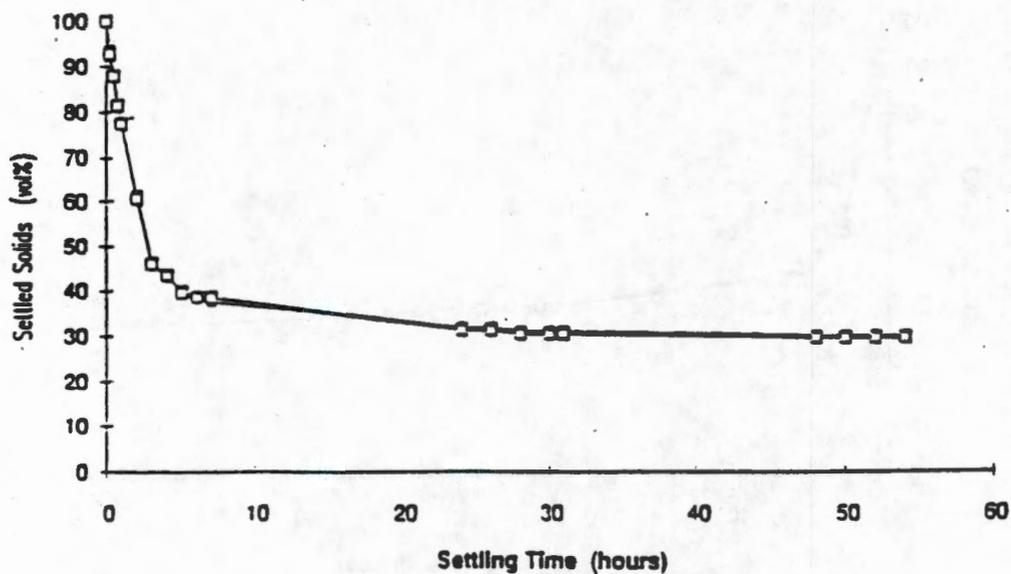


Table A-8. Composite Level Radiochemical Analysis Results.

Radio-chemical	Core 40 composite 1	Core 40 composite 2	Core 41 composite 1	Core 41 composite 2
U ²³⁸ $\mu\text{g/g}$	1.56E3 - 2.07E3	1.86E3 - 2.18E3	2.82E3	2.29E4 - 2.52E4*
Pu ^{239/240} $\mu\text{Ci/g}$	3.49E-2 - 5.32E-2	7.57E-2 - 8.34E-02	5.49E-2 - 5.64E-2	4.49E-2 - 5.41E-2
AT $\mu\text{Ci/g}$	7.89E-2 - 9.47E-2	7.17E-2 - 7.28E-2	1.13E-1 - 1.23E-1	1.15E-1 - 1.01E-1
TB $\mu\text{Ci/g}$	5.02E+1 - 5.03E+1	3.28E+1 - 3.88E+1	5.23E+1 - 5.24E+1	4.58E+1 - 5.12E+1
Ce ¹³⁷ $\mu\text{Ci/g}$	1.34E+1 - 1.35E+1	1.09E+1 - 1.16E+1	2E+1 - 2.04E+1	2.43E+1 - 2.51E+1
Sr ⁹⁰ $\mu\text{Ci/g}$	9.76 - 10.2	7.71 - 8.97	11.8 - 12	8.74 - 9.96
Am ²⁴¹ $\mu\text{Ci/g}$	8.05E-3 - 1.23E-2	1.38E-2 - 1.41E-2	1.28E-2 - 1.62E-2	1.31E-2 - 1.34E-2
Tc ⁹⁹ $\mu\text{Ci/g}$	3.56E-2 - 4.06E-2	2.83E-2 - 2.96E-2	4.23E-2 - 4.92E-2	3.24E-2 - 3.74E-2
H ³ $\mu\text{Ci/g}$	3.89E-4 - 4.02E-4	4.83E-4 - 7.79E-4	3.66E-4 - 4.2E-4	6.97E-4 - 1.08E-3
C ¹⁴ $\mu\text{Ci/g}$	1.33E-2 - 3.06E-4	1.82E-4 - 4.21E-4	2.89E-4 - 3.49E-4	2.24E-4 - 2.99E-4

*Possibly attributed to reporting error.

Table A-9. Cores 40 and 41 (1992) Analytical Data Summary.

Core	40	40	41	41	Analytical Standard Deviation	Analytical Data Minimum	Analytical Data Maximum	Analytical Cumulative Average	Estimated Inventory
Composite	1	2	1	2					
	Average	Average	Average	Average	Metallic Elements				
Units	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	Kgs
ICP.f.Be9	1.50	1.50	1.49	1.50	0.00	1.49	1.50	1.50	2.81
ICP.a.Be	0.28	0.29	0.29	0.28	0.01	0.27	0.2	0.28	
ICP.w.Be	0.31	0.31	0.3	0.30	0.00	0.30	0.31	0.31	
ICP.f.B11	18.37	18.81	6.65	16.17	5.33	4.98	20.63	15.00	
ICP.a.B	49.75	62.30	50.80	25.35	14.44	21.20	68.50	47.05	88.47
ICP.w.B	27.86	28.74	20.70	22.84	3.38	20.50	28.94	25.04	
ICP.f.Na23	103947	100699	101924	95021	3556	94482	106007	100398	
ICP.a.Na	100250	102500	103500	100000	1758	98500	104000	101563	190977
ICP.w.Na	53291	66992	80100	71999	18100	26264	81115	68096	
ICP.f.Mg24	111	89.78	131	115.70	15.64	85.54	131.55	111.95	
ICP.a.Mg	169.50	149.50	189	126	31.69	125	216	158.50	298.04
ICP.w.Mg	16.50	34.40	6.51	8.41	12.67	6.43	44.15	16.46	
ICP.f.Al27	13855	11989	17258	14068	1991	11178	17294	14293	26875
ICP.a.Al	11850	12350	16200	16550	2162	11400	16600	14238	
ICP.w.Al	105.29	80.38	71.60	279.40	91.99	69.90	350	134.17	
ICP.f.Si28	6600	6001	7986	6535	791.76	5635	7993	6780	12750
ICP.a.Si	1535	1720	1745	1655	138.38	1410	1860	1664	

Core	40	40	41	41	Analytical Standard Deviation	Analytical Data Minimum	Analytical Data Maximum	Analytical Cumulative Average	Estimated Inventory
Composite	1	2	1	2					
	Average	Average	Average	Average	Metallic Elements				
Units	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	Kgs
ICP.w.Si	414	364.34	136	262	119.89	133	523	294	
ICP.f.P31	24168	20759	27359	21450	2804	19390	28838	23434	44066
ICP.a.P	19750	21350	23950	27200	2860	19500	27500	23063	
ICP.w.P	4752	5001	4605	4812	193.03	4560	5258	4793	
ICP.f.S32	4832	4647	4148	4131	314.58	4033	4869	4439	8348
ICP.a.S	4760	5005	4475	4030	364.75	4030	5070	4568	
ICP.w.S	4556	4631	4395	4152	187.51	4130	4687	4433	
ICP.a.K	264.50	199	306	282	40.14	199	313.00	262.88	494.31
ICP.w.K	167.09	137.22	156	129.51	15.46	127	170.17	147.45	
ICP.f.Ca40	360.12	306.39	368.74	549.32	93.19	303	578.37	396.14	
ICP.a.Ca	2143	547.50	1168	296	1057.05	286	3500	1039	1953
ICP.w.Ca	752.18	713.66	147.50	120.98	373.98	105.50	1133	433.58	
ICP.f.Ti48	3.25	5.80	7.27	9.05	2.52	2.00	11.21	6.34	11.93
ICP.a.Ti	3.18	3.60	4.5	4.07	0.56	3.18	4.94	3.86	
ICP.w.Ti	0.41	0.41	0.51	0.41	0.04	0.41	0.51	0.43	
ICP.f.V51	18.30	7.81	10.45	0.00	7.06	0.00	23.59	9.14	17.19
ICP.a.V	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	

Table A-9. Cores 40 and 41 (1992) Analytical Data Summary.

Table A-9. Cores 40 and 41 (1992) Analytical Data Summary.

Core	40	40	41	41	Analytical Standard Deviation	Analytical Data Minimum	Analytical Data Maximum	Analytical Cumulative Average	Estimated Inventory
Composite	1	2	1	2					
	Average	Average	Average	Average					
Metallic Elements									
Units	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	Kgs
ICP.w.V	0.82	0.82	0.00	0.00	0.41	0.00	0.82	0.41	
ICP.f.Cr52	972.16	838.14	1080	983.02	91.05	787.29	1083	968	1821
ICP.a.Cr	854.50	877	1070	1030	95.25	820.00	1070	958	
ICP.w.Cr	157.43	133.04	156	159.42	12.30	131.35	171.23	151.47	
ICP.f.Mn55	62.78	54.38	73.44	67.63	8.52	49.86	81.63	64.56	121.39
ICP.a.Mn	35.40	33.35	47.20	43.10	5.79	33.00	48.20	39.76	
ICP.w.Mn	1.08	1.25	0.31	1.30	0.53	0.31	1.72	0.98	
ICP.f.Fe56	11010	8869	13281	11093	1619	8348	13361	11063	20803
ICP.a.Fe	9450	9425	12850	12300	1597	9040	12900	11006	
ICP.w.Fe	54.72	41.98	58.35	318.22	126.77	41.24	422.30	118.32	
ICP.f.Ni59	7203	1989	2856	10657	3932	1417	13676	5677	
ICP.a.Ni	10.95	10.37	13.95	13.60	1.64	9.93	14.30	12.22	22.97
ICP.w.Ni	1.43	1.43	1.84	1.42	0.18	1.42	1.84	1.53	
ICP.f.Co59	4.99	5.00	4.98	12.78	3.71	4.98	15.88	6.94	13.04
ICP.a.Co	2.08	1.70	2.32	0.95	0.54	0.91	2.55	1.76	
ICP.w.Co	1.02	1.02	1.22	1.09	0.09	1.02	1.22	1.09	
ICP.f.Cu64	67.29	71.62	26.47	41.20	19.84	25.88	73.62	51.65	97.12

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Table A-9. Cores 40 and 41 (1992) Analytical Data Summary.

Core	40	40	41	41	Analytical Standard Deviation	Analytical Data Minimum	Analytical Data Maximum	Analytical Cumulative Average	Estimated Inventory
Composite	1	2	1	2					
	Average	Average	Average	Average	Metallic Elements				
Units	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	Kgs
ICP.a.Cu	62.75	72.55	18.75	16.95	25.18	16.70	72.70	42.75	
ICP.w.Cu	0.73	0.78	0.61	0.87	0.14	0.61	1.07	0.75	
ICP.f.Zn65	75.31	48.12	88.15	121.73	27.22	44.92	132.77	83.33	156.69
ICP.a.Zn	56.10	35.50	81.05	65.50	19.23	31.30	85.00	59.54	
ICP.w.Zn	25.64	23.82	2.55	5.12	14.49	2.54	43.33	14.28	
ICP.f.As75	14.97	14.99	14.94	14.99	0.02	14.94	15.00	14.97	
ICP.a.As	2.80	2.86	3.03	2.77	0.12	2.74	3.1	2.86	
ICP.w.As	3.06	3.06	3.16	3.05	0.05	3.05	3.16	3.08	
HYAA .As	0.43	0.39	0.36	0.54	0.07	0.32	0.55	0.43	.81
ICP.f.Se79	41.42	41.46	41.33	330.25	125.28	41.33	344.73	113.61	
ICP.a.Se	312.50	295.50	369	7.67	140.56	7.59	372.00	246.17	
ICP.w.Se	8.46	8.46	13.35	68.22	25.45	8.45	74.48	24.62	
HYAA.Se	1.50	1.92	2.02	0.65	0.55	0.60	2.07	1.52	2.86
ICP.f.Sr89	170.72	160.05	187.76	155.31	14.25	144.20	187.94	168.46	316.77
ICP.a.Sr	154	157.50	185	170.00	12.70	147.00	185	166.63	
ICP.w.Sr	1.70	1.66	1.46	5.00	1.66	1.41	6.50	2.46	
ICP.f.Zr91	189.17	66.17	120.95	165.97	56.28	53.36	196.91	135.57	254.92

Table A-9. Cores 40 and 41 (1992) Analytical Data Summary.

Core	40	40	41	41	Analytical Standard Deviation	Analytical Data Minimum	Analytical Data Maximum	Analytical Cumulative Average	Estimated Inventory
Composite	1	2	1	2					
	Average	Average	Average	Average					
Metallic Elements									
Units	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	Kgs
ICP.a.Zr	94.60	92.15	87.95	110.00	12.57	77.20	115	96.18	
ICP.w.Zr	1.54	1.11	1.21	6.09	2.27	0.81	7.85	2.49	
ICP.f.Ag108	4.99	7.02	6.29	5.46	1.27	4.98	8.48	5.94	11.17
ICP.a.Ag	0.93	0.95	0.96	0.92	0.02	0.91	0.97	0.94	
ICP.w.Ag	1.02	1.02	1.02	1.02	0.00	1.02	1.02	1.02	
ICP.f.Cd112	3.68	3.51	4.35	5.26	0.86	3.49	6.22	4.20	7.90
ICP.a.Cd	1.86	1.93	2.74	2.56	0.40	1.67	2.77	2.27	
ICP.w.Cd	0.71	0.71	0.71	0.71	0.00	0.71	0.71	0.71	
ICP.f.Sn119	11.48	11.49	11.45	11.49	0.02	11.45	11.50	11.48	21.58
ICP.a.Sn	3.29	6.29	7.19	11.20	3.63	2.17	11.70	6.99	
ICP.w.Sn	2.34	2.34	1.63	2.34	0.31	1.63	2.35	2.16	
ICP.f.Sb122	182.63	182.82	182.27	182.82	0.26	182.27	183	182.64	343.42
ICP.a.Sb	34.15	34.85	35.15	33.80	0.71	33.50	35.60	34.49	
ICP.a.Sb	34.15	34.85	35.15	33.80	0.71	33.50	35.60	34.49	
ICP.f.Ba137	6.07	8.07	11.82	10.56	2.42	4.17	12.00	9.13	17.17
ICP.a.Ba	6.75	6.38	10.30	9.31	1.69	6.22	10.50	8.18	
ICP.w.Ba	0.38	0.53	0.31	0.55	0.14	0.31	0.67	0.44	

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Table A-9. Cores 40 and 41 (1992) Analytical Data Summary.

Core	40	40	41	41	Analytical Standard Deviation	Analytical Data Minimum	Analytical Data Maximum	Analytical Cumulative Average	Estimated Inventory
Composite	1	2	1	2					
	Average	Average	Average	Average					
Metallic Elements									
Units	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$	Kgs
ICP.f.La139	7.98	7.99	7.97	7.99	0.01	7.97	8.00	7.98	15.01
ICP.a.La	1.49	1.53	1.54	1.48	0.03	1.46	1.56	1.51	
ICP.w.La	1.63	1.63	2.14	1.63	0.22	1.63	2.14	1.76	
ICP.f.Ce140	92.10	177.56	201	228.81	60.85	48.90	252.00	174.87	328.83
ICP.a.Ce	150.50	169	191	203	20.30	147.00	205.00	178.38	
ICP.w.Ce	9.99	9.99	8.76	9.96	0.53	8.75	10.00	9.67	
CV.Hg201	0.61	0.37	0.63	0.64	0.12	0.37	0.72	0.56	1.06
ICP.f.Pb207	44.96	65.19	99.55	88.23	25.52	35.43	123.34	74.48	140.05
ICP.a.Pb	54.25	50.50	77.10	69.30	11.09	46.10	77.50	62.79	
ICP.w.Pb	7.23	7.23	8.66	7.77	0.60	7.23	8.67	7.72	
ICP.f.Bi209	21868	20587	28075	18589	3618	18296	28159	22280	41894
ICP.a.Bi	21200	22100	27150	26200	2582	20500	27300	24163	
ICP.w.Bi	106.37	80.24	113.50	634.73	253.32	78.34	838.66	234	

Table A-9. Cores 40 and 41 (1992) Analytical Data Summary.

Core	40	40	41	41	Analytical Standard Deviation	Analytical Data Minimum	Analytical Data Maximum	Analytical Cumulative Average	Estimated Inventory
Composite	1	2	1	2					
	Average	Average	Average	Average					
Units	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	Kgs
Spec.f.U238 µg/g	1815	2020	2820	2405	416.53	1560	2820	2265	4370 kgs
Units	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g	µCi/g	KCi
Radionuclides									
RAD.f.TA	8.68e-02	7.23e-02	1.18e-01	1.08e-01	1.88e-02	7.17e-02	1.23e-01	9.63e-02	1.81e-01
Am-241	1.02e-02	1.40e-02	1.45e-02	1.33e-02	2.16e-03	8.05e-03	1.62e-02	1.30e-02	2.44e-02
PU239/240	4.41e-02	7.96e-02	5.57e-02	4.95e-02	1.46e-02	3.49e-02	8.34e-02	5.72e-02	1.08e-01
U238	9.32e-06	5.27e-06	0.00	0.00	3.92e-06	0.00	9.32e-06	3.65e-06	6.86e-06
RAD.f.TB	5.03e+01	3.58e+01	5.24e+01	4.85e+01	6.76e+00	3.28e+01	5.24e+01	4.67e+01	8.79e+01
GEA.Cs-137	1.34e+01	1.13e+01	2.02e+01	2.47e+01	5.36e+00	1.09e+01	2.51e+01	1.74e+01	3.27e+01
Tc-99	3.81e-02	2.90e-02	4.58e-02	3.49e-02	6.55e-03	2.83e-02	4.92e-02	3.69e-02	6.94e-02
Sr-90	9.98e+00	8.25e+00	1.19e+01	9.35e+00	1.39e+00	7.71e+00	1.20e+01	9.87e+00	1.86e+01
C-14	2.20e-04	3.02e-04	3.19e-04	0.00	1.48e-04	0.00	4.21e-04	2.10e-04	3.95e-04
H-3	3.96e-04	6.31e-04	3.93e-04	8.89e-04	2.38e-04	3.66e-04	1.08e-03	5.77e-04	1.08e-03
Anions									
Units	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
IC.w.F- 19	8510	8415	8880	10950	1055	8110	11000	9189	17278
IC.w.Cl- 35	1155	1175	1040	1175	61.43	1020	1210	1136	2137
IC.w.NO2- 46	9685	9155	12450	1195	4198	1180	12500	8121	15271
Spec.w.NO2-	13150	10300	13250	12450	1241	10000	13900	12288	
IC.w.NO3- 62	142000	149500	126000	129500	9884	123000	153000	136750	257143
TIC.w.	1150	1148	12445	1100	242.96	699	1596	1161	2182

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Table A-9. Cores 40 and 41 (1992) Analytical Data Summary.

Core	40	40	41	41	Analytical Standard Deviation	Analytical Data Minimum	Analytical Data Maximum	Analytical Cumulative Average	Estimated Inventory
Composite	1	2	1	2					
	Average	Average	Average	Average					
IC.w.PO4-- 95	14400	14950	13400	14200	565.55	13400	15100	14238	26772
IC.w.SO4-- 96	14050	14150	13000	12250	792.05	12200	14200	13363	25127
TOC.w	700.00	0.29	0.0	896.50	408.16	0.00	997.00	399.20	750.65
NH4+ .w 17	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Dir.CN- 26	3.11	1.43	2.99	2.43	0.72	1.06	3.14	2.49	4.68
pH	9.63	9.68	9.82	9.72	0.11	9.58	9.95	9.71	
Physical Properties									
Dir.TGA(%)	56.45	63.50	64.05	52.50	5.07	50.70	66.30	59.13	
Dir. %Water	57.75	57.10	56.10	55.95	0.76	55.90	58.00	56.73	

Table A-10. BX-107 Charge Balance.

Analyte	Atomic / Molecular Weight	Prep Type	Oxidation State Used For Calc's	Oxidation Number	meq Wt. (g/meq)	milliequivalents Core 40 Comp 1	milliequivalents Core 40 Comp 2	milliequivalents Core 41 Comp 1	milliequivalents Core 41 Comp 2
Al	26.98	ICP Acid	Al+3	3	0.00899	1.323	1.368	1.801	1.846
Sb	121.75	ICP Acid	Sb+3	5	0.02435				
As	74.92	AA	As+3	5	0.01498				
Ba	137.34	ICP Acid	Ba+2	2	0.06867				
Be	9.01	ICP Acid	Be+2	2	0.00451				
Cd	112.4	ICP Acid	Cd+2	2	0.05620				
Ca	40.08	ICP Acid	Ca+2	2	0.02004	0.107		0.058	
Cr	52	ICP Acid	Cr+3	3	0.01733	0.049	0.051	0.062	0.059
Co	58.93	ICP Acid	Co+2	2	0.02946				
Cu	63.55	ICP Acid	Cu+2	2	0.03178				
Fe	55.85	ICP Acid	Fe+3	3	0.01862	0.508	0.507	0.693	0.661
Pb	207.2	ICP Acid	Pb+2	2	0.10360				
Mg	24.31	ICP Acid	Mg+2	2	0.01216				
Mn	54.94	ICP Acid	Mn+4	4	0.01373				
Ni	58.71	ICP Acid	Ni+2	2	0.02936				
K	39.1	ICP Acid	K+	1	0.03910				
Se	78.96	AA	Se+4	6	0.01316				
Ag	107.87	ICP Acid	Ag+	1	0.10787				
Na	22.99	ICP Acid	Na+	1	0.02299	4.350	4.437	4.480	4.350
Zn	65.37	ICP Acid	Zn+2	2	0.03269				
Bi	208.98	ICP Acid	Bi+3	3	0.06966	0.304	0.317	0.389	0.376
B	10.81	ICP Acid	B+3	3	0.00360				
Ce	140.12	ICP Acid	Ce+3	3	0.04671				
La	138.91	ICP Acid	La+3	3	0.04630				
Sr	87.62	ICP Acid	Sr+2	2	0.04381				
Sn	118.69	ICP Acid	Sn+4	4	0.02967				
Tl	47.9	ICP Acid	Tl+4	4	0.01198				
Zr	91.22	ICP Acid	Zr	4	0.02281				
Hg	200.59	AA	Hg+2	2	0.10030				
U up/g	238.02	Fluorimetry	UO2+2	2	0.11901	0.015	0.017	0.024	0.203
F	19	Direct IC	F-	-1	0.01900	0.448	0.443	0.467	0.576
Cl	35.45	Direct IC	Cl-	-1	0.03545	0.033	0.033	0.029	0.033
NO3	62	Direct IC	NO3-	-1	0.06200	2.290	2.411	2.032	2.089
NO2	46	Direct IC	NO2-	-1	0.04600	0.211	0.199	0.271	0.026
P	30.97	Acid ICP	PO4-3	-3	0.01032	1.908	2.063	2.315	2.635
S	32.06	Acid ICP	SO4-2	-2	0.01603	0.297	0.312	0.279	0.251
Si	28.09	Pusion ICP	SiO3=	-2	0.01405	0.470	0.427	0.569	0.466
TOC	59.02	TOC/TIC	Acetate	-1	0.05902				0.015
TIC	60	TOC/TIC	CO3=	-2	0.03000	0.038	0.038	0.041	
CN-	26.02	Direct CN	CN-	-1	0.02602				
					TOTAL CATIONS				
					(meq)	6.641	6.679	7.484	7.292
					TOTAL ANIONS				
					(meq)	5.695	5.927	6.004	6.091
					Ratio	1.166	1.127	1.246	1.197

Table A-11. BX-107 Material Balance.

