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

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Westinghouse Hanford Company, Richland, WA 99352
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# Tank Characterization Report for Single-Shell Tank 241-BX-107

G. F. Raphael

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February 1996

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Office of Environmental Restoration and
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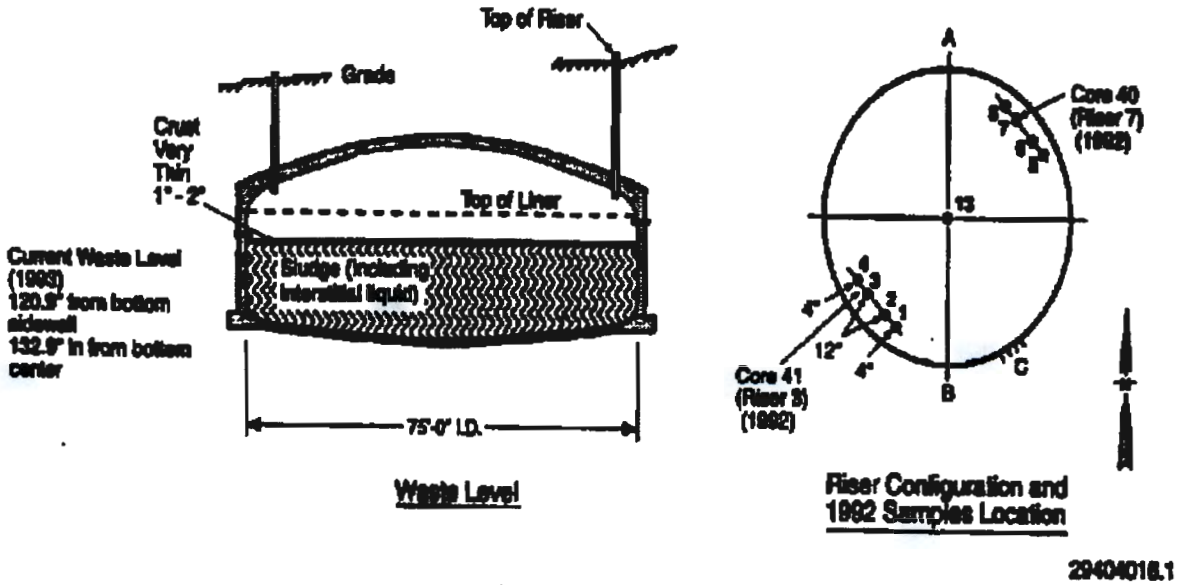
## EXECUTIVE SUMMARY

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 is 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.



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].

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

[2]DOE-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<br><5,000 gal supernatant |
| | Partially interim isolated | Sealed to prevent inadvertent liquid addition |
| Safety classification | Non Watch List | Based on safety criteria, analytical results, and surveillance data |
| Tank capacity | 2,006,050 L<br>(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<br>(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. (3 pages)

| 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 | | $\mu$Ci/g | KCi |
| AT | | 0.0963 | 0.1811 |
| $^{239/240}Pu$ | | 0.0572 | 0.1076 |
| $^{238}U$ | | 7.467E-4 | 0.0014 |
| TB | | 46.7 | 87.81 |
| $^{137}Cs$ | | 17.4 | 32.72 |
| $^{90}Sr$ | | 9.87 | 18.56 |
| Total radionuclides inventory* | | 46.80 | ~88 |

Table ES-2. Chemical, Radiochemical, and Physical Analysis Summary. (3 pages)

| 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. (3 pages)

| Group | Mol. Wt. | 1992 Cores 40 and 41 Average Total Concentration Wet Basis | Tank inventory Based on 1992 core data |
|---|---|---|---|
| Particle size distribution ± 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.