Analyte	Atomic / Molecular Weight	Oxide Form	Method	No. Mole Atoms	No. Oxygen	Element Wt. to Oxide Wt. Conv. Factor	Wt. Percent Core 40 Comp 1	Wt. Percent Core 40 Comp 2	Wt. Percent Core 41 Comp 1	Wt. Percent Core 41 Comp 2
Al	26.98	Al2O3	ICP Acid	2	3	1.889	2.25	2.92	3.06	3.14
Sb	121.75	Sb2O5	ICP Acid	2	5	1.529				
As	74.92	As2O5	Direct AA	2	5	1.534				
Ba	137.34	BaO	ICP Acid	1	1	1.116				
Be	9.01	BeO	ICP Acid	1	1	2.776				
Cd	112.4	CdO	ICP Acid	1	1	1.142				
Cu	40.08	CuO	ICP Acid	1	1	1.999	0.50		0.16	
Cr	52	CrO3	ICP Acid	1	3	1.923	0.16	0.17	0.21	0.20
Co	58.93	CoO	ICP Acid	1	1	1.271				
Cu	63.55	CuO	ICP Acid	1	1	1.252				
Fe	55.85	Fe3O4	ICP Acid	3	4	1.382	1.51	1.30	1.78	1.70
Pb	207.2	PbO	ICP Acid	1	1	1.077				
Mg	24.31	MgO	ICP Acid	1	1	1.458				
Mn	54.94	MnO2	ICP Acid	1	2	1.582				
Ni	58.71	NiO	ICP Acid	1	1	1.273				
K	39.1	K	ICP Acid	1	0	1.000				
Se	78.96	SeO3	Direct AA	1	3	1.608				
Ag	107.87	Ag2O	ICP Acid	2	1	1.074				
Na	22.99	Na	ICP Acid	1	0	1.000	10.00	10.20	10.50	10.00
Zn	65.37	ZnO	ICP Acid	1	1	1.243				
Bi	208.98	Bi2O3	ICP Acid	2	3	1.115	2.36	2.46	3.02	2.92
B	10.81	B2O3	ICP Acid	2	3	3.220				
Ca	140.12	Ca2O3	ICP Acid	2	3	1.171				
La	138.91	La2O3	ICP Acid	2	3	1.173				
Si	28.09	SiO2	ICP Fusion	1	2	2.159	1.41	1.28	1.71	1.40
Sr	87.62	SrO	ICP Acid	1	1	1.183				
Ti	47.9	TiO2	ICP Acid	1	2	1.668				
Zr	91.22	ZrO2	ICP Acid	1	2	1.351				
F	19	F	Direct IC	1	0	1.000	0.85	0.84	0.89	1.10
Cl	35.45	Cl	Direct IC	1	0	1.000	0.12	0.12	0.10	0.12
NO3	62	NO3	Direct IC	1	0	1.000	14.20	14.95	12.60	12.95
NO2	46	NO2	Direct IC	1	0	1.000	0.97	0.92	1.25	0.12
P	30.97	PO4	ICP Acid	1	4	3.066	4.47	4.70	4.52	4.52
S	32.06	SO4	ICP Acid	1	4	2.996	4.09	4.16	3.94	3.73
CN	26.01	CN	Direct CN	1	0	1.000				
Org. Carbon	59.04	Amats	TOC	1	0	4.916				0.44
Inorg. Carbon	60.01	Carbonate	TIC	1	0	4.997	0.57	0.57	0.62	
% Water		Water	% Water				57.75	57.10	56.25	55.95
U	238.029	UO2	Fluorimetry	1	2	1.154	0.21	0.23	0.32	2.73
TOTAL %							101.02	101.33	100.54	101.01

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APPENDIX B

**STATISTICAL CHARACTERIZATION REPORT FOR
SINGLE-SHELL TANK 241-BX-107**

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JUL 21 1994

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LIST OF TERMS

95% LL	lower limit of a 95% confidence interval on the mean
95% UL	upper limit of a 95% confidence interval on the mean
ANOVA	analysis of variance
BX107	tank 241-BX-107
CI	confidence interval
df	degrees of freedom
Dir.	direct
DL	detection limit
GEA	gamma energy analysis
IC	ion chromatography
ICP	inductively coupled plasma analysis
ICP.a.	inductively coupled plasma analysis on a acid digested sample
ICP.f.	inductively coupled plasma analysis on a fusion digested sample
ICP.w.	inductively coupled plasma analysis on a water leached sample
Spec.w.	spectroscopic method on a water leached sample
KOH/Ni	Potassium Hydroxide/Nickel
Mean/DL	the mean of two sample results (sample and duplicate) divided by the detection limit
NA	not available
RAD.f.	radiochemistry analysis of a fusion digested sample
REML	restricted maximum likelihood estimation method
SST	single-shell tank
TGA	thermal gravimetric analysis
\bar{y}	weighted mean of the concentration data
$\hat{\sigma}^2(\bar{y})$	estimated variance of \bar{y}
$\mu\text{Ci/L}$	microcuries per liter
$\mu\text{g/g}$	micrograms per gram

**STATISTICAL CHARACTERIZATION REPORT
FOR
SINGLE-SHELL TANK 241-BX-107**

1.0 SUMMARY

This report provides the results of the statistical analyses of data from two core samples obtained from single-shell tank (SST) 241-BX-107 (BX107). Three topics are addressed. The results are summarized in this section.

Section 3.0 includes estimates of the mean concentration analytes found in BX107. In addition, 95% confidence intervals (CI) on the mean concentration are also given. The CIs tend to include a wide range due to limited number of degrees of freedom.

Section 4.0 includes the results of a statistical test conducted to determine the ability of the 222-S Analytical Laboratory to homogenize solid core segments. For 92% (23 out of 25) of the analytes, the variability between subsamples taken from different locations could not be distinguished from zero. Based on the results of this statistical test, it is generally concluded that the 222-S Analytical Laboratory can satisfactorily homogenize core segments.

Section 5.0 include estimates of the spatial variability (variability between cores) and two types of analytical error. These two types of analytical error are compositing variability (variability between composite samples within the same core) and analytical measurement error (variability between a sample and its duplicate analyses within each core composite sample). Statistical tests were conducted to test the hypothesis that the spatial variability of waste in BX107 is zero (i.e., to test whether or not the BX107 waste is homogeneous). For 31% (23 out of 74) of the analytes tested, the spatial variability was significantly different from zero (i.e., for these analytes the waste is heterogeneous).

Statistical tests were also conducted to determine the ability of the 222-S Analytical Laboratory to create reproducible core composite samples (i.e., test the hypothesis that the compositing variance is zero). For 33 out of 74 analytes, the compositing variance was significantly greater than zero. That is, for 45% of the analytes, the 222-S Analytical Laboratory cannot create reproducible core composite samples.

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2.0 INTRODUCTION

Two core samples (cores 40 and 41) were taken from BX107. The segment recoveries for each core are shown in Table 2-1. For core 40, there was 100% segment recovery for segments 2, 4, 5, and 6. For core 41 there was 100% segment recovery for segments 2, 6 and 7. The remaining segments had marginal to poor segment recovery. The average recovery for core 40 and 41 was 67% and 51% respectively. The data package (Kristofzski 1993) contains a complete description of the core samples and chemical analyses performed.

Table 2-1. Tank 241-BX-107 Core Recoveries.

Core	Segment number						
	1	2	3	4	5	6	7
40	0%	100%	0%	100%	100%	100%	66%
41	0%	100%	50%	0%	5%	100%	100%

The incomplete core recovery introduces a potential for a bias in the results given in this report. If the material recovered is assumed to be a random sample of the waste, then the reported mean analyte concentration values are unbiased estimates of the true concentrations. In addition, the CIs incorporate the "error" due to incomplete recovery. If the material recovered is not a random sample of the waste, then the results given here are biased. The magnitude of the bias is unknown.

Two core composite samples were made for each core from homogenized solid segment waste. Two samples were taken from each core composite. For the homogenization test, additional samples and duplicate samples were taken from two different locations in the mixed waste from segments 2 and 6 of core 41.

The Inductively Coupled Plasma (ICP) analysis Potassium Hydroxide/Nickel (KOH/Ni) fusion dissolution, ICP water leach and Ion Chromatography (IC) water leach analyses were performed on all composite core samples. The ICP acid digestion, radio chemistry, and Gamma Energy Analysis (GEA) were performed on the core composite samples and homogenization test samples. The tables in Appendix A list the data and are identified by the analysis method, the type of dissolution, and analyte (e.g., the notation ICP.a.A1 refers to aluminum, acid digestion, and an ICP analysis).

The core composite sample results are listed in Table A-1 (Appendix A). The homogenization test results are shown in Table A-2 (Appendix A). The ratios for the mean of each sample and the duplicate divided by the detection limit for that pair are also included in Tables A-1 and A-2. The purpose of these ratios will be discussed later in this section. A complete report of the sample results and the laboratory quality control data are outlined in Addendum 2 of the data package (Kristofzski 1993). The core composite data for each analyte are illustrated in the figures of Appendix B. Figures were not included for analytes that were eliminated from statistical analysis for reasons discussed below in this section.

The results from the chemical analyses of the core composite samples were used to obtain mean concentration estimates of the analytes in the waste. Some segment level data was used to evaluate the ability of the 222-S Analytical Laboratory to homogenize segments.

In general, statistical analyses of the data were completed for analytes with concentrations more than 10 times their detection limits (DL). Analytical Evaluation and Reporting personnel, within the Tank Waste Remediation System Engineering, identified a list of critical analytes that are exceptions to this rule. Table 2-2 includes this special list of analytes. Statistical analyses were completed for the analytes on this special list, if the concentrations were more than 3 times their DL. Analyte results with concentrations less than the DL were not used. For a number of analytes, the concentrations in some samples were more than a particular limit (3 or 10 times the DL), and the other samples were less than that limit. In these cases, the statistics were calculated using all of the data. The results, however, are to be used with caution. These rules do not apply to alpha or beta/gamma counting methods. The ratios (Mean/DL) reported in Tables A-1 and A-2 are provided to show the magnitude of the analyte concentrations relative to the DL.

Table 2-2. Special Analyte List.

Aluminum	Nitrate
Bismuth	Nitrite
Calcium	Phosphate
Chromium	Carbonate
Iron	Fluoride
Silicon	Chloride
Sodium	Total Organic Carbon
Zirconium	Cyanide

A thorough examination of the data revealed several potential anomalies in the core composite data. These potential anomalies are listed below.

- For ICP.a.Ca, ICP.a.Zn, ICP.f.Cu, ICP.w.P, and Spec.w.NO₂, a single sample result is significantly different than the cluster of remaining sample results within the particular core.
- The sample results from composite 2 of core 41 were significantly larger and more variable than the remaining core composite sample results for ICP.w.Bi, ICP.w.Fe, ICP.w.Al, ICP.w.Sr, and Fluor.U.
- For IC.w.NO₂, there are statistical differences between the results reported for the two composites for core 41.

There is no direct evidence that the results noted above are due to analytical measurement errors. Consequently, the statistical analysis was performed on the data as it is reported in Table A-1. The potential anomalies in the data noted above can be clearly seen in the figures in Appendix B.

3.0 MEAN CONCENTRATION ESTIMATES

One of the tasks outlined in the Tank Waste Characterization Plan, Section 5.1.1.2 (Bell 1993), is to estimate the constituent inventories in the waste. The inventories are estimated by computing mean concentrations and 95% CIs on the mean concentrations for each constituent. The estimate of the inventory and CI on the inventory of an analyte in the tank are equal to the corresponding mean concentration estimates and CI multiplied by the volume of waste in the tank. These estimates are not given in this document.

Table A-1 includes core composite data used to compute the mean concentration estimates and the CIs. The "NA" symbol indicates that the data are not available.

3.1 STATISTICAL METHODS

The concentration estimates shown in Table A-1 are shown as 95% CIs on the mean concentration. It is assumed that each sample and its duplicate are analyzed independently. The two analytical results are used to estimate the analytical measurement error. Due to the hierarchical structure of the data, the analytical measurement error alone is not the appropriate error term to use in computing the CIs. A linear combination of the analytical measurement variances and the spatial variance is the appropriate variance of the mean for the CIs. Appendix C includes a description of the statistical model and formulas used to calculate estimates of the mean, the variance of the mean, and the CI on the mean.

3.2 STATISTICAL RESULTS

Table 3-1 is a summary of statistics, by analyte, for ICP acid digestion, ICP water leach, ICP KOH/Ni fusion dissolution, radio chemistry, and IC analyses. The summary statistics are as follows:

\bar{y}	weighted mean of the concentration data
$\hat{\sigma}^2(\bar{y})$	estimated variance of \bar{y}
df	degrees of freedom
95% LL	lower limit to the 95% CI on the mean
95% UL	upper limit to the 95% CI on the mean.

For some analytes the lower confidence limit (95% LL) was negative. Because concentrations are more than or equal to zero, any negative 95% LL values were set equal to zero.

Because there was only two core samples taken from BX107, the CIs on the mean concentration include a very wide range relative to the range of the data. The 95% UL were on the average twice as large as the largest sample result reported in the composite data. There was only one degree of freedom for these CIs.

Table 3-1. Concentration Estimate Statistics. (sheet 1 of 3)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Analyte	\bar{y}	$s^2(\bar{y})$	df	95% LL	95% UL
ICP.a.Al	1.42E+04	4.57E+06	1	0.00	4.14E+04
ICP.a.B	4.71E+01	8.06E+01	1	0.00	1.61E+02
ICP.a.Ba	8.18E+00	2.62E+00	1	0.00	2.88E+01
ICP.a.Bi	2.42E+04	6.31E+06	1	0.00	5.61E+04
ICP.a.Ca	1.04E+03	1.69E+05	1	0.00	6.26E+03
ICP.a.Ce	1.78E+02	3.47E+02	1	0.00	4.15E+02
ICP.a.Cr	9.58E+02	8.49E+03	1	0.00	2.13E+03
ICP.a.Cu	4.28E+01	6.20E+02	1	0.00	3.59E+02
ICP.a.Fe	1.10E+04	2.46E+06	1	0.00	3.09E+04
ICP.a.K	2.63E+02	9.69E+02	1	0.00	6.58E+02
ICP.a.Mg	1.59E+02	1.82E+02	1	0.00	3.30E+02
ICP.a.Mn	3.98E+01	2.90E+01	1	0.00	1.08E+02
ICP.a.Na	1.02E+05	7.32E+05	1	9.07E+04	1.12E+05
ICP.a.Ni*	1.22E+01	2.43E+00	1	0.00	3.20E+01
ICP.a.P	2.31E+04	6.31E+06	1	0.00	5.50E+04
ICP.a.Pb*	6.28E+01	1.08E+02	1	0.00	1.95E+02
ICP.a.S	4.57E+03	9.92E+04	1	5.65E+02	8.57E+03
ICP.a.Se**	3.36E+02	1.04E+03	1	0.00	7.46E+02
ICP.a.Si	1.66E+03	2.74E+03	1	9.99E+02	2.33E+03
ICP.a.Sr	1.67E+02	1.18E+02	1	2.84E+01	3.05E+02
ICP.a.Ti*	3.86E+00	2.20E-01	1	0.00	9.81E+00
ICP.a.Zn	5.95E+01	1.89E+02	1	0.00	2.34E+02
ICP.a.Zr	9.62E+01	2.31E+01	1	3.51E+01	1.57E+02
ICP.w.Al	1.34E+02	2.39E+03	1	0.00	7.56E+02
ICP.w.B	2.50E+01	1.07E+01	1	0.00	6.65E+01
ICP.w.Bi	2.34E+02	1.97E+04	1	0.00	2.02E+03
ICP.w.Ca	4.34E+02	8.96E+04	1	0.00	4.24E+03
ICP.w.Cr	1.51E+02	3.89E+01	1	7.22E+01	2.31E+02
ICP.w.Fe	1.18E+02	4.90E+03	1	0.00	1.01E+03
ICP.w.K	1.47E+02	7.38E+01	1	3.83E+01	2.57E+02
ICP.w.Mg	1.65E+01	8.09E+01	1	0.00	1.31E+02
ICP.w.Na	6.81E+04	6.33E+07	1	0.00	1.69E+05
ICP.w.P	4.79E+03	7.06E+03	1	3.73E+03	5.86E+03
ICP.w.S	4.43E+03	2.56E+04	1	2.40E+03	6.47E+03

*: Analytes with a portion of the data below 3 times the DL.
*: Analytes with a portion of the data below 10 times the DL.

Table 3-1. Concentration Estimate Statistics. (sheet 2 of 3)
(Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Analyte	\bar{y}	$s^2(\bar{y})$	df	95% LL	95% UL
ICP.w.Si	2.94E+02	9.02E+03	1	0.00	1.50E+03
ICP.w.Sr**	2.46E+00	7.24E-01	1	0.00	1.33E+01
ICP.w.Zn**	1.43E+01	1.09E+02	1	0.00	1.47E+02
ICP.f.Al	1.43E+04	1.88E+06	1	0.00	3.17E+04
ICP.f.Bi	2.23E+04	4.19E+06	1	0.00	4.83E+04
ICP.f.Ca**	3.96E+02	3.95E+03	1	0.00	1.19E+03
ICP.f.Cr	9.68E+02	3.99E+03	1	1.66E+02	1.77E+03
ICP.f.Cu	5.16E+01	3.17E+02	1	0.00	2.78E+02
ICP.f.Fe	1.11E+04	1.26E+06	1	0.00	2.53E+04
ICP.f.Mg	1.12E+02	1.33E+02	1	0.00	2.59E+02
ICP.f.Mn	6.46E+01	3.57E+01	1	0.00	1.40E+02
ICP.f.Na	1.00E+05	3.71E+06	1	7.59E+04	1.25E+05
ICP.f.Ni	5.68E+03	4.06E+06	1	0.00	3.13E+04
ICP.f.P	2.34E+04	2.25E+06	1	4.36E+03	4.25E+04
ICP.f.S	4.44E+03	8.99E+04	1	6.30E+02	8.25E+03
ICP.f.Si	6.78E+03	2.31E+05	1	6.80E+02	1.29E+04
ICP.f.Sr	1.68E+02	5.18E+01	1	7.70E+01	2.60E+02
ICP.f.Zn*	8.33E+01	4.67E+02	1	0.00	3.58E+02
ICP.f.Zr	1.36E+02	7.36E+02	1	0.00	4.80E+02
Dir.XTGA	5.91E+01	7.87E+00	1	2.35E+01	9.48E+01
Dir.%Water	5.67E+01	4.90E-01	1	4.78E+01	6.56E+01
Dir.CN	2.49E+00	1.48E-01	1	0.00	7.37E+00
IC.w.F	9.19E+03	5.27E+05	1	0.00	1.84E+04
IC.w.Cl	1.14E+03	1.05E+03	1	7.24E+02	1.55E+03
IC.w.NO3	1.37E+05	8.10E+07	1	2.24E+04	2.51E+05
IC.w.NO2	8.12E+03	5.85E+06	1	0.00	3.89E+04
IC.w.PO4	1.42E+04	1.91E+05	1	8.68E+03	1.98E+04
IC.w.SO4	1.34E+04	5.44E+05	1	3.99E+03	2.27E+04
Spec.w.NO2	1.23E+04	4.71E+05	1	3.57E+03	2.10E+04
pH	9.71E+00	3.45E-03	1	8.96E+00	1.05E+01
TIC.w.CO3**	1.16E+03	8.49E+03	1	0.00	2.33E+03
Fluor.U*	7.68E+03	3.32E+07	1	0.00	8.08E+04

*: Analytes with a portion of the data below 3 times the DL.
*: Analytes with a portion of the data below 10 times the DL.

Table 3-1. Concentration Estimate Statistics. (sheet 3 of 3)
 (Units $\mu\text{g/g}$ Except Radionuclides $\mu\text{Ci/g}$)

Analyte	\bar{y}	$s^2(\bar{y})$	df	95% LL	95% UL
RAD.f.AT	9.63E-02	2.80E-04	1	0.00	3.09E-01
RAD.f.TB	4.67E+01	1.39E+01	1	0.00	9.41E+01
GEA.Cs-137	1.74E+01	2.55E+01	1	0.00	8.16E+01
Pu-239/240	5.72E-02	6.11E-05	1	0.00	1.56E-01
Am-241	1.30E-02	9.33E-07	1	6.98E-04	2.52E-02
Tc-99	3.69E-02	1.22E-05	1	0.00	8.14E-02
Sr-90	9.87E+00	5.86E-01	1	1.46E-01	1.96E+01
H-3*	5.77E-04	1.39E-08	1	0.00	2.07E-03
C-14**	2.61E-04	NA	1	NA	NA

•: Analytes with a portion of the data below 3 times the DL.
 *: Analytes with a portion of the data below 10 times the DL.

4.0 HOMOGENIZATION TEST

A second task in complying with the Tank Waste Characterization Plan, Section 5.1.1.7 (Bell 1993) was to evaluate the ability of the 222-S Analytical Laboratory to homogenize segments.

Segments 2 and 6 from core 41 were homogenized and arbitrarily divided into two parts. One subsample was obtained from each part. Two aliquots were taken from each subsample and prepared for chemical analysis. The homogenization test data are given in Table A-2 of Appendix A. The ICP acid digestion and GEA analyses were conducted on the samples.

4.1 STATISTICAL METHODS AND RESULTS

Due to the nested structure (subsamples within segments, aliquots within subsamples) of the data, a hierarchical statistical model was fit to the data. Snedecor (1980), page 284, contains a description of this type of model. This type of model is used to estimate different components of variability in the data. This model is explicitly described in Section 5.3 of Jensen and Remund (1993). The total variability in the data is decomposed into three components: 1) one due to variability between segments, 2) one due to the variability between samples taken from different locations within each homogenized segment [$\sigma^2(L)$], and 3) one due to the analytical measurement error [$\sigma^2(A)$]. The analytical measurement error accounts for the differences between aliquots taken from the same location.

To quantify the contribution of $\sigma^2(L)$ (the component of variability due to location or homogenization), the analysis of variance (ANOVA) corresponding to the hierarchical model is used. From the ANOVA, an F-test is used to test the hypothesis that $\sigma^2(L)$ is equal to zero. If $\sigma^2(L)$ is significantly different from zero, then the laboratory does not have the ability to homogenize segments. If $\sigma^2(L)$ is not significantly different from zero, then the laboratory has the ability to homogenize core segments. The reason underlying this test is that if $\sigma^2(L)=0$, then the mean concentrations at the two locations are equal (i.e., there is no difference between the locations).

An F-test, with 2 and 4 degrees of freedom, is used to determine whether or not $\sigma^2(L)$ is significantly different from zero. The p-values (the attained level of significance) from these tests are shown in Table 4-1. If the p-value is smaller than 0.05, then $\sigma^2(L)$ is significantly different from zero. In 23 out of 25 cases (92%), the p-values are more than 0.05. This indicates that, except for two cases, $\sigma^2(L)$ is not significantly different from zero. The two cases where the p-values are less than 0.05 were ICP.a.Se and GEA.Cs-137. The differences between sampled locations for GEA.Cs-137 are statistically different but may not be practically different. Based on the results of this statistical test, it can be concluded that the 222-S Analytical Laboratory can adequately homogenize core segments, with the exception of the two cases noted above. However, it needs to be noted that there is no reference value available to check the degree of homogenization (e.g., the differences between the results from the two locations must be within 2% of each other). If such a value were available, the conclusions in this section may not be valid.

Table 4-1. Homogenization Test Statistical Results.

Analyte	Test: $\sigma^2(L)=0$ p-value
ICP.a.Al	0.207
ICP.a.B	0.068
ICP.a.Ba	0.896
ICP.a.Bi	0.273
ICP.a.Ca	0.204
ICP.a.Ce	0.910
ICP.a.Cr	0.683
ICP.a.Cu	0.792
ICP.a.Fe	0.668
ICP.a.K	0.343
ICP.a.Mg	0.370
ICP.a.Mn	0.899
ICP.a.Na	0.060
ICP.a.Ni	0.115
ICP.a.P	0.070
ICP.a.Pb	0.553
ICP.a.S	0.301
ICP.a.Se	0.025
ICP.a.Si	0.265
ICP.a.Sr	0.492
ICP.a.Ti	0.841
ICP.a.Zn	0.308
ICP.a.Zr	0.651
RAD.a.TA	0.236
GEA.Cs-137	0.000

5.0 COMPARISON OF THE VARIANCE COMPONENT ESTIMATES

Using the hierarchical structure of the core composite data, the between core spatial variability, the compositing variability, and the analytical measurement variability can be separated from each other. The spatial variance is a measure of the variability between cores. The compositing variance measures the variability between composite samples within the same core. The analytical measurement variance includes the segment homogenization error, the sample handling error, and the chemical analysis error. This variance is a measure of the difference between the analytical results from the primary and duplicate samples.

The estimate of the variance of the mean is a linear function of the spatial, compositing, and analytical measurement variances. To help evaluate the magnitude of these three variance components, explicit estimates of each variance component are given in this section.