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## LIST OF TERMS

| | |
|---|---|
| AAS | Atomic absorption spectroscopy |
| AEA | Alpha energy analysis |
| AT | Total alpha reading |
| CRQL | Contract Required Quantification Limits |
| CASS | Computer Automated Surveillance System |
| CVAA | Cold vapor atomic absorption |
| DOE | U.S. Department of Energy |
| DOT | U.S. Department of Transportation |
| DSC | Differential scanning calorimetry |
| DST | Double-shell tank |
| EPA | U.S. Environmental Protection Agency |
| EOX | Extractable organic halides |
| FIC | Food Instrument Corporation |
| GC/MS | Gas chromatography/mass spectroscopy |
| GEA | Gamma energy analysis |
| HAS | Hanford Analytical Services |
| HWVP | Hanford Waste Vitrification Plant |
| HYAA | Hydride atomic absorbtion |
| IC | Ion chromatography |
| ICP | Inductively coupled plasma |
| IDL | Instrument detection limit |
| LANL | Los Alamos National Laboratory |
| LOW | Liquid observation wells |
| MDA | Method detectable activity |
| MDL | Method detection limit |
| MW | Metal waste |
| NCPLX | Non-complexed |
| NPH | Normal paraffin hydrocarbon |
| PIC | Person in charge |
| PNNL | Pacific Northwest National Laboratory |
| RPD | Relative percent difference |
| 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 |

## LIST OF TERMS (Continued)

| | |
|---|---|
| TOX/EOX | Total/extractable organic halides |
| Tri-Party Agreement | *Hanford Federal Facility Agreement and Consent Order* |
| TWRS | Tank Waste Remediation System |
| USQ | Unreviewed Safety Question |
| VOA | 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 single-shell tank (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 double-shell tanks (DST) 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 12 (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, dry wells, and liquid observation wells (LOW). 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 LOWs. 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 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).

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

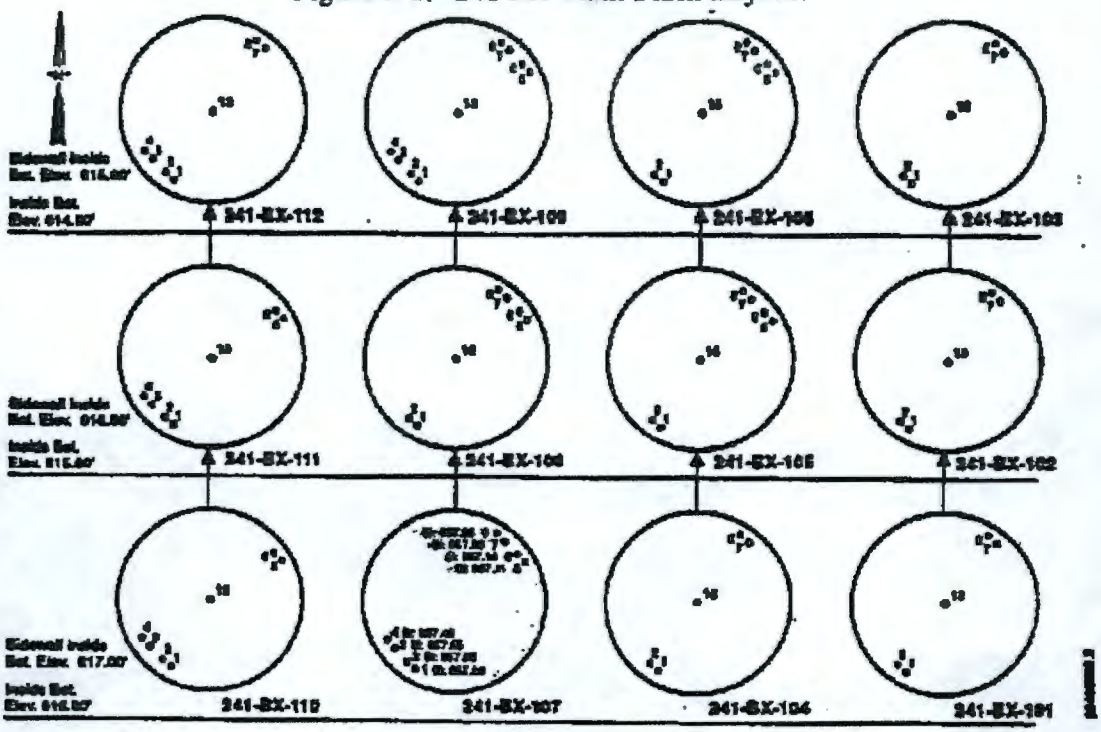
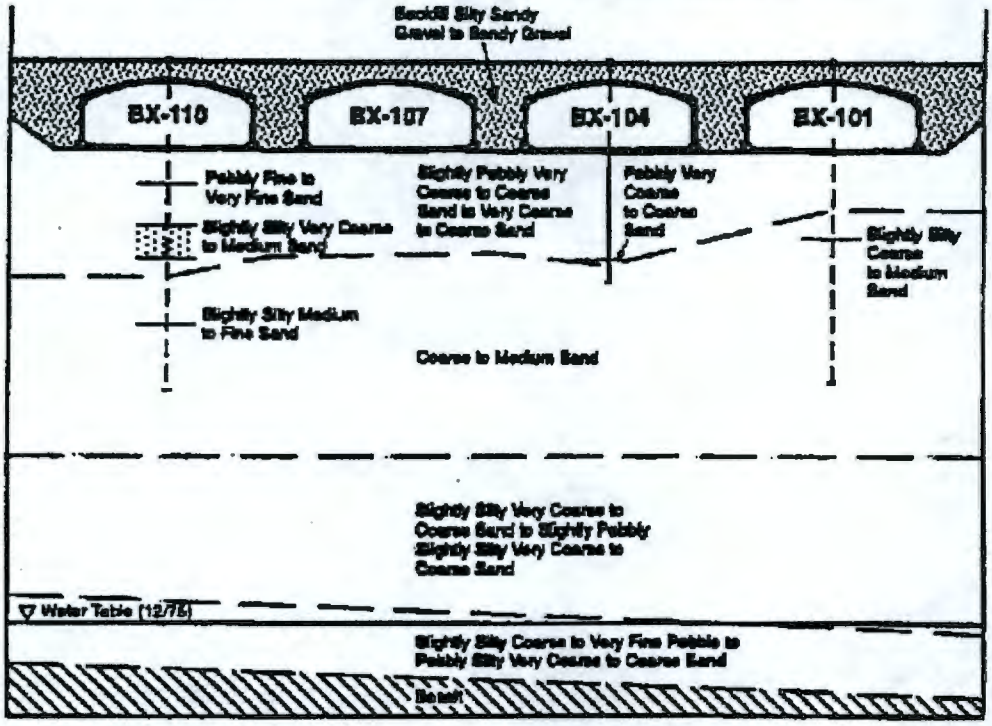



Figure 2-2. 241-BX Tank Farm Vertical Section Layout.



### 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-diameter (75-ft-diameter), 4.88-m (16-ft) elevation tank with a .3-m (12-in.) deep dished bottom. Although 8 vertical risers and 1 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).

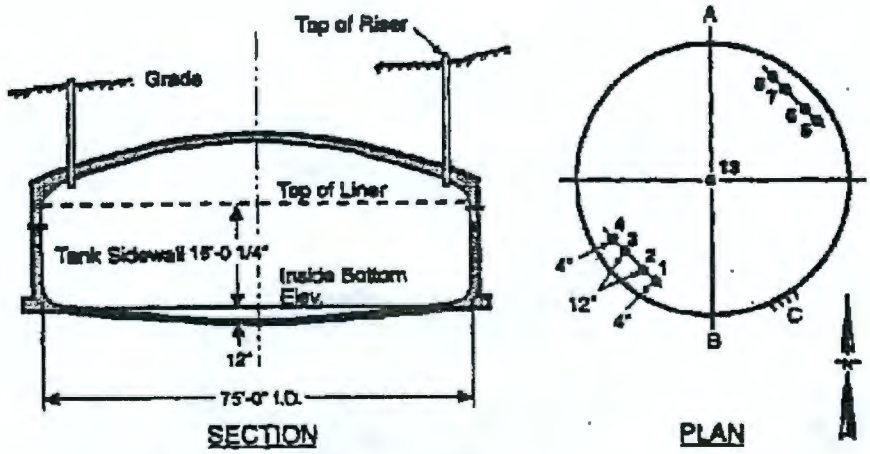
In the last quarter of 1968, 598,030 L (158,000 gal) were transferred to tank BX-106, which left tank BX-107 with 1,548,065 L (409,000 gal) of sludge. The sludge was a mixture of 1C and TBP residues.