5.1 STATISTICAL METHODS

Estimates of the spatial variance [$\hat{\sigma}^2(S)$], compositing variance [$\hat{\sigma}^2(C)$], and analytical measurement variance [$\hat{\sigma}^2(A)$] were obtained for each analyte using Restricted Maximum Likelihood Estimation (REML) methods. This method is discussed by Harville (1977).

An ANOVA was computed for a hierarchical statistical model. Appendix C includes a description of the model. An F-test from the ANOVA was used to test the hypothesis that $\sigma^2(S)$ is equal to zero (i.e., the tank is homogeneous). Another F-test from the ANOVA was used to test the hypothesis that there is not a significant difference between composite samples within the same core [$\hat{\sigma}^2(C)=0$]. If $\hat{\sigma}^2(C)$ is significantly different from zero, then it is concluded that the laboratory does not have the ability to create reproducible core composite samples. If $\hat{\sigma}^2(C)$ is not significantly different from zero, then it is concluded that the laboratory has the ability to create reproducible core composites.

5.2 STATISTICAL RESULTS

The REML estimates of each component of variability along with the p-values (the attained level of significance) from the F-tests are shown in Table 5-1. If the p-value is smaller than 0.05, then $\hat{\sigma}^2(S)$ or $\hat{\sigma}^2(C)$ are significantly different from zero.

For 23 (31%) of 74 analytes tested, the p-values from the tests on $\hat{\sigma}^2(S)$ are less than 0.05. For these 23 cases, the spatial variability is significantly greater than zero (i.e., we conclude that BX107 is heterogeneous with respect to these analytes).

In 33 (45%) of 74 cases tested, the p-values from the tests on $\hat{\sigma}^2(C)$ are less than 0.05. For nearly all analytes from IC and ICP KOH/Ni fusion methods, $\hat{\sigma}^2(C)$ was significantly greater than zero. Based on the core composite data from BX107, the 222-S Analytical Laboratory can adequately create reproducible core composites for 41 (55%) of the 77 analytes tested.

Table 5-1. Variance Components Estimates. (sheet 1 of 3)

Analyte	$\sigma^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\sigma^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\sigma^2(A)$
ICP.a.Al	9.09E+06	0.000	3.63E+04	0.302	1.14E+05
ICP.a.B	6.05E+01	0.333	1.74E+02	0.046	5.47E+01
ICP.a.Ba	5.11E+00	0.000	2.02E-01	0.133	1.62E-01
ICP.a.Bi	1.24E+07	0.000	2.88E+05	0.157	2.81E+05
ICP.a.Ca	2.70E-32	0.486	6.63E+04	0.355	1.22E+06
ICP.a.Ce	6.33E+02	0.078	1.17E+02	0.004	8.63E+00
ICP.a.Cr	1.67E+04	0.000	2.04E+02	0.303	6.45E+02
ICP.a.Cu	1.23E+03	0.010	2.34E+01	0.010	2.82E+00
ICP.a.Fe	4.88E+06	0.000	2.31E+04	0.338	1.05E+05
ICP.a.K	1.33E+03	0.216	1.18E+03	0.003	6.86E+01
ICP.a.Mg	1.35E-12	0.941	2.72E+02	0.208	9.14E+02
ICP.a.Mn	5.54E+01	0.001	3.40E+00	0.171	3.71E+00
ICP.a.Na	1.86E-05	0.802	2.04E+06	0.085	1.78E+06
ICP.a.Ni*	4.77E+00	0.000	4.86E-33	0.609	3.52E-01
ICP.a.P	1.10E+07	0.109	3.05E+06	0.015	4.59E+05
ICP.a.Pb*	2.07E+02	0.001	1.35E+01	0.130	1.05E+01
ICP.a.S	1.66E+05	0.131	6.30E+04	0.002	3.13E+03
ICP.a.Se**	2.00E+03	0.005	7.07E+01	0.256	1.48E+02
ICP.a.Si	5.43E-24	0.531	5.58E-19	0.495	2.19E+04
ICP.a.Sr	2.07E+02	0.007	4.60E+01	0.096	2.66E+01
ICP.a.Ti*	3.84E-01	0.009	7.52E-02	0.153	7.09E-02
ICP.a.Zn	2.94E+02	0.047	6.87E+01	0.292	1.96E+02
ICP.a.Zr	3.69E-21	0.596	3.82E+00	0.349	1.77E+02
ICP.w.Al	1.16E-11	0.512	8.30E+03	0.036	2.56E+03
ICP.w.B	2.07E+01	0.030	1.29E+00	0.006	1.12E-01
ICP.w.Bi	5.38E+03	0.154	5.77E+04	0.055	2.08E+04
ICP.w.Ca	1.62E+05	0.017	2.39E-22	0.989	6.70E+04
ICP.w.Cr	2.02E+00	0.200	1.15E+02	0.106	7.33E+01
ICP.w.Fe	1.33E+03	0.156	1.42E+04	0.059	5.43E+03
ICP.w.K	1.52E-21	0.684	2.77E+02	0.007	3.52E+01
ICP.w.Mg	1.21E+02	0.049	4.18E+01	0.242	7.84E+01
ICP.w.Na	3.84E+07	0.276	9.61E-28	0.775	3.52E+08

*: Analytes with a portion of the data below 3 times the DL.
 **: Analytes with a portion of the data below 10 times the DL.

Table 5-1. Variance Component Estimates. (sheet 2 of 3)

Analyte	$\sigma^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\sigma^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\sigma^2(A)$
ICP.w.P	9.52E+02	0.281	9.27E+03	0.318	3.41E+04
ICP.w.S	4.31E+04	0.128	1.48E+04	0.023	2.87E+03
ICP.w.Si	1.57E+04	0.019	1.51E+03	0.328	6.14E+03
ICP.w.Sr**	9.27E-28	0.244	2.30E+00	0.075	1.19E+00
ICP.w.Zn**	1.85E+02	0.043	8.41E-28	0.976	1.34E+02
ICP.f.Al	2.05E+06	0.276	3.03E+06	0.033	7.62E+05
ICP.f.Bi	4.00E-17	0.703	1.62E+07	0.002	1.06E+06
ICP.f.Ca**	3.46E+03	0.314	8.59E+03	0.004	5.80E+02
ICP.f.Cr	4.56E+03	0.266	5.97E+03	0.042	1.75E+03
ICP.f.Cu	6.05E+02	0.002	1.20E+01	0.378	9.40E+01
ICP.f.Fe	1.35E+06	0.280	2.15E+06	0.019	3.78E+05
ICP.f.Mg	1.80E+02	0.222	1.49E+02	0.048	4.89E+01
ICP.f.Mn	5.84E+01	0.053	2.25E+00	0.418	4.77E+01
ICP.f.Na	1.36E+05	0.419	1.29E+07	0.035	3.32E+06
ICP.f.Ni	5.87E-08	0.510	1.29E+07	0.053	6.58E+06
ICP.f.P	5.70E-18	0.627	7.91E+06	0.025	2.20E+06
ICP.f.S	1.75E+05	0.000	3.89E+03	0.274	9.49E+03
ICP.f.Si	1.53E+05	0.346	5.27E+05	0.050	1.77E+05
ICP.f.Sr	1.05E-20	0.607	1.59E+02	0.061	9.55E+01
ICP.f.Zn*	7.01E+02	0.183	4.26E+02	0.022	8.06E+01
ICP.f.Zr	4.37E-11	0.740	1.98E+03	0.096	1.92E+03
Dir.XTGA	3.22E-29	0.825	2.94E+01	0.007	4.17E+00
Dir.XWater	9.24E-01	0.001	8.25E-02	0.116	5.75E-02
Dir.CN	2.06E-24	0.670	5.23E-01	0.022	1.36E-01
IC.w.F	5.18E+05	0.296	1.02E+06	0.007	9.77E+04
IC.w.Cl	1.06E-11	0.488	3.59E+03	0.044	1.24E+03
IC.w.NO3	1.53E+08	0.002	9.00E+06	0.237	1.63E+07
IC.w.NO2	3.46E-19	0.690	2.33E+07	0.000	1.35E+05
IC.w.PO4	2.65E+05	0.213	2.25E+05	0.007	2.13E+04
IC.w.SO4	1.02E+06	0.060	1.31E+05	0.020	2.38E+04
Spec.w.NO2	6.78E-12	0.527	1.75E+06	0.011	2.59E+05

•: Analytes with a portion of the data below 3 times the DL.
 *: Analytes with a portion of the data below 10 times the DL.

Table 5-1. Variance Component Estimates. (sheet 3 of 3)

Analyte	$\sigma^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\sigma^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\sigma^2(A)$
pH	4.08E-03	0.169	4.81E-20	0.641	1.13E-02
TIC.W.CO3**	1.08E-43	0.914	1.04E-34	0.914	6.80E+04
Fluor.U*	9.98E+06	0.391	1.12E+08	0.000	7.07E+05
RAD.f.AT	5.21E-04	0.003	4.37E-05	0.218	6.84E-05
RAD.f.TB	2.51E-21	0.427	5.15E+01	0.016	8.15E+00
GEA.Cs-137	4.79E+01	0.056	6.17E+00	0.001	1.67E-01
Pu-239/240	5.30E-39	0.658	2.14E-04	0.025	6.06E-05
Am-241	8.91E-31	0.300	1.87E-06	0.235	3.73E-06
Tc-99	7.69E-22	0.439	4.28E-05	0.039	1.24E-05
Sr-90	4.00E-24	0.430	2.16E+00	0.017	3.61E-01
H-3**	4.37E-24	0.521	4.07E-08	0.080	2.97E-08
C-14**	NA	NA	NA	NA	NA

•: Analytes with a portion of the data below 3 times the DL.
 *: Analytes with a portion of the data below 10 times the DL.

6.0 REFERENCES

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APPENDIX A
CORE COMPOSITE AND HOMOGENIZATION TEST DATA

A-1

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Table A-1. Core Composite Data. (sheet 1 of 9)
 (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$)

Analyte	Core 40 Comp. 1	Mean/DL	Core 40 Comp. 2	Mean/DL	Core 41 Comp. 1	Mean/DL	Core 41 Comp. 2	Mean/DL
ICP.a.Ag	9.50E-01 9.14E-01	9.32E-01	9.43E-01 9.62E-01	9.52E-01	9.49E-01 9.73E-01	9.61E-01	9.14E-01 9.33E-01	9.23E-01
ICP.a.Al	1.23E+04 1.14E+04	3.40E+03	1.25E+04 1.22E+04	3.51E+03	1.62E+04 1.62E+04	4.63E+03	1.66E+04 1.65E+04	4.74E+03
ICP.a.As	2.85E+00 2.74E+00	9.33E-01	2.83E+00 2.88E+00	9.53E-01	3.13E+00 2.92E+00	1.01E+00	2.74E+00 2.80E+00	9.23E-01
ICP.a.B	4.70E+01 5.25E+01	4.98E+01	6.85E+01 5.61E+01	6.23E+01	5.76E+01 4.40E+01	5.08E+01	2.12E+01 2.95E+01	2.53E+01
ICP.a.Ba	7.28E+00 6.22E+00	2.25E+01	6.33E+00 6.42E+00	2.13E+01	1.01E+01 1.05E+01	3.43E+01	9.34E+00 9.27E+00	3.10E+01
ICP.a.Be	2.85E-01 2.74E-01	9.33E-01	2.83E-01 2.88E-01	9.53E-01	2.85E-01 2.92E-01	9.60E-01	2.74E-01 2.80E-01	9.23E-01
ICP.a.Bi	2.19E+04 2.05E+04	3.66E+03	2.22E+04 2.20E+04	3.81E+03	2.73E+04 2.70E+04	4.67E+03	2.64E+04 2.60E+04	4.52E+03
ICP.a.Ca	3.50E+03 7.86E+02	7.87E+01	3.65E+02 7.30E+02	2.01E+01	4.16E+02 1.92E+03	4.30E+01	3.06E+02 2.86E+02	1.09E+01
ICP.a.Cd	2.04E+00 1.67E+00	2.66E+00	2.03E+00 1.83E+00	2.76E+00	2.70E+00 2.77E+00	3.91E+00	2.69E+00 2.43E+00	3.66E+00
ICP.a.Ce	1.54E+02 1.47E+02	1.54E+01	1.69E+02 1.69E+02	1.72E+01	1.92E+02 1.90E+02	1.95E+01	2.05E+02 2.01E+02	2.07E+01
ICP.a.Co	2.22E+00 1.94E+00	2.08E+00	1.64E+00 1.75E+00	1.69E+00	2.09E+00 2.55E+00	2.32E+00	9.14E-01 9.88E-01	9.51E-01
ICP.a.Cr	8.89E+02 8.20E+02	7.12E+02	8.77E+02 8.77E+02	7.31E+02	1.07E+03 1.07E+03	8.92E+02	1.04E+03 1.02E+03	8.58E+02
ICP.a.Cu	6.51E+01 6.04E+01	1.57E+02	7.24E+01 7.27E+01	1.81E+02	1.86E+01 1.89E+01	4.67E+01	1.72E+01 1.67E+01	4.25E+01
ICP.a.Fe	9.86E+03 9.04E+03	6.30E+03	9.43E+03 9.42E+03	6.29E+03	1.28E+04 1.29E+04	8.60E+03	1.25E+04 1.21E+04	8.20E+03
ICP.a.K	2.73E+02 2.56E+02	2.05E+01	1.99E+02 1.99E+02	1.54E+01	2.99E+02 3.13E+02	2.37E+01	2.78E+02 2.86E+02	2.19E+01

NA: Not available.

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Table A-1. Core Composite Data. (sheet 2 of 9)
 (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$)

Analyte	Core 40 Comp. 1	Mean/DL	Core 40 Comp. 2	Mean/DL	Core 41 Comp. 1	Mean/DL	Core 41 Comp. 2	Mean/DL
ICP.a.La	1.52E+00 1.46E+00	9.31E-01	1.51E+00 1.54E+00	9.50E-01	1.52E+00 1.56E+00	9.63E-01	1.46E+00 1.49E+00	9.25E-01
ICP.a.Mg	2.02E+02 1.37E+02	2.43E+02	1.56E+02 1.43E+02	2.13E+02	1.62E+02 2.16E+02	2.70E+02	1.25E+02 1.27E+02	1.80E+02
ICP.a.Mn	3.78E+01 3.30E+01	1.18E+02	3.32E+01 3.35E+01	1.11E+02	4.62E+01 4.82E+01	1.57E+02	4.39E+01 4.23E+01	1.44E+02
ICP.a.Na	1.02E+05 9.85E+04	3.57E+04	1.03E+05 1.02E+05	3.64E+04	1.04E+05 1.03E+05	3.68E+04	1.00E+05 1.00E+05	3.57E+04
ICP.a.Ni	1.16E+01 1.03E+01	7.79E+00	1.08E+01 9.93E+00	7.43E+00	1.36E+01 1.43E+01	1.00E+01	1.39E+01 1.33E+01	9.71E+00
ICP.a.P	1.95E+04 2.00E+04	1.99E+03	2.21E+04 2.06E+04	2.15E+03	2.44E+04 2.35E+04	2.41E+03	2.69E+04 2.75E+04	2.75E+03
ICP.a.Pb	5.55E+01 5.30E+01	7.65E+00	5.49E+01 4.61E+01	7.11E+00	7.75E+01 7.67E+01	1.09E+01	6.92E+01 6.94E+01	9.76E+00
ICP.a.S	4.76E+03 4.76E+03	1.06E+03	5.07E+03 4.94E+03	1.11E+03	4.52E+03 4.43E+03	9.93E+02	4.03E+03 4.03E+03	8.96E+02
ICP.a.Sb	3.48E+01 3.35E+01	9.32E-01	3.45E+01 3.52E+01	9.54E-01	3.47E+01 3.56E+01	9.62E-01	3.35E+01 3.41E+01	9.23E-01
ICP.a.Se	3.26E+02 2.99E+02	3.76E+01	2.90E+02 3.01E+02	3.55E+01	3.66E+02 3.72E+02	4.45E+01	7.59E+00 7.74E+00	9.23E-01
ICP.a.Si	1.66E+03 1.41E+03	3.58E+02	1.63E+03 1.81E+03	4.00E+02	1.63E+03 1.86E+03	4.05E+02	1.77E+03 1.54E+03	3.86E+02
ICP.a.Sn	3.34E+00 3.23E+00	1.43E+00	2.17E+00 1.04E+01	2.74E+00	5.34E+00 9.04E+00	3.13E+00	1.07E+01 1.17E+01	4.87E+00
ICP.a.Sr	1.61E+02 1.47E+02	3.85E+02	1.58E+02 1.57E+02	3.92E+02	1.85E+02 1.85E+02	4.62E+02	1.72E+02 1.68E+02	4.25E+02
ICP.a.Tl	3.18E+00 3.18E+00	7.95E+00	3.72E+00 3.47E+00	8.97E+00	4.23E+00 4.94E+00	1.15E+01	4.08E+00 4.05E+00	1.01E+01
ICP.a.V	NA NA	NA	NA NA	NA	NA NA	NA	NA NA	NA

NA: Not available.

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Table A-1. Core Composite Data. (sheet 3 of 9)
 (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$)

Analyte	Core 40 Comp. 1	Mean/DL	Core 40 Comp. 2	Mean/DL	Core 41 Comp. 1	Mean/DL	Core 41 Comp. 2	Mean/DL
ICP.a.Zn	7.50E+01 3.72E+01	2.24E+01	3.13E+01 3.97E+01	1.42E+01	7.71E+01 8.50E+01	3.24E+01	6.65E+01 6.45E+01	2.62E+01
ICP.a.Zr	1.12E+02 7.72E+01	1.18E+02	8.72E+01 9.71E+01	1.15E+02	8.64E+01 8.95E+01	1.10E+02	1.15E+02 1.05E+02	1.38E+02
ICP.w.Ag	1.02E+00 1.02E+00	1.02E+00	1.02E+00 1.02E+00	1.02E+00	1.02E+00 1.02E+00	1.02E+00	1.02E+00 1.02E+00	1.02E+00
ICP.w.Al	1.15E+02 9.57E+01	3.01E+01	8.00E+01 8.07E+01	2.30E+01	7.33E+01 6.99E+01	2.11E+01	3.50E+02 2.09E+02	7.98E+01
ICP.w.As	3.06E+00 3.05E+00	1.02E+00	3.05E+00 3.06E+00	1.02E+00	3.16E+00 3.16E+00	1.02E+00	3.05E+00 3.05E+00	1.02E+00
ICP.w.B	2.79E+01 2.78E+01	2.79E+01	2.89E+01 2.85E+01	2.87E+01	2.05E+01 2.09E+01	1.09E+01	2.32E+01 2.25E+01	2.28E+01
ICP.w.Be	4.52E-01 3.05E-01	1.26E+00	3.89E-01 6.74E-01	1.77E+00	3.06E-01 3.05E-01	1.02E+00	6.36E-01 4.69E-01	1.84E+00
ICP.w.Be	3.06E-01 3.05E-01	1.02E+00	3.05E-01 3.06E-01	1.02E+00	3.06E-01 3.05E-01	1.02E+00	3.05E-01 3.05E-01	1.02E+00
ICP.w.Bi	9.96E+01 1.13E+02	1.83E+01	7.83E+01 8.21E+01	1.38E+01	1.16E+02 1.11E+02	1.33E+01	8.39E+02 4.31E+02	1.09E+02
ICP.w.Ca	1.13E+03 3.71E+02	2.77E+01	9.47E+02 4.80E+02	2.62E+01	1.41E+02 1.54E+02	6.12E+01	1.36E+02 1.05E+02	4.45E+00
ICP.w.Cd	7.14E-01 7.13E-01	1.02E+00	7.13E-01 7.14E-01	1.02E+00	7.14E-01 7.13E-01	1.02E+00	7.11E-01 7.11E-01	1.02E+00
ICP.w.Ce	1.00E+01 9.98E+00	1.02E+00	9.98E+00 1.00E+01	1.02E+00	8.77E+00 8.75E+00	1.02E+00	9.96E+00 9.96E+00	1.02E+00
ICP.w.Co	1.02E+00 1.02E+00	1.02E+00	1.02E+00 1.02E+00	1.02E+00	1.22E+00 1.22E+00	1.02E+00	1.17E+00 1.02E+00	1.09E+00
ICP.w.Cr	1.58E+02 1.57E+02	1.31E+02	1.31E+02 1.35E+02	1.11E+02	1.54E+02 1.58E+02	1.11E+02	1.71E+02 1.48E+02	1.33E+02
ICP.w.Cu	6.87E-01 7.66E-01	1.82E+00	7.72E-01 7.80E-01	1.94E+00	6.12E-01 6.11E-01	1.02E+00	1.07E+00 6.70E-01	2.18E+00

NA: Not available.

B-29

A-5

WHC-SD-WM-TI-603, Rev. 0

WHC-EP-0739

9513322.1426

Table A-1. Core Composite Data. (sheet 4 of 9)
(Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$)

Analyte	Core 40 Comp. 1	Mean/DL	Core 40 Comp. 2	Mean/DL	Core 41 Comp. 1	Mean/DL	Core 41 Comp. 2	Mean/DL
ICP.w.Fe	5.16E+01 5.78E+01	3.65E+01	4.27E+01 4.12E+01	2.80E+01	6.15E+01 5.52E+01	2.92E+01	4.22E+02 2.14E+02	2.12E+02
ICP.w.K	1.70E+02 1.64E+02	1.30E+01	1.31E+02 1.43E+02	1.06E+01	1.51E+02 1.61E+02	1.17E+01	1.28E+02 1.31E+02	1.00E+01
ICP.w.La	1.63E+00 1.63E+00	1.02E+00	1.63E+00 1.63E+00	1.02E+00	2.14E+00 2.14E+00	1.02E+00	1.63E+00 1.63E+00	1.02E+00
ICP.w.Mg	2.43E+01 8.72E+00	2.36E+01	4.41E+01 2.47E+01	4.91E+01	6.59E+00 6.43E+00	1.08E+01	9.57E+00 7.25E+00	1.20E+01
ICP.w.Mn	1.53E+00 6.31E-01	3.61E+00	1.58E+00 9.12E-01	4.16E+00	3.06E-01 3.05E-01	1.02E+00	1.72E+00 8.88E-01	4.34E+00
ICP.w.Na	2.63E+04 8.03E+04	1.90E+04	5.29E+04 8.11E+04	2.39E+04	7.94E+04 8.08E+04	9.21E+03	7.26E+04 7.14E+04	2.57E+04
ICP.w.Ni	1.43E+00 1.43E+00	1.02E+00	1.43E+00 1.43E+00	1.02E+00	1.84E+00 1.83E+00	1.02E+00	1.42E+00 1.42E+00	1.02E+00
ICP.w.P	4.77E+03 4.74E+03	4.80E+02	5.26E+03 4.74E+03	5.05E+02	4.56E+03 4.65E+03	5.75E+02	4.81E+03 4.81E+03	4.86E+02
ICP.w.Pb	7.24E+00 7.23E+00	1.02E+00	7.23E+00 7.24E+00	1.02E+00	8.67E+00 8.65E+00	1.02E+00	7.49E+00 8.04E+00	1.09E+00
ICP.w.S	4.54E+03 4.57E+03	1.01E+03	4.69E+03 4.57E+03	1.03E+03	4.35E+03 4.44E+03	7.32E+02	4.17E+03 4.13E+03	9.23E+02
ICP.w.Sb	3.73E+01 3.73E+01	1.02E+00	3.73E+01 3.73E+01	1.02E+00	3.63E+01 3.62E+01	1.02E+00	3.72E+01 3.72E+01	1.02E+00
ICP.w.Se	8.47E+00 8.45E+00	1.02E+00	8.45E+00 8.47E+00	1.02E+00	1.34E+01 1.33E+01	1.02E+00	7.45E+01 6.20E+01	8.22E+00
ICP.w.Si	5.24E+02 3.03E+02	9.62E+01	3.68E+02 3.61E+02	8.47E+01	1.33E+02 1.39E+02	5.91E+01	2.71E+02 2.53E+02	6.09E+01
ICP.w.Sn	2.35E+00 2.34E+00	1.02E+00	2.34E+00 2.35E+00	1.02E+00	1.63E+00 1.63E+00	1.02E+00	2.34E+00 2.34E+00	1.02E+00
ICP.w.Sr	1.99E+00 1.41E+00	4.25E+00	1.90E+00 1.43E+00	4.16E+00	1.48E+00 1.44E+00	2.43E+00	6.50E+00 3.51E+00	1.25E+01

NA: Not available.