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).

*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.

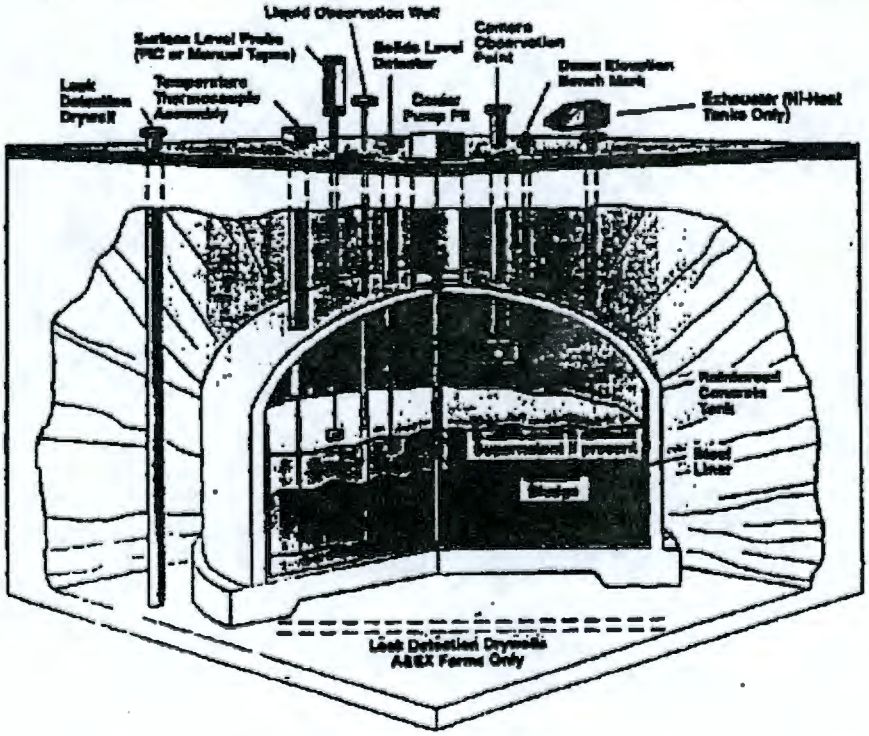
Figure 2-3. Tank 241-BX-107 Configuration.

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 | 657.08 | |

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



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). Figure 2-6 is a top view color 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, Figure 2-6 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 [$Fe_3(PO_4)_2$ (insoluble), $FeSO_4.7H_2O$ (soluble)]. The reddish spots could be attributed to ferric oxide/hydroxide compounds (such as FeO(OH) insoluble crystals). Other ferric and ferrous compounds such as phosphates ($Fe(H_2PO_2)_3$, $Fe_4(P_2O_7)_3.H_2O$,$FePO_4$, $Fe(PO_4)_2$), nitrates ($Fe(NO_3)_3.9H_2O$), and sulfates ($Fe_2(SO_4)_3$) 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).

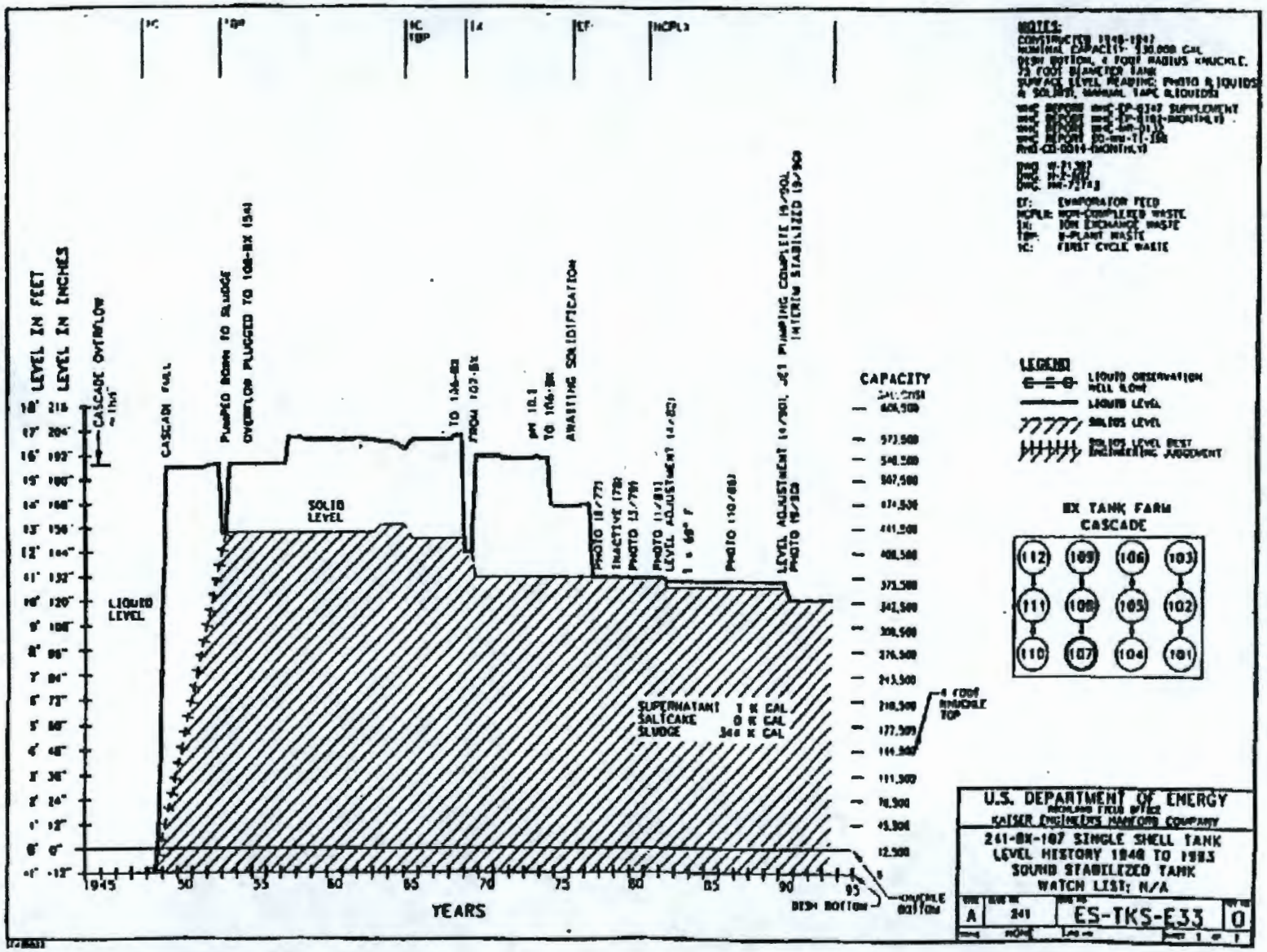



Figure 2-5. Tank 241-BX-107 Waste Level History.