B-30

A-6

WHC-SD-WM-TI-603, Rev. 0

WHC-EP-0739

Table A-1. Core Composite Data. (sheet 5 of 9)
 (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$)

Analyte	Core 40 Comp. 1	Mean/DL	Core 40 Comp. 2	Mean/DL	Core 41 Comp. 1	Mean/DL	Core 41 Comp. 2	Mean/DL
ICP.w.Tl	4.08E-01 4.07E-01	1.02E+00	4.07E-01 4.08E-01	1.02E+00	5.10E-01 5.09E-01	1.02E+00	4.06E-01 4.06E-01	1.02E+00
ICP.w.V	8.16E-01 8.14E-01	1.02E+00	8.14E-01 8.16E-01	1.02E+00	NA NA	NA	NA NA	NA
ICP.w.Zn	4.33E+01 7.94E+00	1.03E+01	3.31E+01 1.46E+01	9.53E+00	2.55E+00 2.54E+00	1.02E+00	5.28E+00 4.95E+00	2.05E+00
ICP.w.Zr	1.49E+00 1.60E+00	NA	8.14E-01 1.40E+00	NA	1.12E+00 1.30E+00	1.10E+00	7.85E+00 4.33E+00	NA
ICP.f.Ag	4.99E+00 4.99E+00	9.98E-01	8.48E+00 5.57E+00	1.40E+00	7.59E+00 4.98E+00	6.29E-01	5.92E+00 4.99E+00	1.09E+00
ICP.f.Al	1.34E+04 1.43E+04	7.92E+02	1.12E+04 1.28E+04	6.85E+02	1.72E+04 1.73E+04	4.93E+02	1.33E+04 1.49E+04	8.04E+02
ICP.f.As	1.50E+01 1.50E+01	9.98E-01	1.50E+01 1.50E+01	9.99E-01	1.49E+01 1.49E+01	4.98E-01	1.50E+01 1.50E+01	9.99E-01
ICP.f.B	1.61E+01 2.06E+01	3.67E+00	2.01E+01 1.75E+01	3.76E+00	8.32E+00 4.98E+00	6.65E-01	1.88E+01 1.35E+01	3.23E+00
ICP.f.Ba	4.17E+00 7.97E+00	4.05E+00	8.35E+00 7.78E+00	5.38E+00	1.20E+01 1.16E+01	3.94E+00	1.06E+01 1.05E+01	7.04E+00
ICP.f.Be	1.50E+00 1.50E+00	9.98E-01	1.50E+00 1.50E+00	9.99E-01	1.49E+00 1.49E+00	4.98E-01	1.50E+00 1.50E+00	9.99E-01
ICP.f.Bi	2.13E+04 2.25E+04	7.54E+02	1.93E+04 2.19E+04	7.10E+02	2.82E+04 2.80E+04	4.84E+02	1.89E+04 1.83E+04	6.41E+02
ICP.f.Ca	3.43E+02 3.77E+02	2.65E+00	3.03E+02 3.10E+02	2.25E+00	3.65E+02 3.72E+02	1.36E+00	5.20E+02 5.78E+02	4.04E+00
ICP.f.Cd	3.49E+00 3.87E+00	1.05E+00	3.49E+00 3.53E+00	1.00E+00	4.73E+00 3.96E+00	6.21E-01	4.30E+00 6.22E+00	1.50E+00
ICP.f.Ce	4.89E+01 1.35E+02	1.88E+00	2.11E+02 1.44E+02	3.62E+00	2.30E+02 1.72E+02	2.05E+00	2.52E+02 2.06E+02	4.67E+00
ICP.f.Co	4.99E+00 4.99E+00	9.98E-01	4.99E+00 5.00E+00	9.99E-01	4.98E+00 4.98E+00	4.98E-01	9.68E+00 1.59E+01	2.56E+00

NA: Not available.

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A-7

WHC-SD-MN-TI-603, Rev. 0

WHC-EP-0739

950322.142

Table A-1. Core Composite Data. (sheet 6 of 9)
(Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$)

Analyte	Core 40 Comp. 1	Mean/DL	Core 40 Comp. 2	Mean/DL	Core 41 Comp. 1	Mean/DL	Core 41 Comp. 2	Mean/DL
ICP.f.Cr	9.54E+02 9.90E+02	1.62E+02	7.87E+02 8.89E+02	1.40E+02	1.08E+03 1.08E+03	9.00E+01	9.58E+02 1.01E+03	1.64E+02
ICP.f.Cu	6.10E+01 7.36E+01	3.36E+01	7.27E+01 7.05E+01	3.58E+01	2.59E+01 2.71E+01	6.62E+00	5.33E+01 2.91E+01	2.06E+01
ICP.f.Fe	1.07E+04 1.13E+04	1.47E+03	8.35E+03 9.39E+03	1.18E+03	1.34E+04 1.32E+04	8.85E+02	1.05E+04 1.17E+04	1.48E+03
ICP.f.K	NA NA	NA	NA NA	NA	NA NA	NA	NA NA	NA
ICP.f.La	7.98E+00 7.98E+00	9.98E-01	7.98E+00 8.00E+00	9.99E-01	7.97E+00 7.97E+00	4.98E-01	8.00E+00 7.98E+00	9.99E-01
ICP.f.Mg	1.08E+02 1.14E+02	3.17E+01	8.55E+01 9.40E+01	2.57E+01	1.31E+02 1.32E+02	1.88E+01	1.07E+02 1.24E+02	3.31E+01
ICP.f.Mn	6.51E+01 6.05E+01	4.19E+01	4.99E+01 5.89E+01	3.63E+01	6.52E+01 8.16E+01	2.45E+01	6.60E+01 6.92E+01	4.51E+01
ICP.f.Na	1.02E+05 1.06E+05	7.42E+03	9.97E+04 1.02E+05	7.19E+03	1.01E+05 1.03E+05	3.64E+03	9.45E+04 9.56E+04	6.79E+03
ICP.f.Ni	8.52E+03 5.89E+03	1.03E+03	2.48E+03 1.50E+03	2.84E+02	4.30E+03 1.42E+03	2.04E+02	7.64E+03 1.37E+04	1.52E+03
ICP.f.P	2.39E+04 2.44E+04	4.88E+02	1.94E+04 2.21E+04	4.19E+02	2.59E+04 2.88E+04	2.76E+02	2.09E+04 2.20E+04	4.33E+02
ICP.f.Pb	3.54E+01 5.45E+01	1.27E+00	6.56E+01 6.48E+01	1.84E+00	1.23E+02 7.58E+01	1.40E+00	1.01E+02 7.51E+01	2.49E+00
ICP.f.S	4.79E+03 4.87E+03	2.15E+02	4.65E+03 4.65E+03	2.07E+02	4.03E+03 4.26E+03	9.22E+01	4.20E+03 4.06E+03	1.84E+02
ICP.f.Sb	1.83E+02 1.83E+02	9.98E-01	1.83E+02 1.83E+02	9.99E-01	1.82E+02 1.82E+02	4.98E-01	1.83E+02 1.83E+02	9.99E-01
ICP.f.Se	4.14E+01 4.14E+01	9.98E-01	4.14E+01 4.15E+01	9.99E-01	4.13E+01 4.13E+01	4.98E-01	3.16E+02 3.45E+02	7.96E+00
ICP.f.Si	6.43E+03 6.77E+03	3.07E+02	5.63E+03 6.37E+03	2.79E+02	7.98E+03 7.99E+03	1.86E+02	6.10E+03 6.97E+03	3.04E+02

NA: Not available.

Table A-1. Core Composite Data. (sheet 7 of 9)
 (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$)

Analyte	Core 40 Comp. 1	Mean/DL	Core 40 Comp. 2	Mean/DL	Core 41 Comp. 1	Mean/DL	Core 41 Comp. 2	Mean/DL
ICP.f.Sn	1.15E+01 1.15E+01	9.98E-01	1.15E+01 1.15E+01	9.99E-01	1.15E+01 1.15E+01	4.98E-01	1.15E+01 1.15E+01	9.99E-01
ICP.f.Sr	1.64E+02 1.77E+02	8.54E+01	1.65E+02 1.55E+02	8.00E+01	1.88E+02 1.88E+02	4.69E+01	1.44E+02 1.66E+02	7.77E+01
ICP.f.Tl	2.00E+00 4.51E+00	1.63E+00	6.35E+00 5.26E+00	2.90E+00	8.14E+00 6.40E+00	1.82E+00	6.89E+00 1.12E+01	4.52E+00
ICP.f.V	2.36E+01 1.30E+01	4.57E+00	7.33E+00 8.30E+00	1.95E+00	1.05E+01 1.04E+01	1.31E+00	NA NA	NA
ICP.f.Zn	7.81E+01 7.25E+01	6.02E+00	5.13E+01 4.49E+01	3.85E+00	9.28E+01 8.35E+01	3.53E+00	1.11E+02 1.33E+02	9.74E+00
ICP.f.Zr	1.81E+02 1.97E+02	NA	7.90E+01 5.34E+01	NA	1.73E+02 6.92E+01	NA	1.35E+02 1.97E+02	NA
Dir.DSC	NO EXO NO EXO	NA	NO EXO NO EXO	NA	NO EXO NA	NA	NO EXO NO EXO	NA
Dir.TGA(%)	56.40 56.50	NA	63.30 63.70	NA	66.30 61.80	NA	50.70 54.30	NA
Dir.X Water	57.50 58.00	NA	57.30 56.90	NA	56.00 56.20	NA	55.90 56.00	NA
NYAA.As	4.62E-01 4.02E-01	1.72E+00	4.33E-01 3.53E-01	1.57E+00	3.19E-01 4.03E-01	1.44E+00	5.17E-01 5.54E-01	2.14E+00
NYAA.Se	1.52E+00 1.48E+00	1.34E-02	1.76E+00 2.07E+00	1.19E-01	2.01E+00 2.03E+00	8.08E+00	5.96E-01 7.04E-01	2.60E+00
CVA.A.Hg	5.60E-01 6.61E-01	4.88E+00	3.66E-01 3.83E-01	3.00E+00	5.39E-01 7.23E-01	5.05E+00	6.27E-01 6.57E-01	5.14E+00
Dir.CN	3.14E+00 3.08E+00	1.56E+00	1.79E+00 1.06E+00	NA	2.88E+00 3.10E+00	2.99E+01	2.07E+00 2.78E+00	2.43E+01
IC.W.F	8.91E+03 8.11E+03	8.51E+02	8.50E+03 8.33E+03	8.42E+04	9.04E+03 8.72E+03	8.88E+02	1.09E+04 1.10E+04	1.10E+04
IC.W.Cl	1.17E+03 1.14E+03	4.89E+01	1.14E+03 1.21E+03	4.98E+03	1.02E+03 1.06E+03	4.41E+01	1.20E+03 1.15E+03	5.02E+01

NA: Not available.

B-33

A-9

WHC-SD-WM-TI-603, Rev. 0

WHC-EP-0739

9513322.1428

Table A-1. Core Composite Data. (sheet 8 of 9)
 (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$)

Analyte	Core 40 Comp. 1	Mean/DL	Core 40 Comp. 2	Mean/DL	Core 41 Comp. 1	Mean/DL	Core 41 Comp. 2	Mean/DL
IC.w.NO3	1.45E+05 1.39E+05	7.63E+01	1.46E+05 1.53E+05	8.04E+03	1.23E+05 1.29E+05	6.77E+01	1.28E+05 1.31E+05	1.30E+03
IC.w.NO2	1.02E+04 9.17E+03	9.69E+01	9.12E+03 9.19E+03	9.16E+03	1.25E+04 1.24E+04	1.25E+02	1.21E+03 1.18E+03	1.20E+01
IC.w.PO4	1.45E+04 1.43E+04	1.44E+02	1.48E+04 1.51E+04	1.50E+04	1.34E+04 1.34E+04	1.34E+02	1.43E+04 1.41E+04	1.42E+02
IC.w.SO4	1.41E+04 1.40E+04	1.41E+02	1.41E+04 1.42E+04	1.42E+04	1.28E+04 1.32E+04	1.30E+02	1.22E+04 1.23E+04	1.23E+02
Spec.w.NO2	1.31E+04 1.32E+04	2.57E+00	1.00E+04 1.06E+04	2.02E+00	1.39E+04 1.26E+04	2.59E+02	1.24E+04 1.25E+04	2.49E+04
pH	9.59 9.66	NA	9.77 9.58	NA	9.95 9.69	NA	9.71 9.72	NA
NH3.w	<4.50E+3 <4.50E+3	0.00	<4.50E+3 <4.50E+3	0.00	<4.5E+3 <4.5E+3	0.00	<4.50E+3 <4.50E+3	NA
TOC.w.Acetate Equivalent	7.00E+02 7.00E+02	1.27E+00	5.80E-01 <5.50E2	0.00	<5.00E2 <5.50E2	0.00	7.96E+02 9.97E+02	NA
TIC.w.CO3	1.10E+03 1.20E+03	2.30E+00	1.60E+03 6.99E+02	2.30E+00	1.39E+03 1.10E+03	1.22E+01	1.10E+03 1.10E+03	4.07E+03
Fluor.U($\mu\text{Ci/g}$)	1.56E+03 2.07E+03	7.89E+00	1.86E+03 2.18E+03	8.78E+00	2.82E+03 2.82E+03	1.23E+01	2.29E+04 2.52E+04	NA
RAD.f.TA	7.89E-02 9.47E-02	2.41E+01	7.17E-02 7.28E-02	2.01E+01	1.23E-01 1.13E-01	3.28E+01	1.15E-01 1.01E-01	6.00E+01
RAD.f.TB	5.03E+01 5.02E+01	3.87E+01	3.88E+01 3.28E+01	2.75E+01	5.24E+01 5.23E+01	4.03E+01	4.58E+01 5.12E+01	2.31E+03
GEA.Cs-137	1.35E+01 1.34E+01	1.86E+03	1.09E+01 1.16E+01	1.56E+03	2.00E+01 2.04E+01	2.80E+03	2.43E+01 2.51E+01	3.43E+03

NA: Not available.

B-34

A-10

WHC-SD-WM-TI-603, Rev. 0

WHC-EP-0739

Table A-1. Core Composite Data. (sheet 9 of 9)
 (Units $\mu\text{g/g}$ except Radionuclides $\mu\text{Ci/g}$)

Analyte	Core 40 Comp. 1	Mean/DL	Core 40 Comp. 2	Mean/DL	Core 41 Comp. 1	Mean/DL	Core 41 Comp. 2	Mean/DL
GEA.Eu-154	<1.85E-2 <2.22E-2	NA	<1.81E-3 <1.94E-2	NA	<1.59E-2 <1.98E-2	NA	<2.10E-2 <2.10E-2	NA
GEA.Eu-155	<3.11E-2 <3.19E-2	NA	<2.90E-2 <3.02E-2	NA	<3.76E-2 <3.77E-2	NA	<3.90E-2 <4.10E-2	NA
GEA.Am-241	<7.48E-2 <7.47E-2	NA	<6.89E-2 <7.19E-2	NA	<8.96E-2 <9.12E-2	NA	<9.70E-2 <9.80E-2	NA
GEA.Co-60	<6.83E-3 <5.73E-3	NA	<6.54E-3 <6.64E-3	NA	<6.82E-3 <5.98E-3	NA	<6.60E-3 <7.50E-3	NA
Pu-239/240	3.49E-02 5.33E-02	1.34E+01	8.34E-02 7.57E-02	2.41E+01	5.49E-02 5.64E-02	NA	4.49E-02 5.41E-02	1.46E+01
Pu-238	<4.49E-3 <4.49E-3	NA	<4.50E-3 <4.50E-3	NA	NA NA	NA	<4.50E-3 <4.50E-3	NA
Am-241	8.05E-03 1.23E-02	2.16E+00	1.41E-02 1.38E-02	1.72E+00	1.28E-02 1.62E-02	8.06E+00	1.31E-02 1.34E-02	2.37E+00
Cm-244	NA NA	NA	NA NA	NA	NA NA	NA	NA NA	NA
Np-237	<2.16E-2 <1.08E-02	NA	<2.16E-2 <1.08E-2	NA	<1.89E-02 <1.89E-02	NA	<3.24E-3 <3.24E-2	NA
Tc-99	3.56E-02 4.06E-02	4.05E+01	2.83E-02 2.96E-02	3.08E+01	4.92E-02 4.23E-02	4.87E+01	3.74E-02 3.24E-02	4.53E+01
I-129	<1.39E-2 <1.45E-2	NA	<9.54E-3 <1.23E-2	NA	<1.83E-2 <1.23E-2	NA	<5.04E-2 <6.74E-2	NA
Sr-90	9.76E+00 1.02E+01	4.99E+03	7.71E+00 8.79E+00	3.59E+03	1.18E+01 1.20E+01	5.17E+02	8.74E+00 9.96E+00	2.28E+03
H-3	3.89E-04 4.02E-04	1.80E+00	7.79E-04 4.83E-04	1.75E+00	4.20E-04 3.66E-04	1.75E+00	6.97E-04 1.08E-03	2.47E+00
C-14	3.06E-04 1.33E-04	2.00E+00	1.82E-04 4.21E-04	2.74E+00	3.49E-04 2.89E-04	2.82E-08	<2.24E-4 <2.99E-4	NA
Se-79	<1.25E-4 <1.22E-4	NA	<1.23E-4 <1.22E-4	NA	<1.26E-4 <1.25E-4	NA	<1.47E-4 <1.44E-4	NA

NA: Not available.

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Table A-2. Homogenization Test Data. (sheet 1 of 2)
(Units $\mu\text{g/g}$ except radionuclides $\mu\text{Ci/g}$)

Analyte	Core 41 Seg. 2 Loc. 1	Mean/DL	Core 41 Seg. 2 Loc. 2	Mean/DL	Core 41 Seg. 6 Loc. 1	Mean/DL	Core 41 Seg. 6 Loc. 2	Mean/DL
ICP.a.Al	1.62E+04 1.60E+04	4.60E+03	1.66E+04 1.62E+04	4.68E+03	1.71E+04 1.72E+04	5.03E+03	1.70E+04 1.67E+04	4.97E+03
ICP.a.B	3.19E+01 3.91E+01	3.55E+01	2.57E+01 1.81E+01	2.19E+01	2.56E+01 3.17E+01	1.51E+01	2.42E+01 2.23E+01	1.22E+01
ICP.a.Ba	1.62E+01 1.64E+01	5.44E+01	1.66E+01 1.60E+01	5.43E+01	8.38E+00 8.77E+00	2.86E+01	8.48E+00 8.42E+00	2.82E+01
ICP.a.Bi	3.10E+04 3.05E+04	5.30E+03	3.20E+04 3.09E+04	5.42E+03	2.82E+04 2.82E+04	3.32E+03	2.79E+04 2.75E+04	3.26E+03
ICP.a.Ca	3.71E+02 3.88E+02	1.40E+01	3.66E+02 3.48E+02	1.31E+01	2.77E+02 3.03E+02	1.21E+02	2.76E+02 2.69E+02	1.14E+02
ICP.a.Ce	2.53E+02 2.67E+02	2.65E+01	2.61E+02 2.58E+02	2.65E+01	1.94E+02 1.90E+02	2.23E+01	1.93E+02 1.86E+02	2.21E+01
ICP.a.Cr	1.22E+03 1.17E+03	9.95E+02	1.25E+03 1.19E+03	1.02E+03	1.04E+03 1.02E+03	7.36E+02	1.03E+03 1.01E+03	7.29E+02
ICP.a.Cu	2.21E+01 2.25E+01	5.58E+01	2.24E+01 2.17E+01	5.51E+01	1.83E+01 1.83E+01	3.05E+01	1.86E+01 1.80E+01	3.05E+01
ICP.a.Fe	1.74E+04 1.67E+04	1.14E+04	1.78E+04 1.70E+04	1.16E+04	1.22E+04 1.21E+04	6.10E+03	1.22E+04 1.18E+04	6.00E+03
ICP.a.K	5.49E+02 5.68E+02	4.33E+01	5.73E+02 5.61E+02	4.40E+01	9.47E+01 9.86E+01	7.27E+00	1.13E+02 1.04E+02	8.20E+00
ICP.a.Mg	1.58E+02 1.78E+02	2.40E+02	1.63E+02 1.48E+02	2.22E+02	1.38E+02 1.45E+02	2.35E+02	1.36E+02 1.30E+02	2.22E+02
ICP.a.Mn	7.06E+01 6.79E+01	2.31E+02	7.15E+01 6.83E+01	2.33E+02	4.01E+01 4.02E+01	1.34E+02	4.04E+01 3.93E+01	1.33E+02
ICP.a.Na	9.59E+04 9.71E+04	3.45E+04	9.88E+04 9.67E+04	3.49E+04	1.01E+05 1.01E+05	1.16E+04	9.82E+04 9.59E+04	1.12E+04
ICP.a.Ni	1.66E+01 1.65E+01	1.18E+01	1.62E+01 1.61E+01	1.16E+01	1.24E+01 1.30E+01	7.06E+00	1.23E+01 1.21E+01	6.78E+00
ICP.a.P	2.63E+04 2.62E+04	2.65E+03	2.67E+04 2.73E+04	2.73E+03	2.59E+04 2.55E+04	3.21E+03	2.53E+04 2.50E+04	3.14E+03

NA: Not available.

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Table A-2. Homogenization Test Data. (sheet 2 of 2)
 (Units $\mu\text{g/g}$ except radionuclides $\mu\text{Ci/g}$)

Analyte	Core 41 Seg. 2 Loc. 1	Mean/DL	Core 41 Seg. 2 Loc. 2	Mean/DL	Core 41 Seg. 6 Loc. 1	Mean/DL	Core 41 Seg. 6 Loc. 2	Mean/DL
ICP.a.Pb	9.23E+01 9.45E+01	1.32E+01	1.02E+02 9.43E+01	1.38E+01	6.48E+01 6.77E+01	7.79E+00	6.06E+01 6.90E+01	7.62E+00
ICP.a.S	4.25E+03 4.26E+03	9.45E+02	4.43E+03 4.32E+03	9.72E+02	4.05E+03 3.92E+03	6.63E+02	4.13E+03 3.99E+03	6.77E+02
ICP.a.Se	3.78E+02 3.72E+02	4.52E+01	3.99E+02 3.91E+02	4.76E+01	1.43E+01 2.19E+01	1.38E+00	2.61E+01 3.26E+01	2.24E+00
ICP.a.Si	2.28E+03 2.17E+03	5.17E+02	2.19E+03 2.33E+03	5.26E+02	1.61E+03 1.67E+03	7.13E+02	1.15E+03 1.54E+03	5.83E+02
ICP.a.Sr	2.18E+02 2.16E+02	5.41E+02	2.25E+02 2.17E+02	5.52E+02	1.74E+02 1.73E+02	2.88E+02	1.74E+02 1.69E+02	2.87E+02
ICP.a.Ti	5.47E+00 6.00E+00	1.43E+01	5.84E+00 5.69E+00	1.44E+01	4.12E+00 4.18E+00	8.30E+00	4.30E+00 4.23E+00	8.52E+00
ICP.a.Zn	1.20E+02 1.18E+02	4.77E+01	1.26E+02 1.20E+02	4.90E+01	4.45E+01 4.39E+01	1.77E+01	4.39E+01 4.35E+01	1.75E+01
ICP.a.Zr.	1.46E+02 1.34E+02	NA	1.37E+02 1.65E+02	NA	1.08E+02 8.97E+01	8.99E+01	7.66E+01 1.01E+02	8.08E+01
RAD.a.TA	1.42E-01 1.55E-01	NA	1.71E-01 1.55E-01	NA	1.41E-01 1.40E-01	2.65E+02	1.45E-01 1.44E-01	NA
GEA.Cs-137	4.57E+01 4.59E+01	NA	4.71E+01 4.72E+01	NA	7.26E+00 7.31E+00	NA	7.20E+00 7.34E+00	NA
GEA.Eu-154	<1.70E-2 <1.90E-2	NA	<1.80E-2 <1.95E-2	NA	<3.70E-3 <4.00E-3	NA	<3.80E-3 <2.70E-3	NA
GEA.Eu-155	<5.20E-2 <5.40E-2	NA	<5.40E-2 <5.40E-2	NA	<1.00E-2 <9.90E-3	NA	<9.80E-3 <9.20E-3	NA
GEA.Am-241	<1.30E-1 <1.30E-1	NA	<1.30E-1 <1.30E-1	NA	<2.40E-2 <2.40E-2	NA	<2.35E-2 <2.20E-2	NA
GEA.Co-60	<6.50E-3 <6.10E-3	NA	<5.80E-3 <5.40E-3	NA	<1.26E-3 <9.40E-4	NA	<1.20E-3 <1.00E-3	NA

NA: Not available.