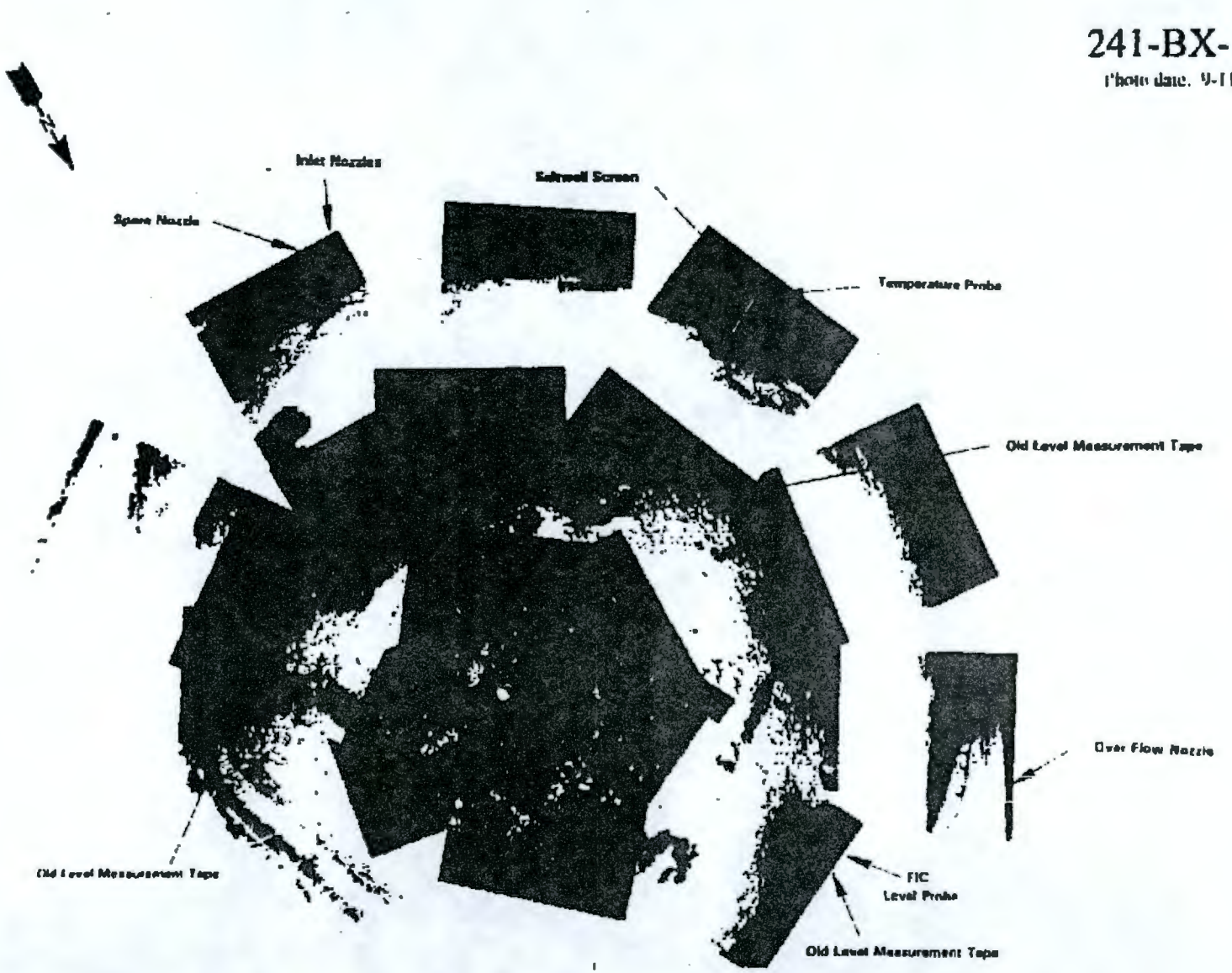



Figure 2-6. Photo Collage of Tank 241-BX-107.

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**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 (USQ) associated with it. It was interim stabilized in September 1990 by jet pumping supernatant liquid and pumpable interstitial liquid.

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 (dry well 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 $BiPO_4$ 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 $BiPO_4$ 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 ($NO_3^-$), and nitrite ($NO_2^-$) are expected to have the highest concentrations, followed by carbonates ($CO_3^{-2}$), sulfates ($SO_4^{-2}$), phosphates ($PO_4^{-3}$), ammonium ($NH_4^+$), silica (Si), aluminum (Al), fluoride ($F^-$), and hydroxide ($OH^-$).