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APPENDIX B
CORE COMPOSITE DATA PLOTS

B-1

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WHC-SD-WM-TI-603, Rev. 0

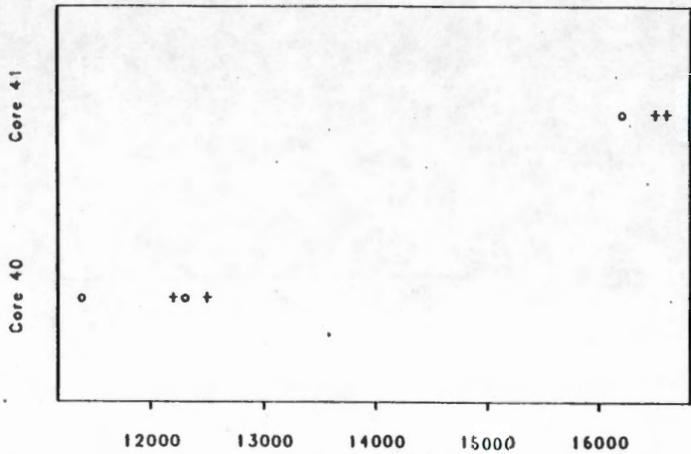
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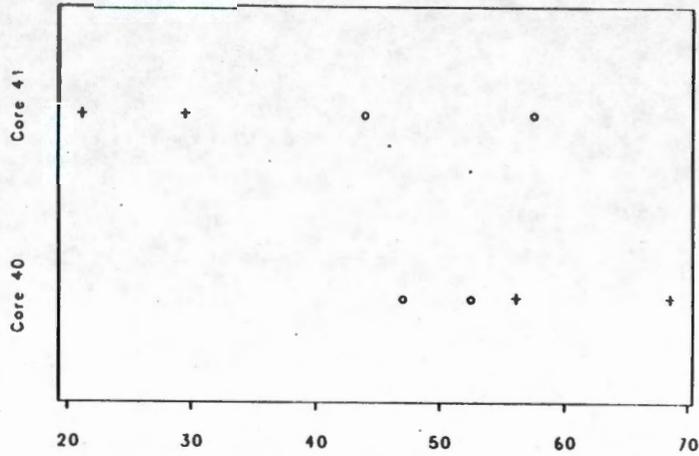
WHC-EP-0739

MHC-SD-WM-TI-603, Rev. 0

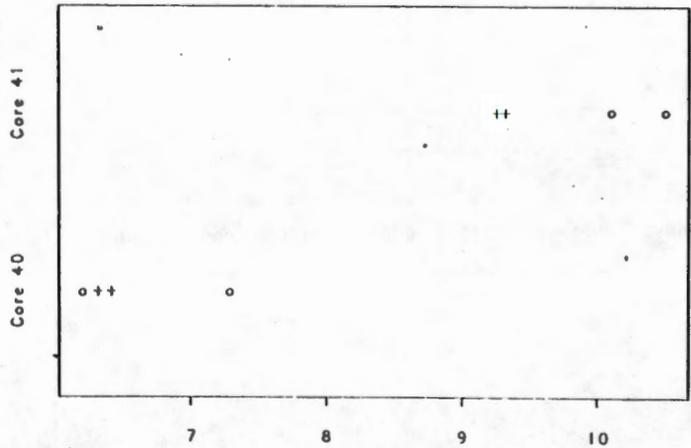
ICP.a.Al



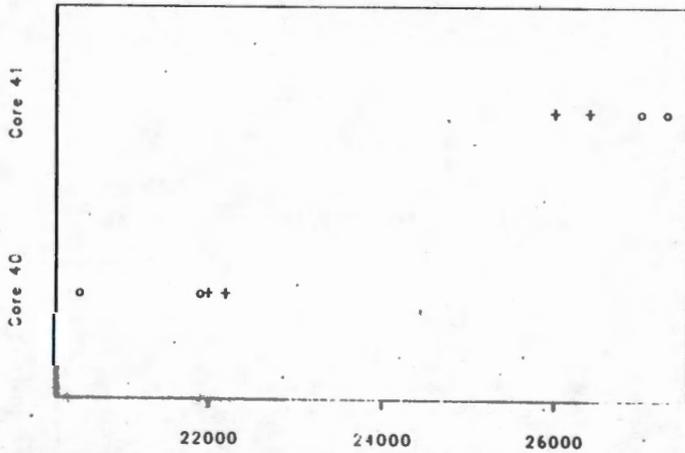
ICP.a.B



ICP.a.Ba



ICP.a.Bi



Composite 1 = o, Composite 2 = +
Units are ug/g except for radionuclides which are uCi/g

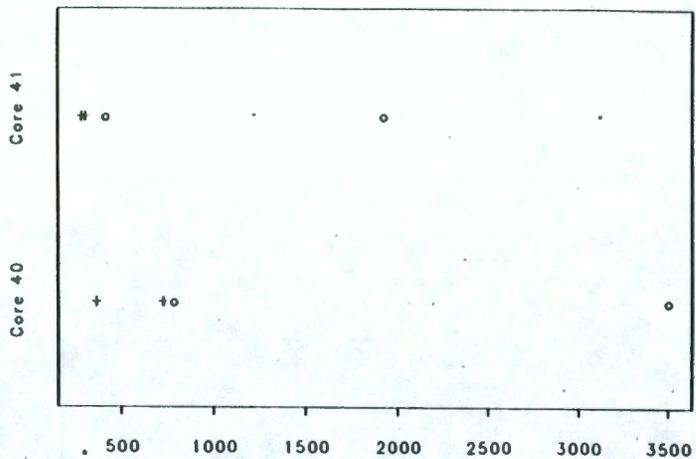
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B-3

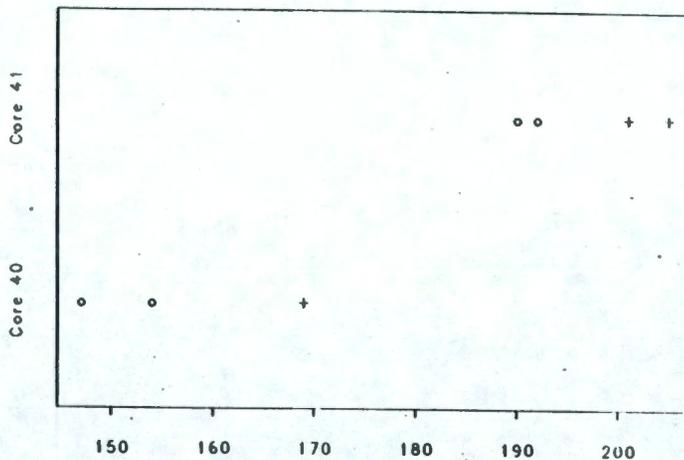
B-42

B-4

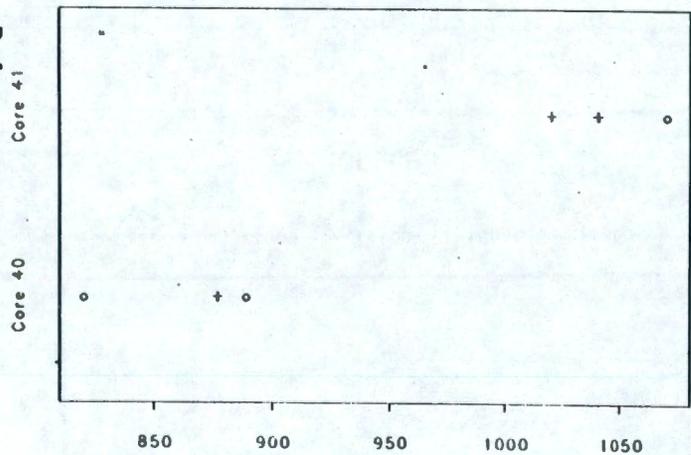
ICP.a.Ca



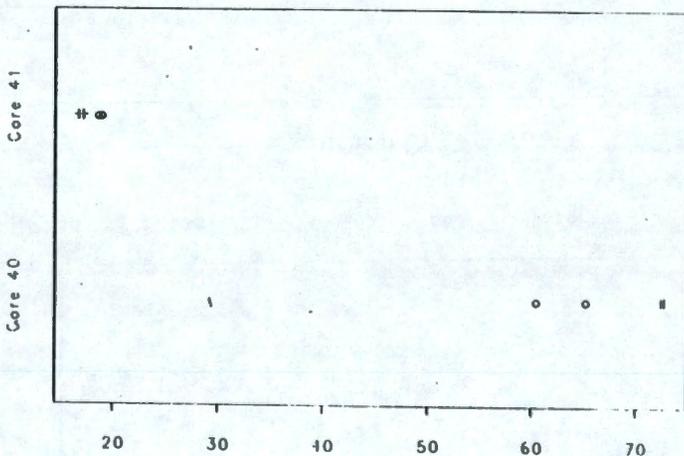
ICP.a.Ce



ICP.a.Cr



ICP.a.Cu



Composite 1 = o, Composite 2 = +
Units are ug/g except for radionuclides which are uCi/g

MHC-SD-WM-TI-603, Rev. 0

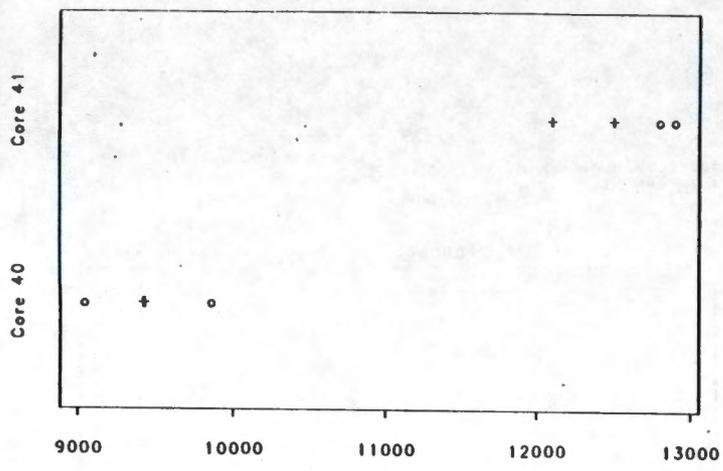
WHC-EP-0739

9513322.1433

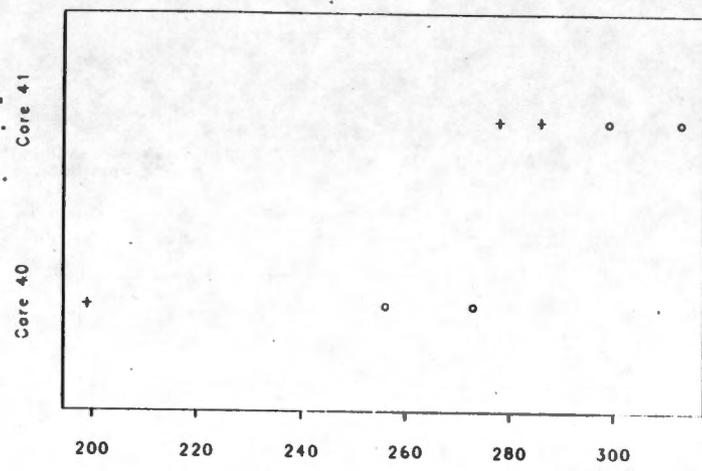
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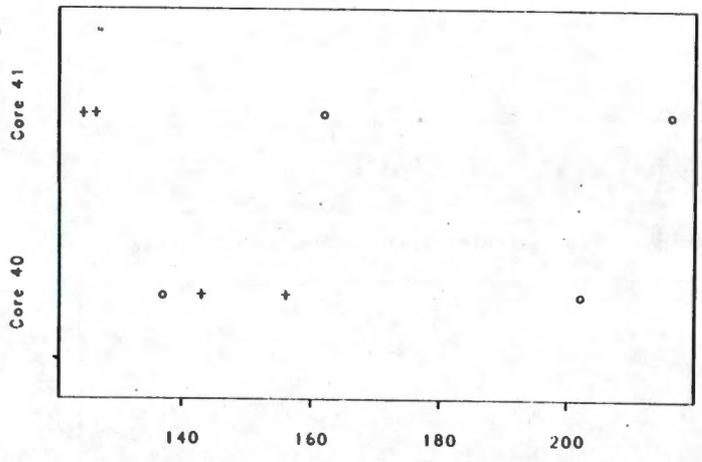
ICP.a.Fe



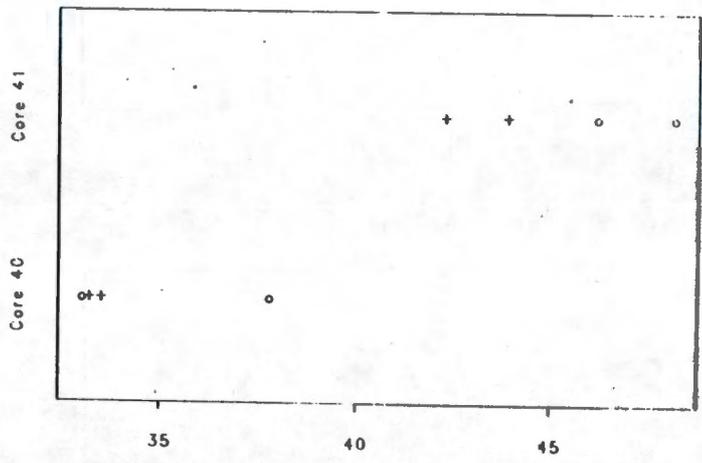
ICP.a.K



ICP.a.Mg



ICP.a.Mn



Composite 1 - o , Composite 2 - +
Units are ug/g except for radionuclides which are uCi/g

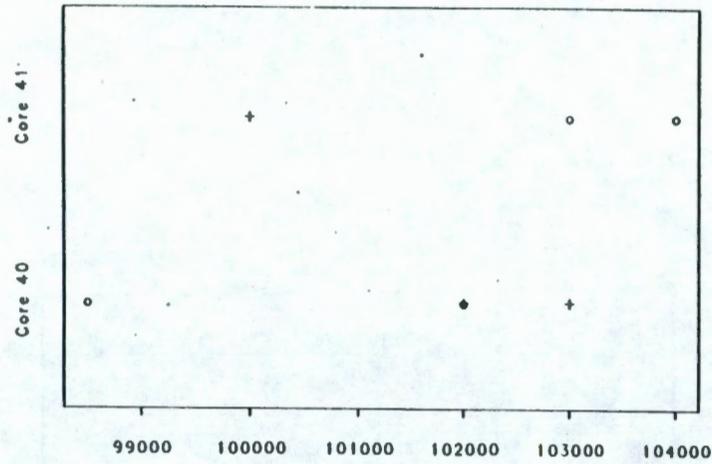
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B-43

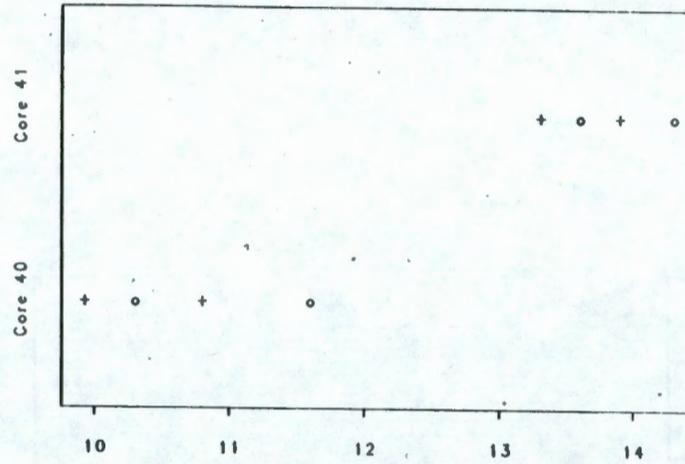
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B-6

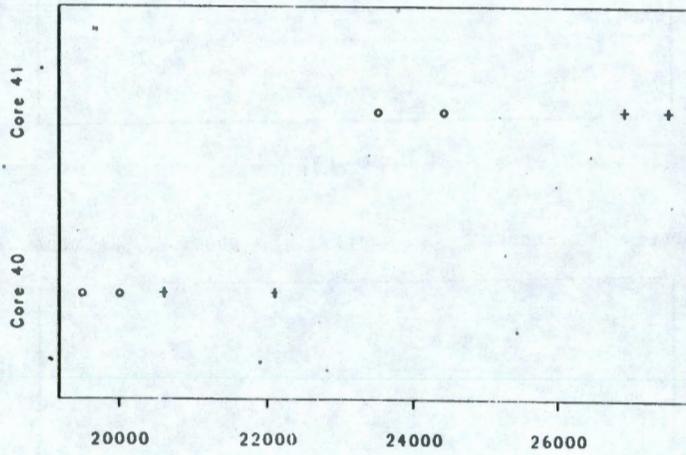
ICP.a.Na



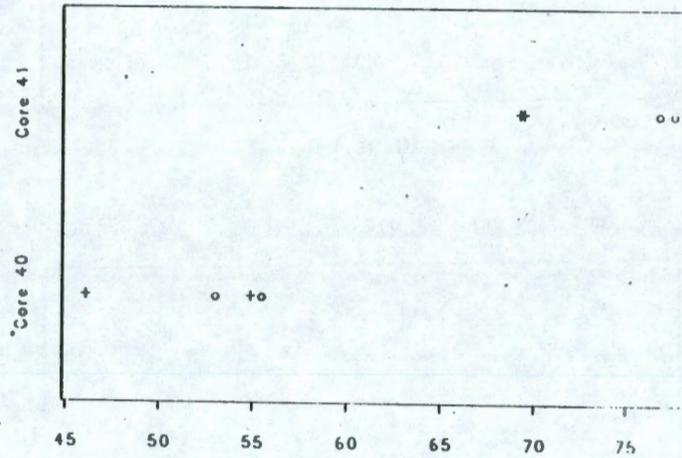
ICP.a.Ni



ICP.a.P



ICP.a.Pb



Composite 1 - o . Composite 2 - +
Units are ug/g except for radionuclides which are uCi/g

WHC-SD-WM-TI-603, Rev. 0

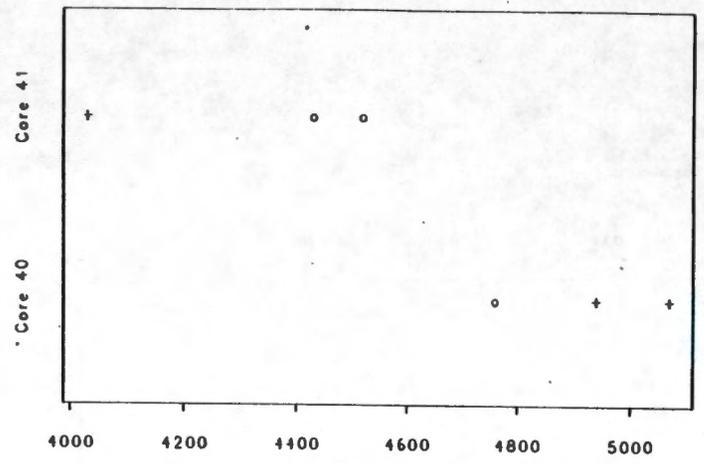
WHC-EP-0739

9513322.1434

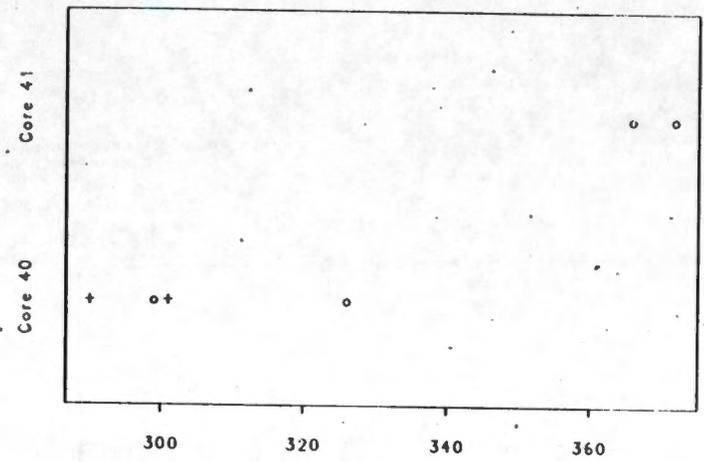
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WHC-SD-MM-TI-603, Rev. 0

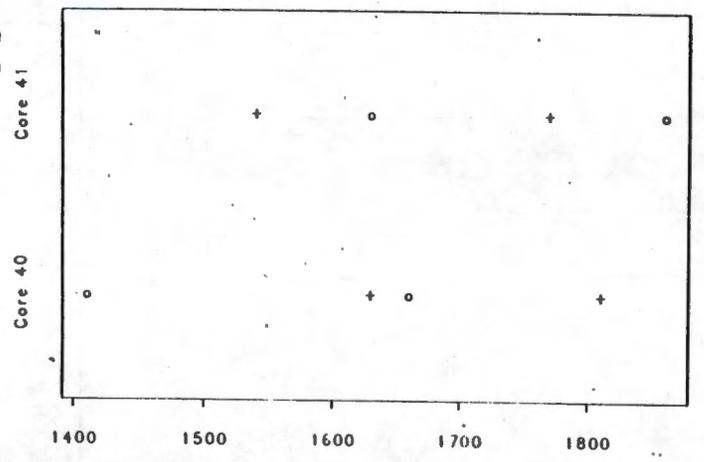
ICP.a.S



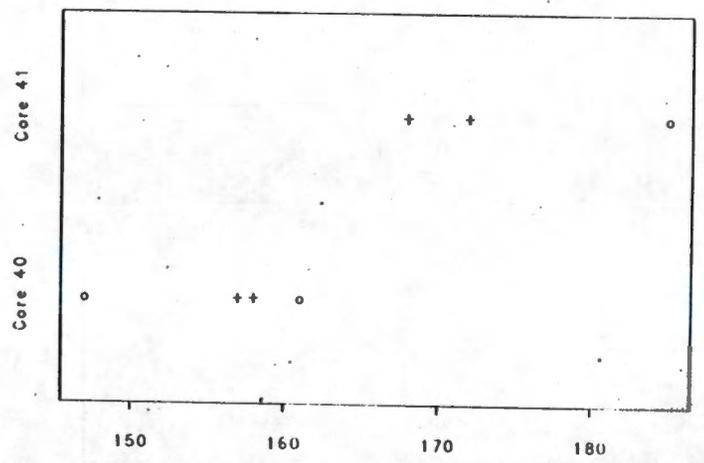
ICP.a.Se



ICP.a.Si



ICP.a.Sr



Composite 1 -- o, Composite 2 -- +
Units are ug/g except for radionuclides which are uCi/g

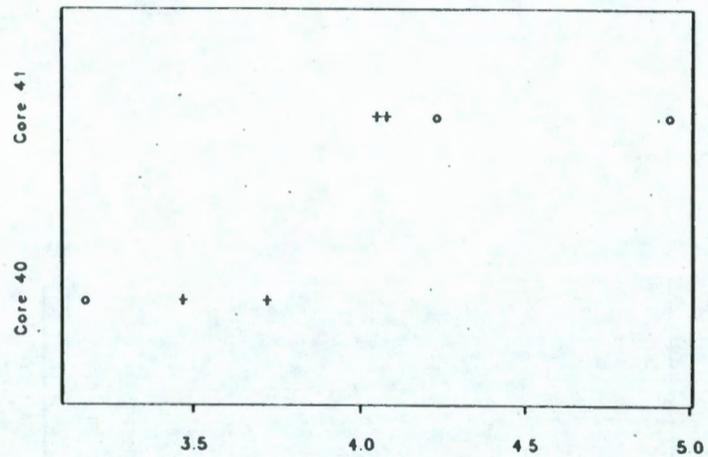
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B-7

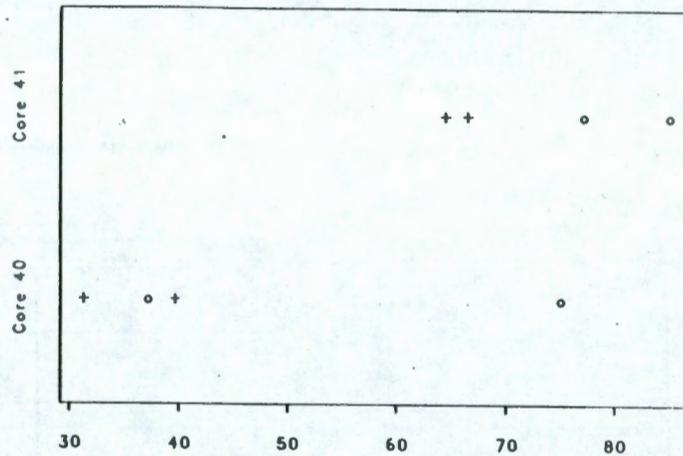
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B-8

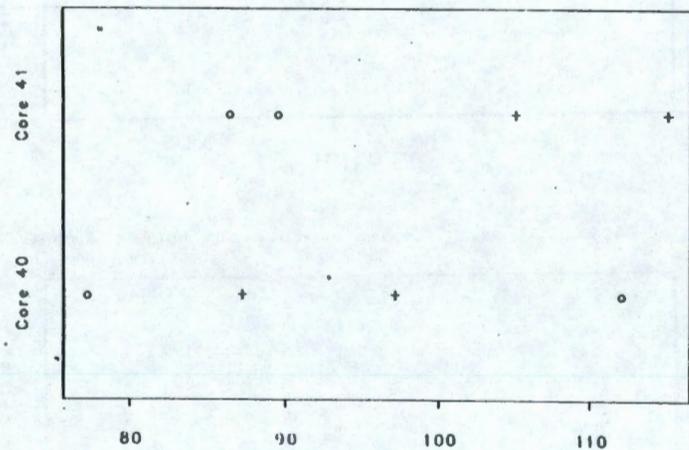
ICP.a.Ti



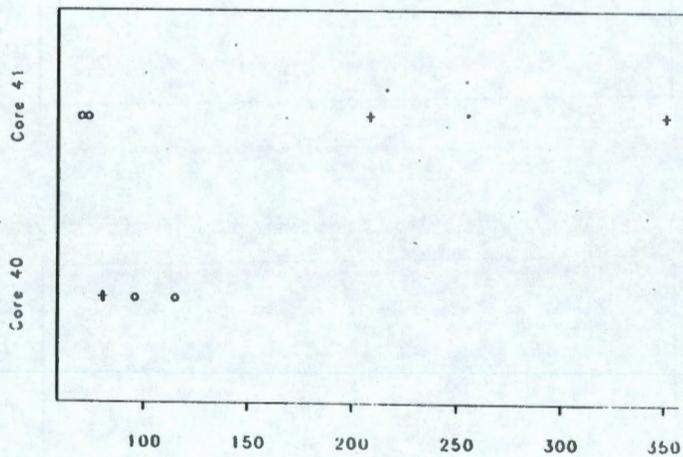
ICP.a.Zn



ICP.a.Zr



ICP.w.Al

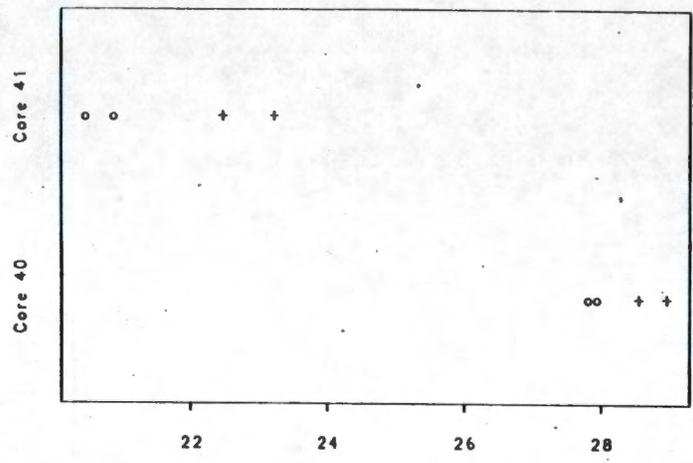


Composite 1 - o , Composite 2 - +
Units are ug/g except for radionuclides which are uCi/g

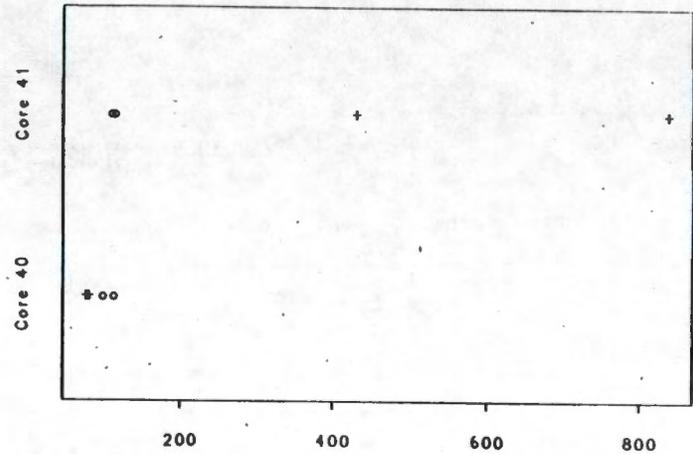
MHC-SD-WM-TI-603, Rev. 0

WHC-EP-0739

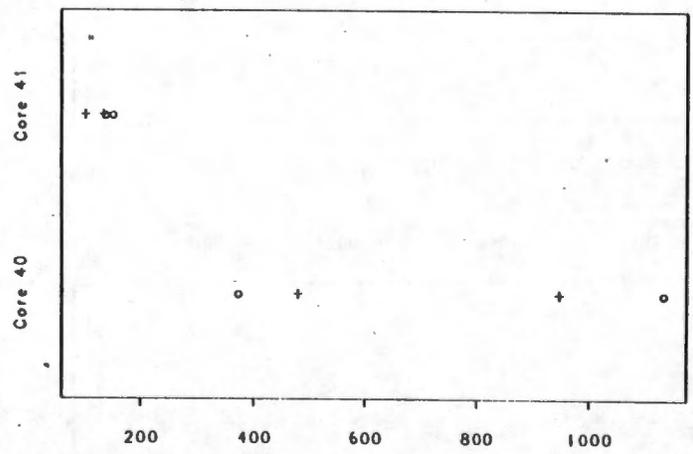
ICP.w.B



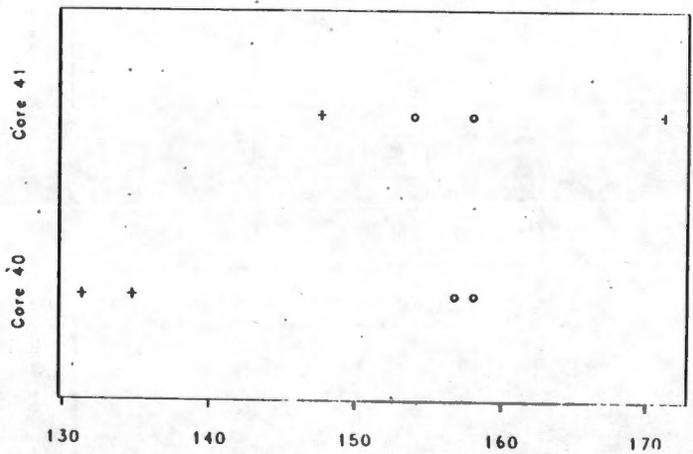
ICP.w.Bi



ICP.w.Ca



ICP.w.Cr



Composite 1 = o, Composite 2 = +
Units are ug/g except for radionuclides which are uCi/g

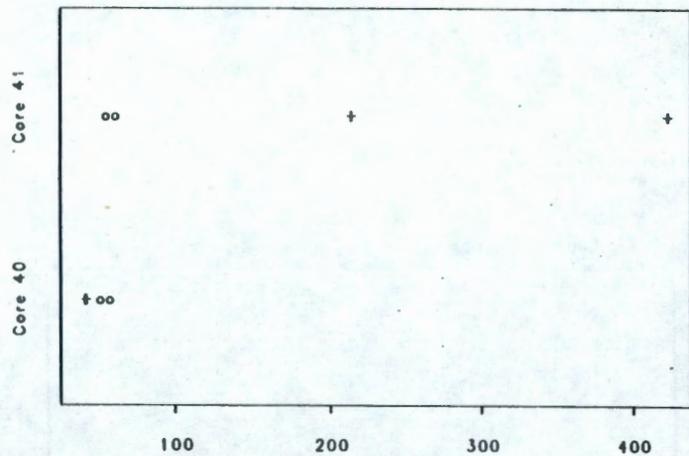
B-9

B-47

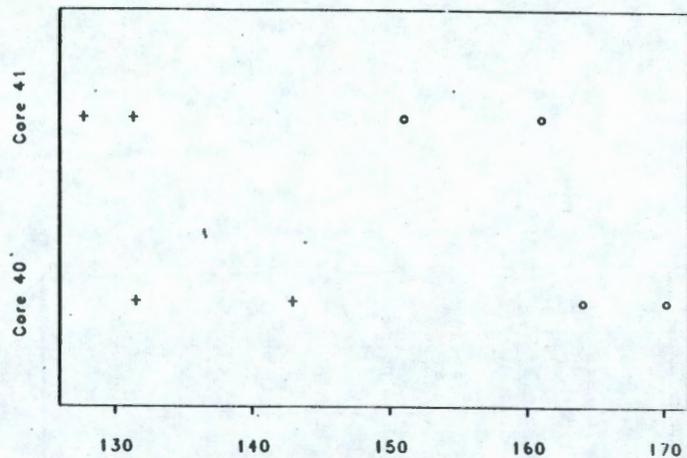
B-48

B-10

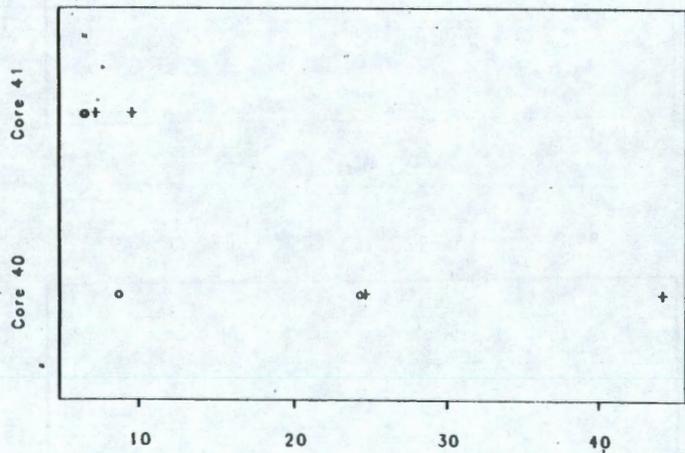
ICP.w.Fe



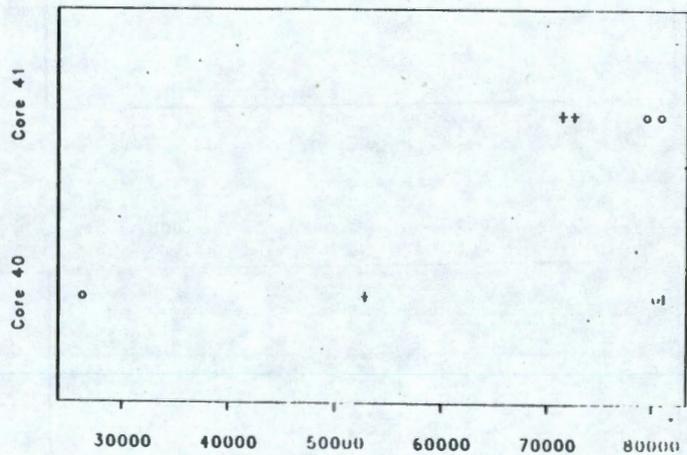
ICP.w.K



ICP.w.Mg



ICP.w.Na



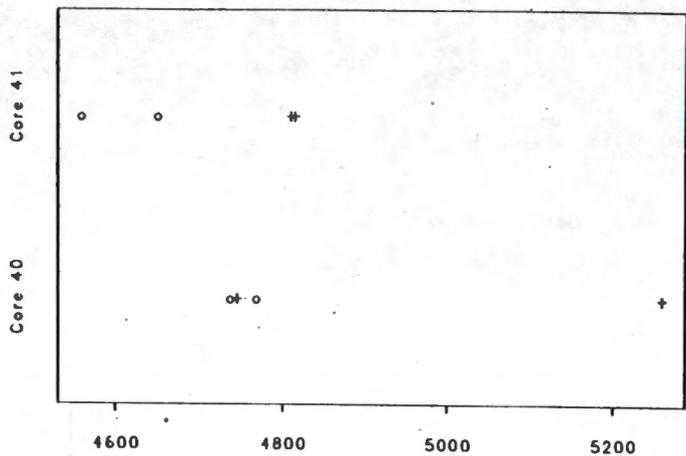
Composite 1 - o ; Composite 2 - +
Units are ug/g except for radionuclides which are uCi/g

WMC-SD-WM-TI-603, Rev. 0

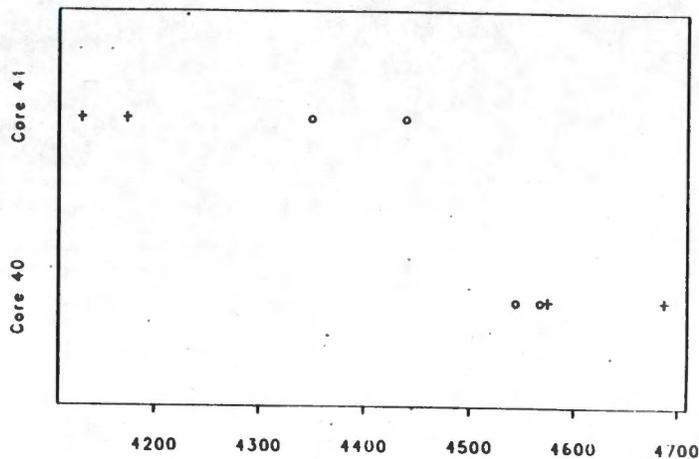
WMC-EP-0739

MHC-SD-WM-TI-603, Rev. 0

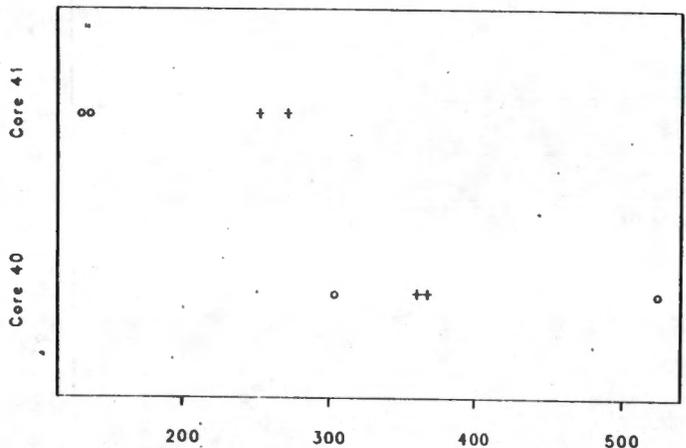
ICP.w.P



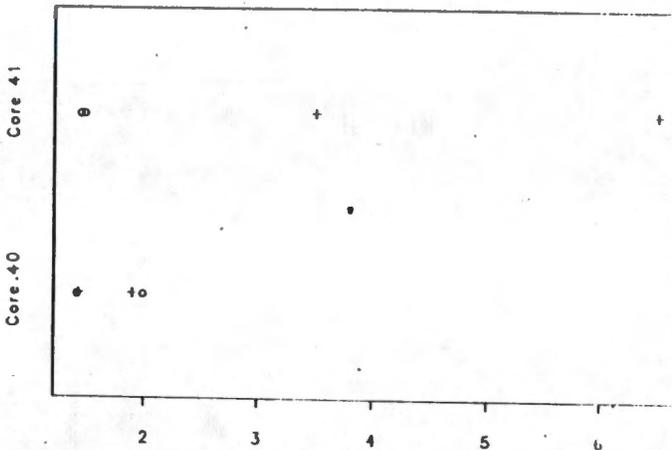
ICP.w.S



ICP.w.Si



ICP.w.Sr



Composite 1 = o, Composite 2 = +
Units are ug/g except for radionuclides which are uCi/g

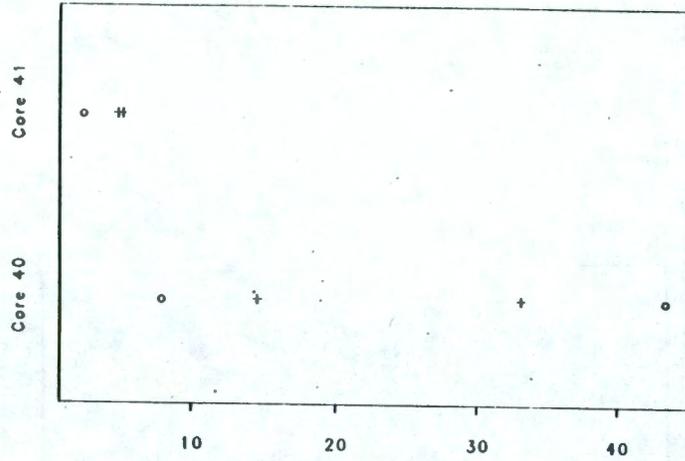
B-11

B-49

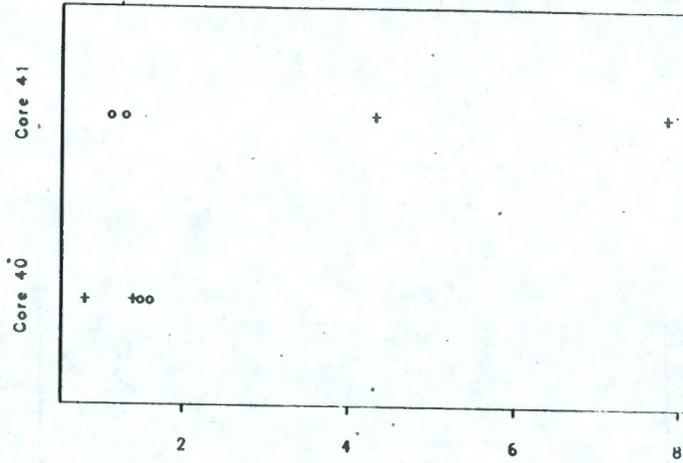
B-50

B-12

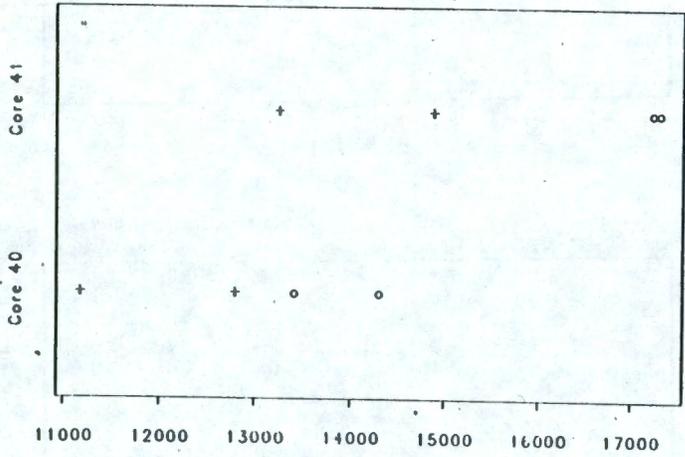
ICP.w.Zn



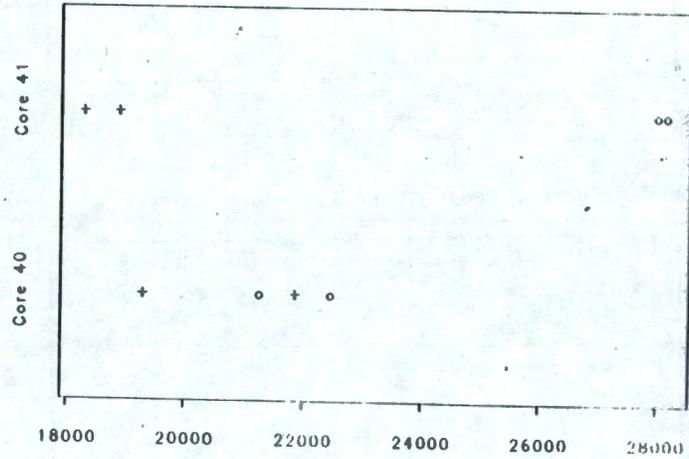
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ICP.f.Al



ICP.f.Bi



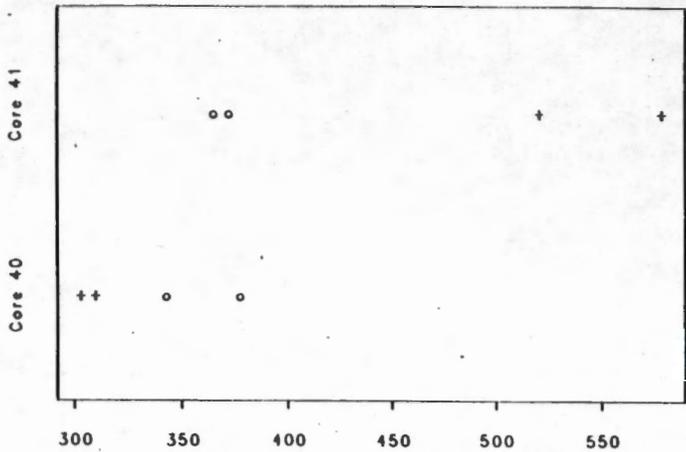
Composite 1 - o . Composite 2 - +
Units are ug/g except for radionuclides which are uCi/g

WHC-SD-WM-TI-603, Rev. 0

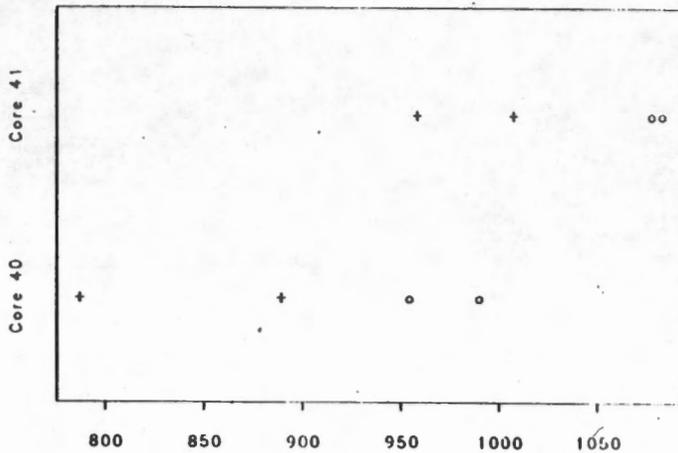
WHC-EP-0739

MHC-SD-WM-TI-603, Rev. 0

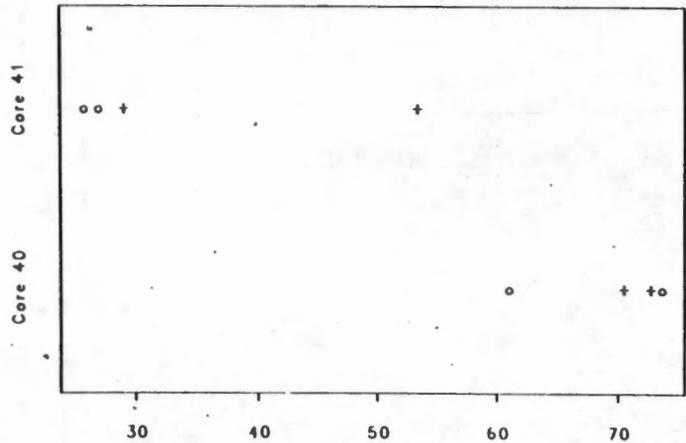
ICP.f.Co



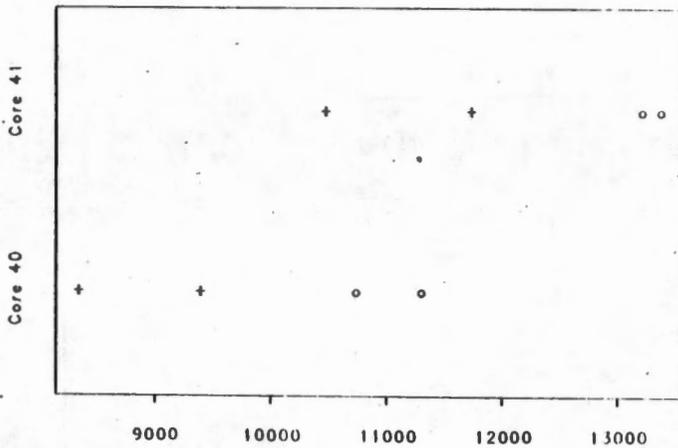
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ICP.f.Cu