It is expected that other fission products (e.g., $^{89/90}Sr$, $^{129}I$, and $^{14}C$) and organics will be found in tank BX-107 waste.

### 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).

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 |
| $Na_3PO_4$ | 0.75 | 1C |
| $Cs(NO_3)$ | 0.01 | 1C |
| $(NH_4)_2SO_4$ | 0.04 | 1C |
| $(NH_4)_2(SiF_6)$ | 0.07 | 1C |
| $NH_4NO_3$ | 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 |
| $K_4Fe[CN]_6$ | 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 2-7 is a reflection of this data.

Source: Anderson (1990); Schneider (1951).

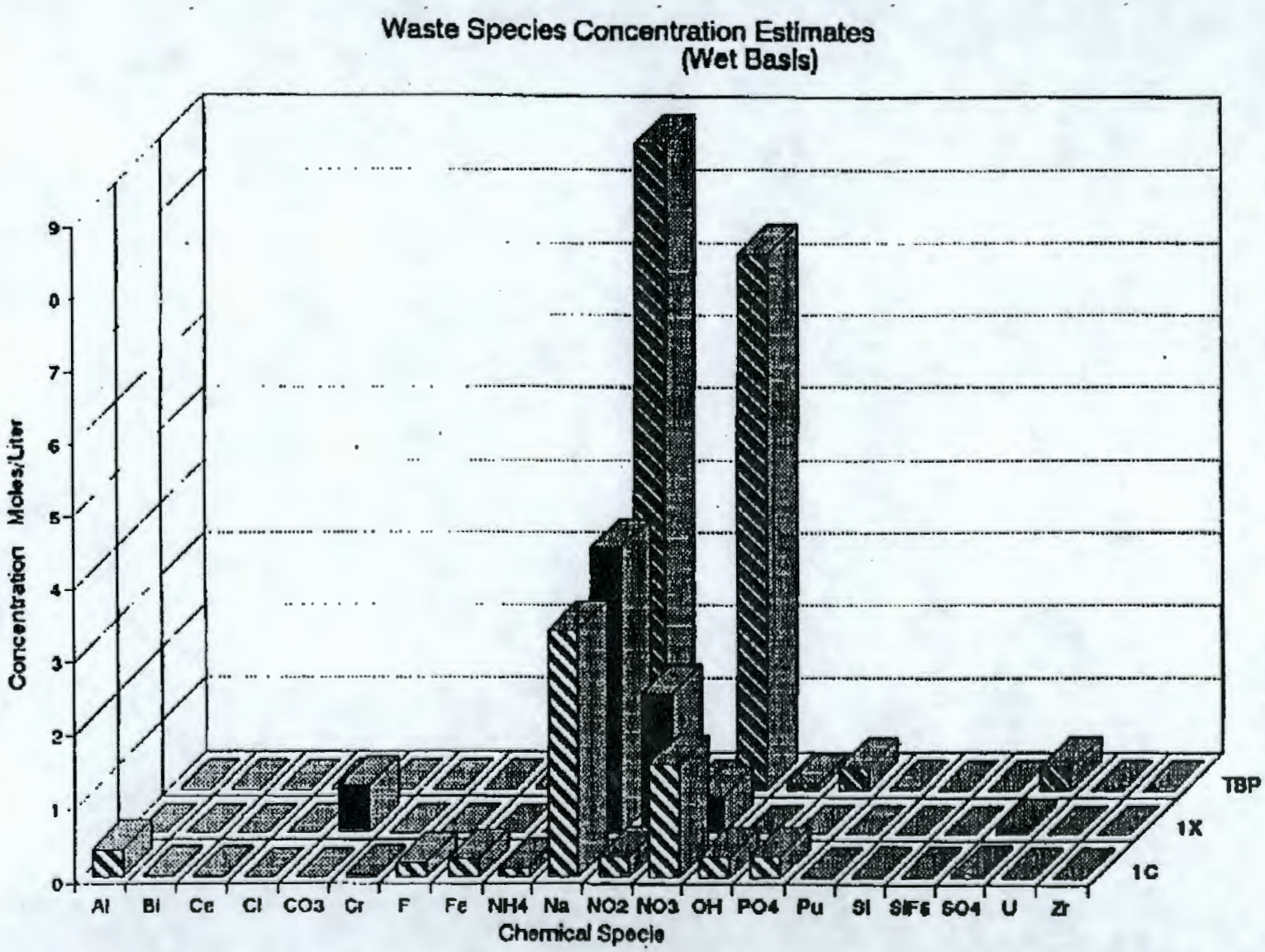


Figure 2-7. Waste Species Concentration Estimates (Wet Basis).

**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 $^{14}C$, 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 $^{99}Tc$ found by sampling is essentially the same as predicted by the TRAC model. However, TRAC under-estimates the inventory of $^{239/240}Pu$ and over-estimates the amounts of $^{241}Am$, $I^{129}$, and $^{14}C$. 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 water were reported at 1.46 g/cm$^3$ 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).

*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) |
|---|---|---|
| $^{241}Am$ | 4E-3 | 3 |
| $^{137}Ba$ | 2E-7 | 2E+4 |
| $^{239}Pu$ | 3 | 5E+1 |
| $^{240}Pu$ | 8E-2 | 5 |
| $^{137}Cs$ | 1 | 2E+4 |
| $^{90}Sr$ | 4 | 5E+4 |
| $^{99}Tc$ | 3 | 5 |
| $^{235}U$ | 7E+1 | 3E-2 |
| $^{238}U$ | 1E+4 | 8E-1 |
| $^{90}Y$ | 1E-3 | 6E+4 |
| $^{93}Zr$ | 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 | $\mu$Ci/gal |
| $^{134}Cs$ | 7.38E+2 |
| $^{137}Cs$ | 3.4E+5 |

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