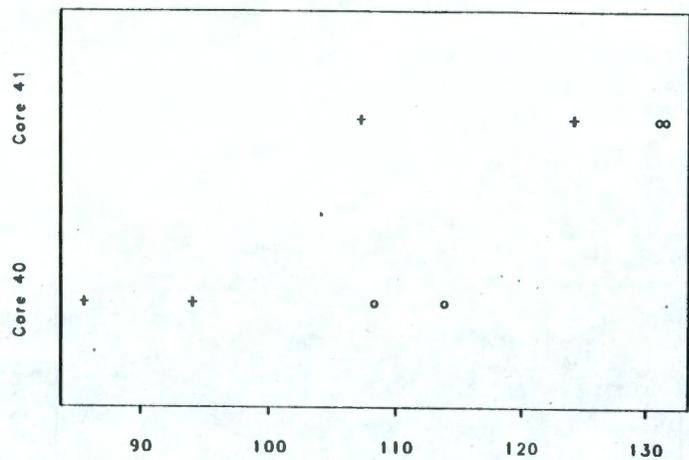


ICP.f.Fe

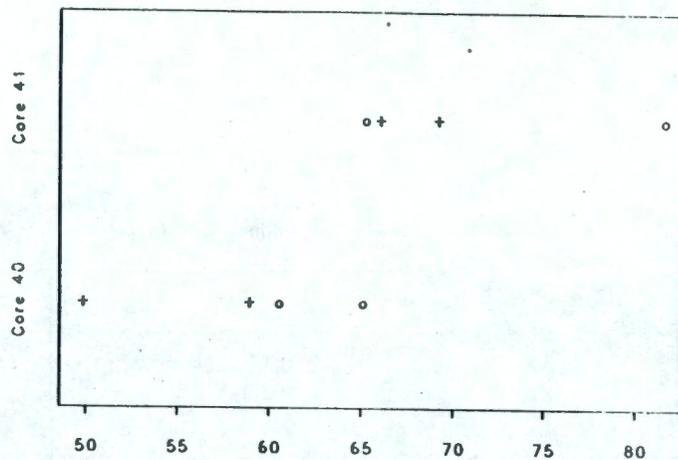


Composite 1 - o, Composite 2 - +
Units are ug/g except for radionuclides which are uCi/g

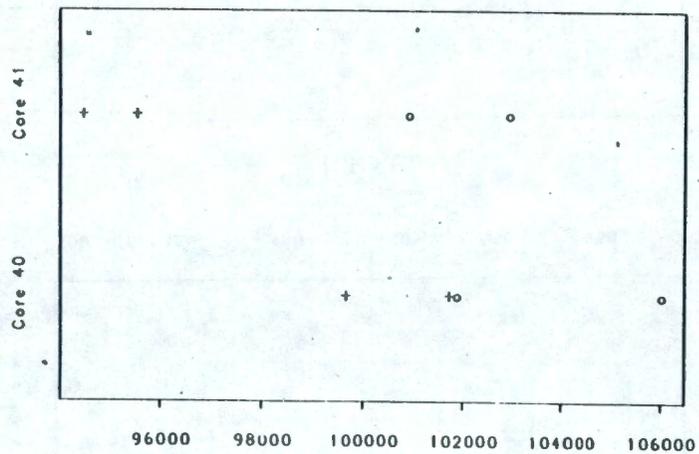
ICP.f.Mg



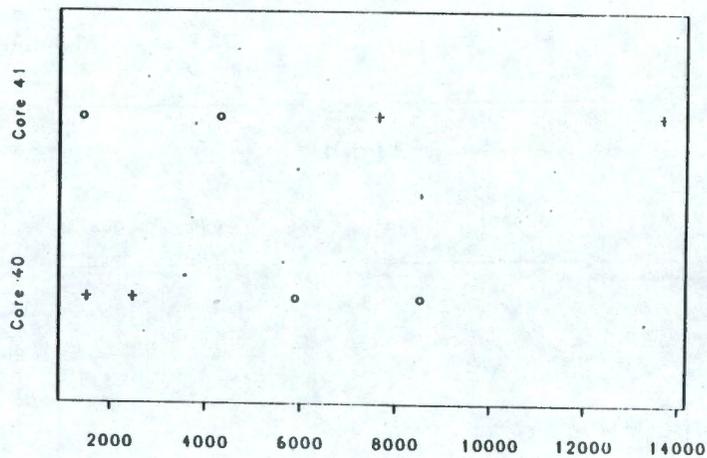
ICP.f.Mn



ICP.f.Na



ICP.f.Ni



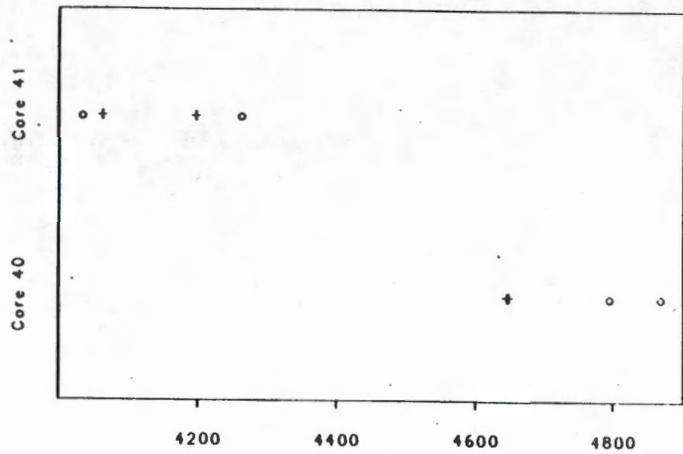
Composite 1 = o , Composite 2 = +
Units are ug/g except for radionuclides which are uCi/g .

9513322.1438

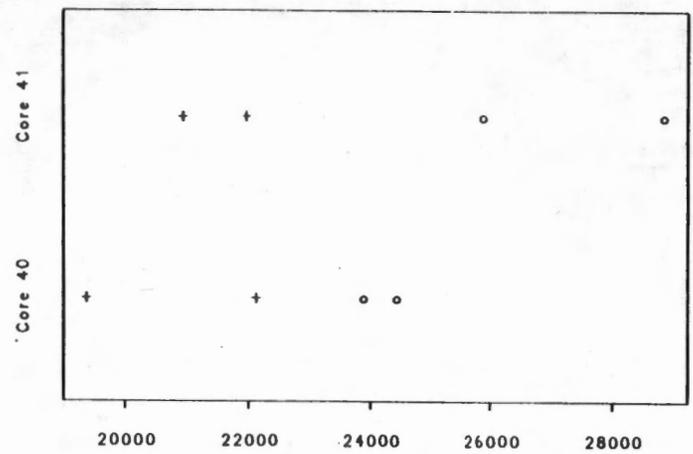
WHC-EP-0739

MHC-SD-WM-TI-603, Rev. 0

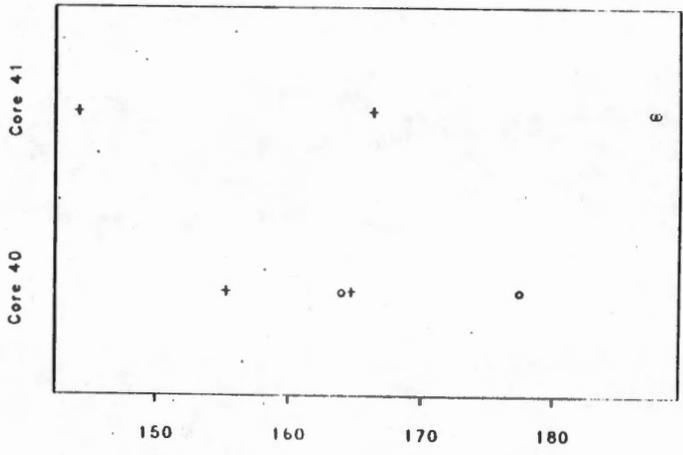
ICP.f.S



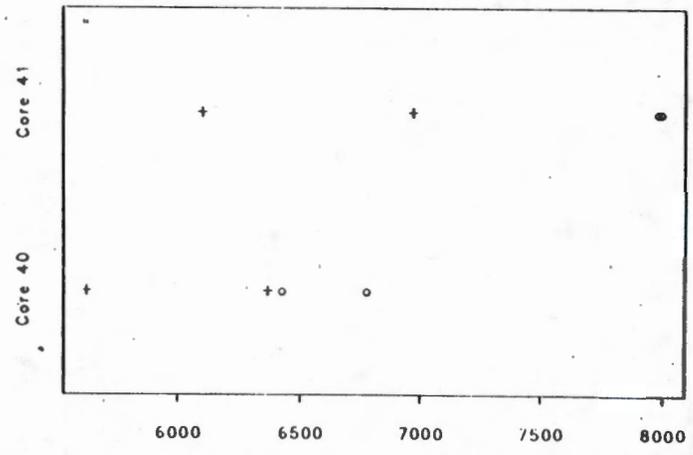
ICP.f.P



ICP.f.Sr



ICP.f.Si



Composite 1 - o , Composite 2 - +
Units are ug/g except for radionuclides which are uCi/g

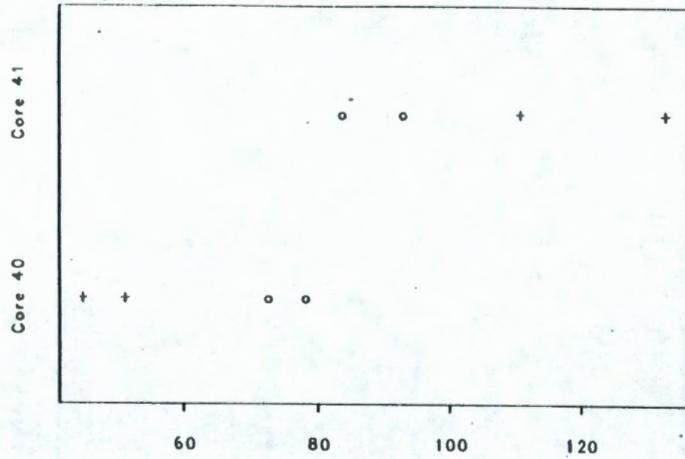
B-53

B-15

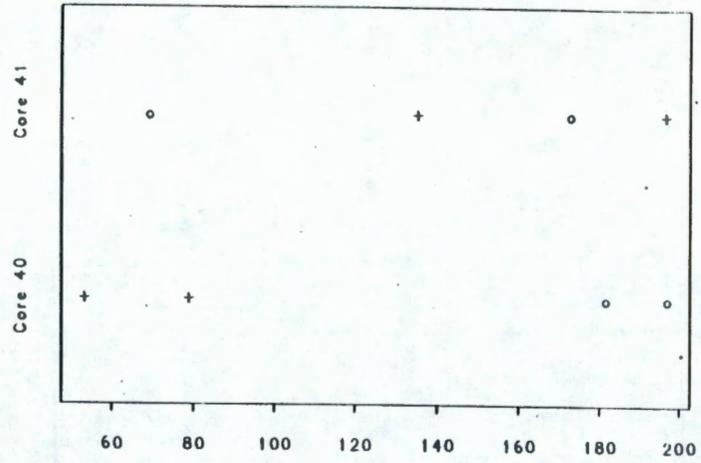
B-54

B-16

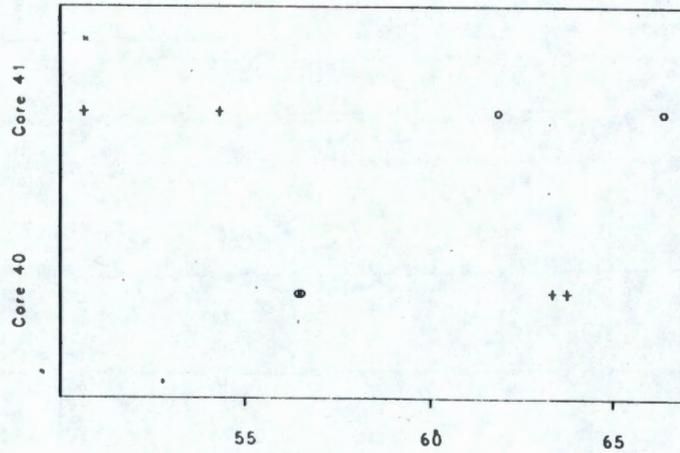
ICP.f.Zn



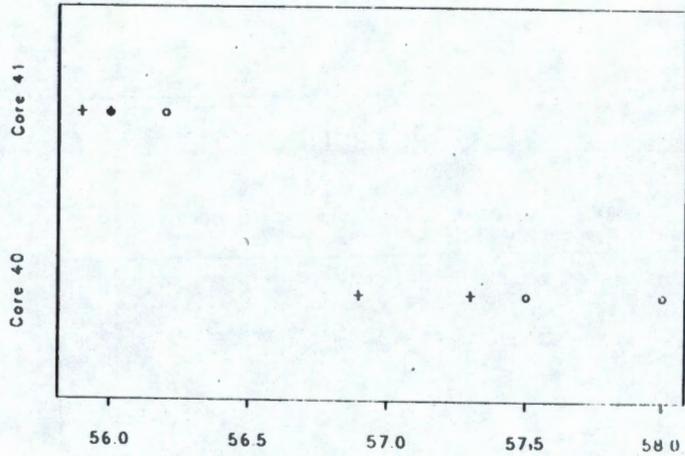
ICP.f.Zr



Dir.%TGA



Dir.%Water



Composite 1 - o , Composite 2 - +
Units are ug/g except for radionuclides which are uCi/g

WHC-SD-WM-TI-603, Rev. 0

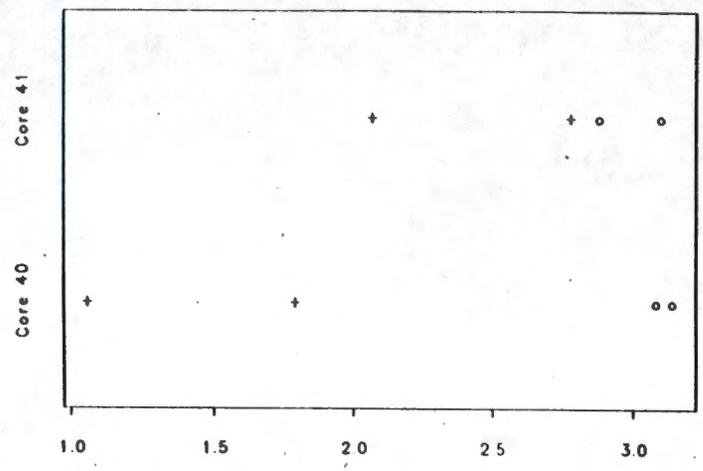
WHC-EP-0739

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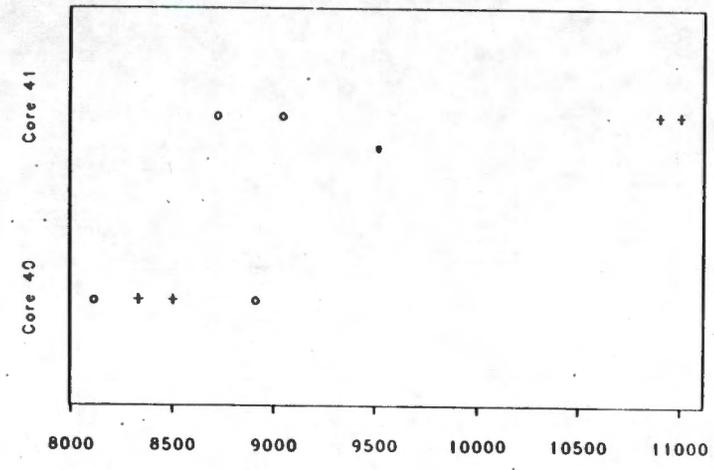
WHC-EP-0739

MHC-SD-WM-TI-603, Rev. 0

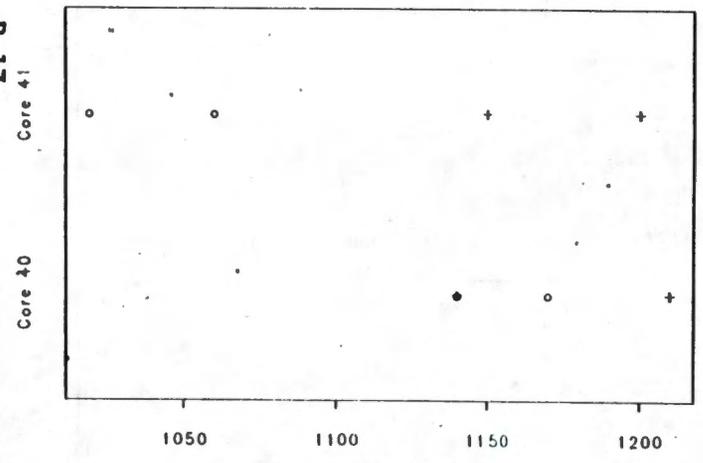
Dir.CN



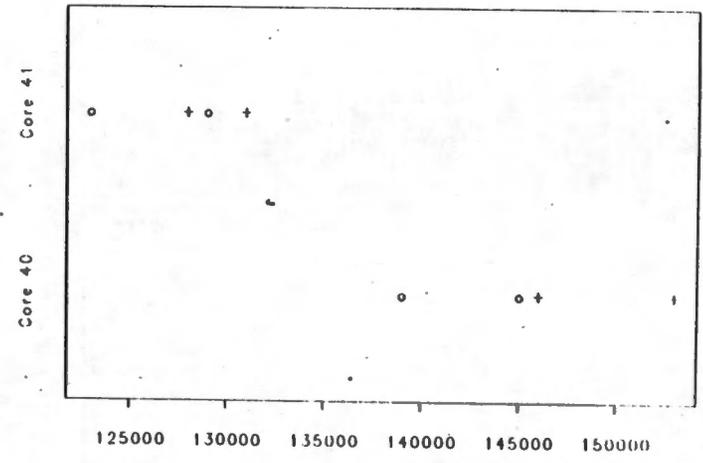
IC.w.F



IC.w.Cl



IC.w.NO3



Composite 1 = o, Composite 2 = +
Units are ug/g except for radionuclides which are uCi/g

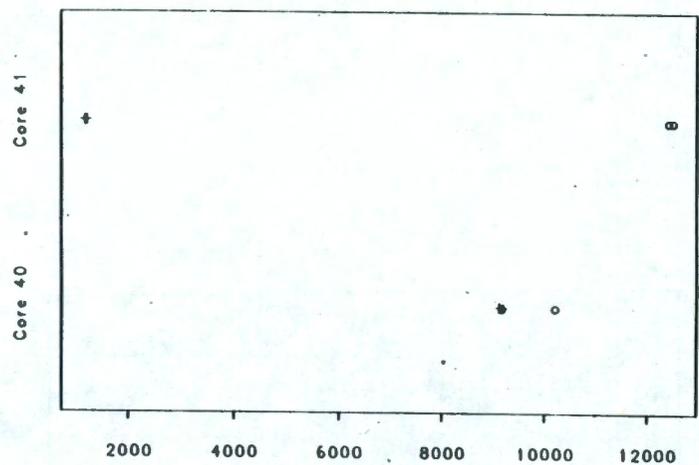
B-55

B-17

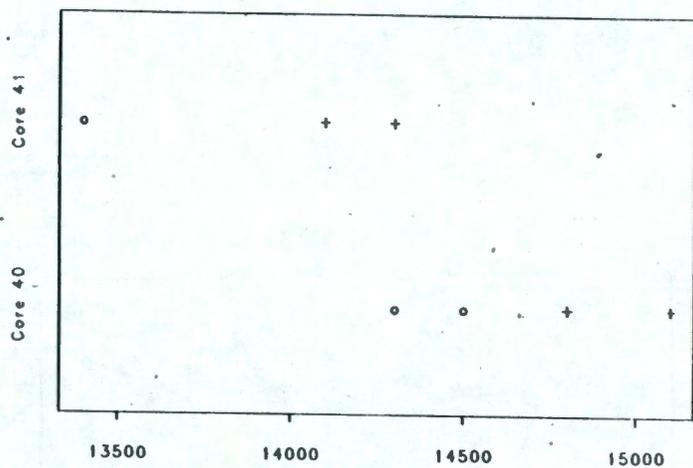
B-56

B-18

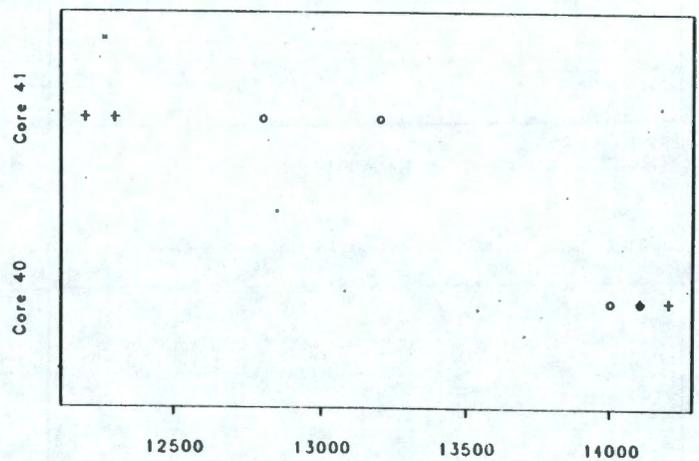
IC.w.NO2



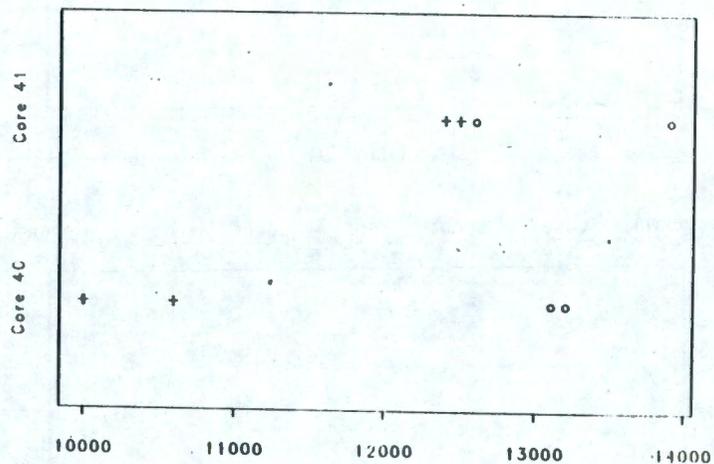
IC.w.PO4



IC.w.SO4



Spec.w.NO2



Composite 1 = o , Composite 2 = +
Units are ug/g except for radionuclides which are uCi/g

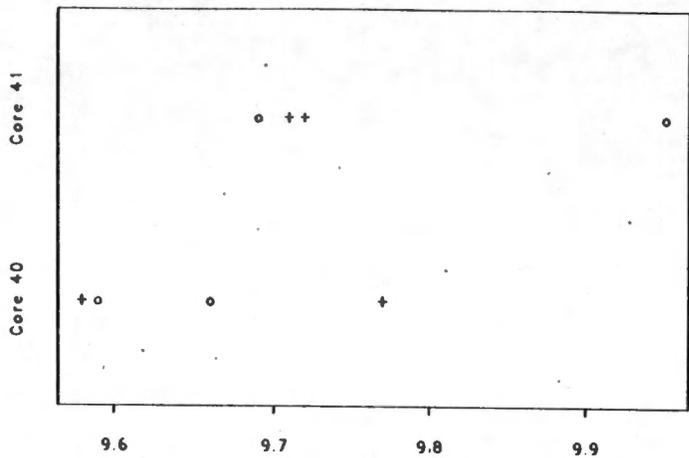
MHC-SD-WM-TI-603, Rev. 0

WHC-EP-0739

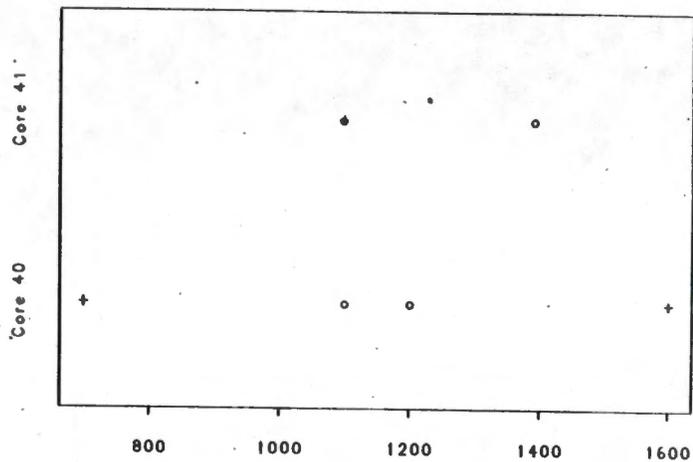
B-57

B-19

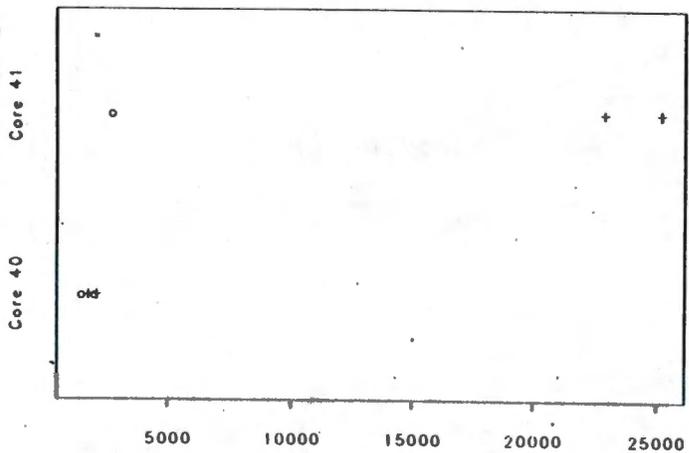
pH



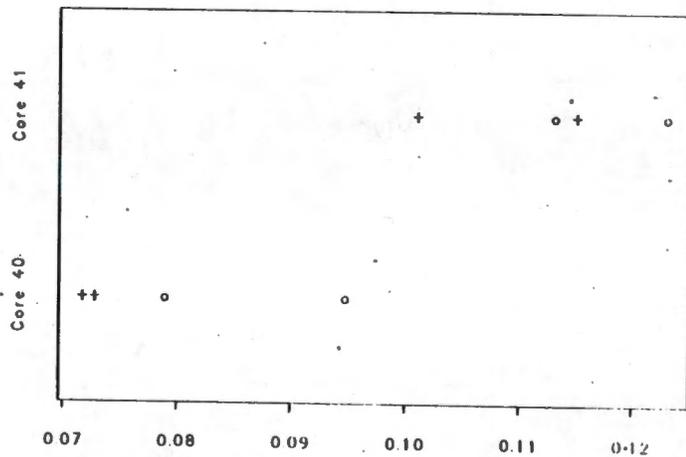
TIC



Fluor.U



RAD.f.AT



Composite 1 = o, Composite 2 = +
Units are ug/g except for radionuclides which are uCi/g

WHC-SD-MM-TI-603, Rev. 0

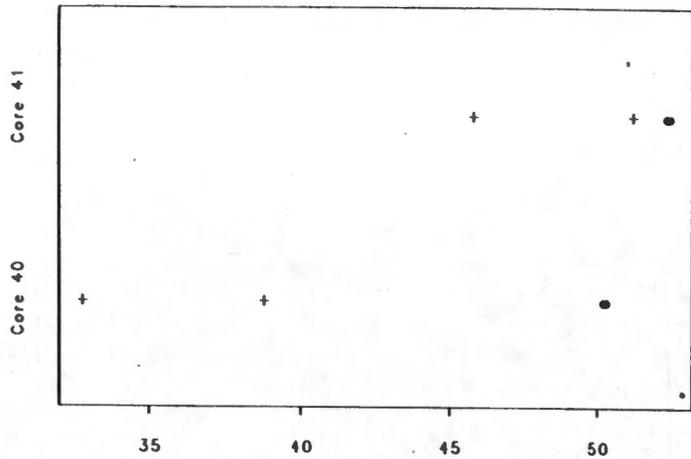
WHC-EP-0739

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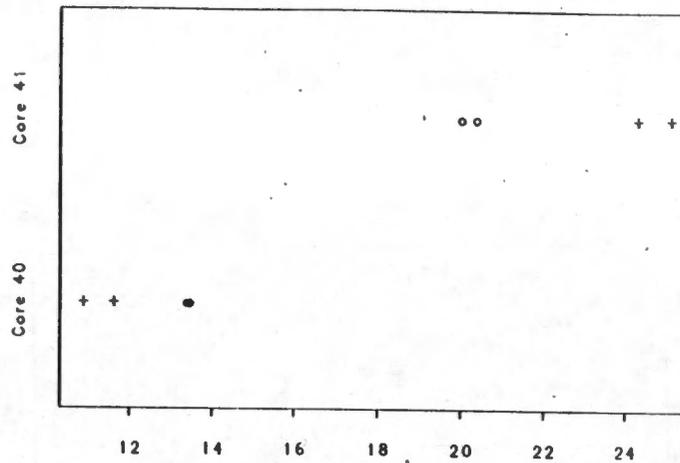
B-58

B-20

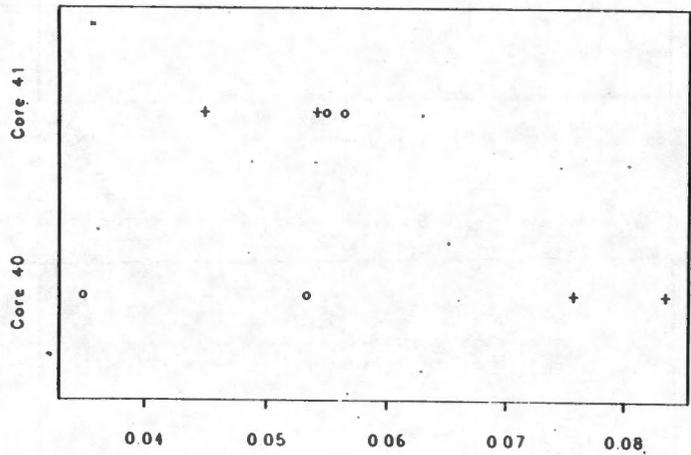
RAD.f.TB



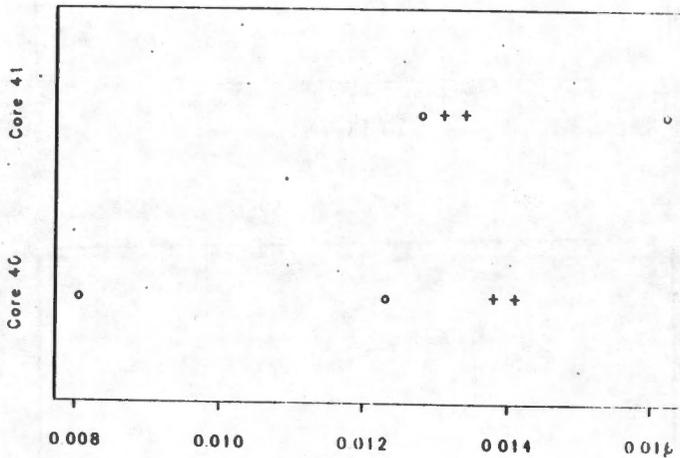
GEA.Cs-137



GEA.PU239/240



GEA.Am-241

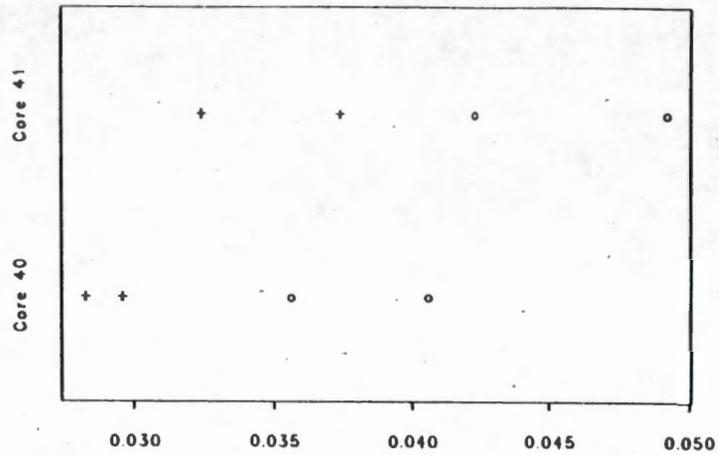


Composite 1 = o . Composite 2 = +
Units are ug/g except for radionuclides which are uCi/g

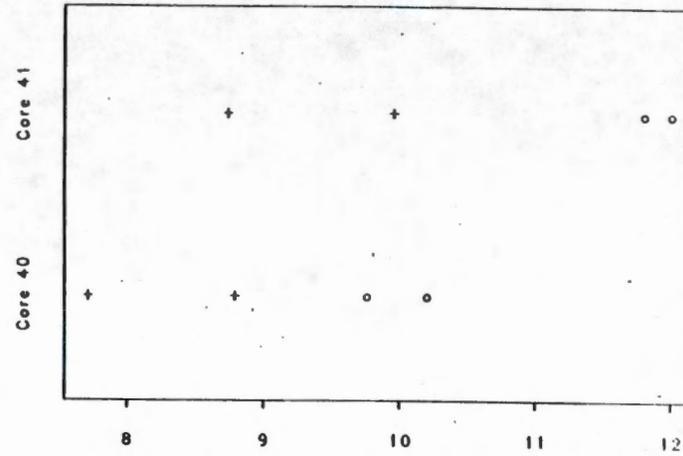
MHC-SD-WM-TI-603, Rev. 0

WHC-EP-0739

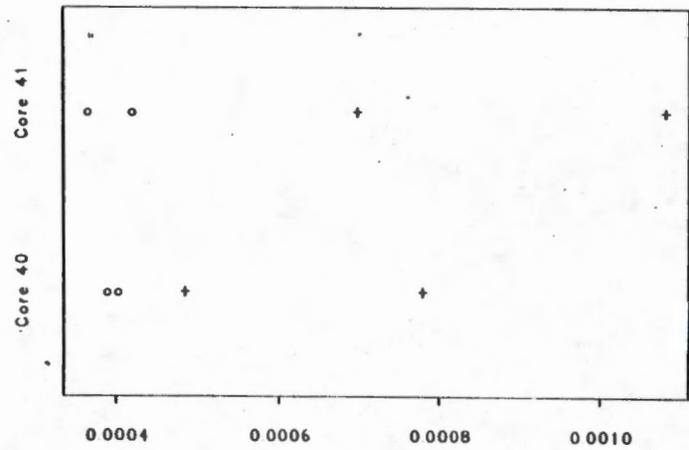
GEA.TC-99



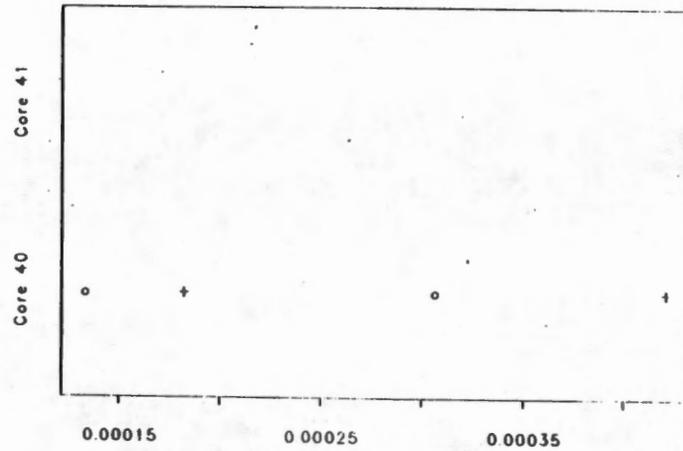
GEA.Sr-90



GEA.H-3



GEA.C-14



Composite 1 = o, Composite 2 = +
 Units are ug/g except for radionuclides which are uCi/g

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WHC-EP-0739

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APPENDIX C
MEAN CONCENTRATION CALCULATION METHODS

C-1

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This appendix includes a description of the statistical model that describes the structure of the data from core samples taken from tank 241-BX-107. Equations are also presented for estimates of the mean concentration, the variance for the mean concentration, and the CI on the mean concentration.

The statistical model that describes the structure of the core composite data are

$$y_{ijk} = \mu + S_i + C_{ij} + A_{ijk}, \quad i=1, \dots, a, \quad j=1, \dots, b_i, \quad k=1, \dots, n_{ij}, \quad (1)$$

where

- y_{ijk} = laboratory results from the k^{th} duplicate of the j^{th} composite of the i^{th} core from the tank,
- μ = the grand mean of all the data,
- S_i = the effect of the i^{th} core (spatial effect),
- C_{ij} = the effect of the j^{th} composite sample from the i^{th} core,
- A_{ijk} = the analytical error associated with the k^{th} duplicate in the j^{th} composite from the i^{th} core,
- a = the number of cores,
- b_i = the number of composite samples in the i^{th} core, and
- n_{ij} = the number of analytical results from the j^{th} composite sample in the i^{th} core.

For cores 40 and 41 there are two core composite samples (i.e., $b_i = 2$).

The variables S_i and C_{ij} are treated as random effects. It is assumed that S_i , C_{ij} , and A_{ijk} are each distributed normally with mean zero and variances of $\sigma^2(S)$, $\sigma^2(C)$, and $\sigma^2(A)$, respectively. Estimates of $\sigma^2(S)$, $\sigma^2(C)$, and $\sigma^2(A)$ were obtained using Restricted Maximum Likelihood Estimation (REML). This method applied to variance component estimation is described by Harville (1977).

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The mean concentration of each analyte of interest in the tank was calculated using the following equation:

$$\bar{y} = \frac{1}{a} \sum_{i=1}^a \bar{y}_{i++} = \frac{1}{a} \sum_{i=1}^a \frac{\sum_{j=1}^{b_i} \sum_{k=1}^{n_{ij}} y_{ijk}}{n_{i+}} = \frac{1}{a} \sum_{i=1}^a \frac{\sum_{j=1}^{b_i} \sum_{k=1}^{n_{ij}} (\mu + S_i + C_{ij} + A_{ijk})}{n_{i+}} \quad (2)$$

where

$$\bar{y}_{i++} = \frac{\sum_{j=1}^{b_i} \sum_{k=1}^{n_{ij}} y_{ijk}}{n_{i+}} \quad \text{and} \quad n_{i+} = \sum_{j=1}^{b_i} n_{ij} \quad (3)$$

This mean gives the results from each core the same weight regardless of the unbalance that may exist for a particular analyte.

The variance of \bar{y} is

$$V(\bar{y}) = C_1 \sigma^2(S) + C_2 \sigma^2(C) + C_3 \sigma^2(A) \quad (4)$$

where

$$C_1 = \frac{1}{a}, \quad C_2 = \frac{1}{a^2} \sum_{i=1}^a \left[\frac{1}{n_{i+}} \right]^2 \left[\sum_{j=1}^{b_i} n_{ij}^2 \right], \quad C_3 = \frac{1}{a^2} \sum_{i=1}^a \left[\frac{1}{n_{i+}} \right] \quad (5)$$

Using $\hat{\sigma}^2(S)$, $\hat{\sigma}^2(C)$, and $\hat{\sigma}^2(A)$ (REML variance component estimates), an estimated variance of \bar{y} is

$$\hat{\sigma}^2(\bar{y}) = C_1 \hat{\sigma}^2(S) + C_2 \hat{\sigma}^2(C) + C_3 \hat{\sigma}^2(A) \quad (6)$$

The approximate degrees of freedom used for $\hat{\sigma}^2(\bar{y})$ is the number of cores with data minus one.

The lower and upper 95% limits (95% LL and 95% UL respectively) on the mean concentration are

$$95\% \text{ LL} = \bar{y} - t_{.975} \sqrt{\hat{\sigma}^2(\bar{y})} \quad \text{and} \quad 95\% \text{ UL} = \bar{y} + t_{.975} \sqrt{\hat{\sigma}^2(\bar{y})} \quad (7)$$

where $t_{.975}$ is the .975 quantile from a Student's t-distribution with the approximate degrees of freedom associated with $\hat{\sigma}^2(\bar{y})$.

APPENDIX C

**LABORATORY AND SAMPLE IDENTIFICATION NUMBERS
AND LABORATORY PROCEDURES**

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Table C-1. Laboratory Identification Numbers
Core Composite Samples.
(222-S Laboratory)

Sample point	STD	BLK	Samples
BX107-C40-C1	J922, J930	J923	J924, J931, J932, J939, J943, J944, J947, J951,
BX107-C40CC1*	J984, J991	J985, J993	J986, J987, J990, J994, J995
BX107-C40-C2			J926, J933, J934, J940, J948, J952
BX107-C41-C1	J928	J929	J925, J935, J936, J941, J945, J946, J949, J953
BX107-C41-C2			J927, J937, J938, J942, J950, J954

*BX107-C40CC1 is the same as sample point BX107-C40-C1.

Table C-2. Laboratory Identification Numbers Core 40 - Segments.
(222-S Laboratory)

Sample point	STD	BLK	Samples	Cask number	Sample number
BX107-C40-S1			J865, J880, J898, J955	C1030	92-019
BX107-C40SIH	J909		J850, J910, J911	NA	NA
BX107-C40-S2	J896		J879, J897, J914, J956	C1027	92-020
BX107-C40S2H			J849, J907, J908	NA	NA
BX107-C40-S3	J978		J864, J878, J895, J957	C1028	92-021
BX107-C40S3H	J904		J846, J905, J906	NA	NA
BX107-C40-S4	J917		J877, J894, J913, J958	C1018	92-022
BX107-C40S4H			J845, J902, J903	NA	NA
BX107-C40-S5			J863, J876, J893, J959	C1023	92-023
BX107-C40S5H	J899		J854, J900, J901	NA	NA
BX107-C40-S6	J891		J875, J892, J912, J960	C1021	92-024
BX107-C40S6H	J853		J843, J854, J855	NA	NA
BX107-C40-S7			J862, J874, J890, J961	1010C	92-025
BX107-C40S7H			J842, J851, J852	NA	NA

Table C-3. Laboratory Identification Numbers Core 41 - Segments.
(222-S Laboratory)

Sample point	STD	BLK	Samples	Cask number	Sample number
BX107-C41-S1	J916		J861, J873, J889, J962	C1031	92-026
BX107-C41S1H	J839		J838, J840, J841	NA	NA
BX107-C41-S2			J860, J872, J888, J963, J964	C1015	92-027
BX107-C41S2H	J834	J835	J831, J832, J833, J836, J837	NA	NA
BX107-C41-S3	J886		J859, J871, J887, J965	1005C	92-028
BX107-C41S3H	J828		J827, J829, J830	NA	NA
BX107-C41-S4			J858, J870, J885, J966		
BX107-C41S4H	J823	J824	J820, J821, J822, J825, J826	NA	NA
BX107-C41-S5			J857, J868, J869, J884, J967	1002C	92-030
BX107-C41S5H			J816, J818, J819	NA	NA
BX107-C41-S6			J856, J867, J883, J968	C1029	92-031
BX107-C41S6H	J812 J817	J813	J808, J809, J810, J814, J815	NA	NA
BX107-C41-S7	J881		J866, J882, J915, J969	C1034	92-032
BX107-C41S7H	J805		J804, J806, J807	NA	NA

Table C-4. Laboratory Identification Numbers for Core 40 and 41 Segments and Composites-PNL.

Pu/U Isotopic by Mass Spec	C40 Comp 1 92-11296-H1 92-11296-H2 92-11296-H3	C40 Comp 2 92-11297-H1 92-11297-H2		C41 Comp 1 92-11298-H1 92-11298-H2	C41 Comp 2 92-09440-H1 92-09440-H2				Sample for Pu/U iso Duplicate Methods blank
Wt% solids	C40 Comp 1 92-11296-K1 92-11296-K2	C40 Comp 2 92-11297-K1 92-11297-K2		C41 Comp 1 92-11298-K-1 92-11298-K-2	C41 Comp 2 92-11299-K-1 92-11299-K-2				Wt% solids sample Wt% solids sample
Semi-VOA	C40 Comp 1 92-11296-E1 92-11296-E2 92-11296-E3 92-11296-E4	C40 Comp 2 92-11297-E1 92-11297-E2		C41 Comp 1 92-11298-E1 92-11298-E2	C41 Comp 2 92-11299-E1 92-11299-E2 92-11299-E3 92-11299-E4				Sample S-VOA and pH Duplicate S-VOA and pH Matrix spike Matrix spike duplicate
TOX/BOX	C40 Comp 1 92-11296-F1 92-11296-F2 92-11296-F3 92-11296-F4	C40 Comp 2 92-11297-F1 92-11297-F2		C41 Comp 1 92-11298-F1 92-11298-F2 92-11298-F4	C41 Comp 92-11299-F1 92-11299-F2				Sample TOX/BOX Duplicate Methods blank Sample spike TOX/BOX
VOA	C40 Seg 5 92-09439-M1 92-09439-M2 92-09439-M3 92-09439-M4	C40 Seg 7 92-09438-M1 92-09438-M2		C41 Seg 2 92-09440-M1 92-09440-M2 92-09440-M3 92-09440-M4	C41 Seg 3 92-09441-M1 92-09441-M2	C41 Seg 5 92-09442-M1 92-09442-M2	C41 Seg 6 92-09443-M1 92-09443-M2 92-09443-M3 92-09443-M4	C41 Seg 7 92-09444-M1 92-09444-M2	Sample VOA Duplicate VOA Matrix spike Matrix spike duplicate
Rheology	C40 Seg 2 92-11293	C40 Seg 4 92-11294	C40 Seg 6 92-11295						Rheology sample

Table C-5. Analytical Methods for Physical and Rheological Testing.

Analyte	Procedure
% Water	PNL-ALO-504 LA-564-101
Rheology	PNL-ALO-501/502
Thermogravimetric analysis	PNL-ALO-508 LA-561-112
Differential scanning analysis	LA-514-113 PNL-ALO-508

Table C-6. Analytical Methods for Chemical and Radionuclide Analyses.

Analyte	Method	Procedure number
Hg	Cold vapor atomic absorption	LA-325-102
F ⁻ , Cl ⁻ , NO ₃ ⁻ , NO ₂ ⁻ , PO ₄ ³⁻ , SO ₄ ²⁻	Ion chromatography	LA-533-105
CN ⁻	Distillation/spectrometric analysis	LA-695-102
Uranium	Laser fluorimetry Alpha energy analysis	LA-925-106 PNL-MA-597
Total alpha Total beta	Proportional counting	LA-508-101
²³⁸ Pu, ^{239,240} Pu, ²⁴¹ Am, ²³⁷ Np	Alpha spectrometry	PNL-ALO-423/ LA-503-156
Total metals	Inductively coupled plasma	LA-505-151
⁹⁰ Sr	Beta proportional counting	LA-220-101
⁹⁹ Tc	Liquid scintillation	LA-438-101
¹²⁹ I	Low energy gamma analysis	LA-378-103
¹⁴ C	Liquid scintillation	LA-348-104
³ H	Liquid scintillation	LA-218-113
¹³⁷ Cs	Gamma energy analysis	LA-548-121
H ⁺	pH	LA-212-103
As Se	Atomic absorption	PNL-ALO-214 PNL-ALO-215
Pu isotopic Ur isotopic	Mass spectrometry	PNL-ALO-455 PNL-MA-697
TOC/TIC	Furnace oxidation Persulfate oxidation	LA-622-102 PNL-ALO-380/381

Table C-7. Analytical Methods For Organic Analyses.

Analysis	Method	Procedure number
VOA	Gas chromatography/mass spectrometry	PNL-ALO-335
SVOA	Gas chromatography/mass spectrometry	PNL-ALO-345
EOX/TOX	Microcoulometric titration	PNL-ALO-320

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