| Analyte | Composite $H_2O$ soluble Weight % | Composite $H_2O$ 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 |

[a]Possibly attributed to reporting error.
[b]Possibly 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 |
|---|---|---|---|---|---|---|
| $^{241}Am$ | NR | NR | NR | NR | 2.7E-9 | g/g |
| $^{134}Cs$ | 8.8 | 3.3 | 5.2 | NR | NR | μCi/kg |
| $^{137}Cs$ | 2.01E+5 | 8.21E+4 | 2.74E+3 | 2.74E+3 | 8.37E+3 | μCi/kg |
| $^{154}Eu$ | 24 | 12 | 21 | NR | NR | μCi/kg |
| $^{155}Eu$ | 1.35E+3 | 68 | 1.01E+3 | NR | NR | μCi/kg |
| Pu | NR | NR | NR | 8.51E-11 | 1.75E-6 | g/g |
| $^{89/90}Sr$ | NR | NR | NR | 2.3E-3 | 14.2 | μCi/g |
| U | NR | NR | NR | 5.5E-7 | 7.99E-5 | g/g |
| $^{144}Ce$ | NR | NR | NR | <2.54E-2 | 8.37 | μCi/g |

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.

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

| Analyte | Concentration | |
|---|---|---|
| Metallic elements | Moles/L | Weight percent |
| 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 | $\mu$Ci/L | |
| Alpha total | 2.9 | NR |
| Total beta | 2.88E4 | NR |
| $^{137}Cs$ | 2.49E4 | NR |
| $^{89/90}Sr$ | 5.71 | NR |
| $^{239/240}Pu$ | 4.24 | NR |
| $^{241}Am$ | 2.49E-1 | NR |
| Organics | grams/L | |
| TOC | 2.7 | NR |
| Physical properties | | |
| Water (weight %) | 72 | |
| pH | 9.64 | |

**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 $\mu$Ci/gal in 1974, were converted to $\mu$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 $CO_2$, 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 $NO_2^-$ and higher soluble $NO_3^-$ concentrations were possibly because of the oxidation of nitrite to nitrate.

- Increase in $PO_4^{-3}$ 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).

- 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 2-8 depicts the tank BX-107 1974 and 1990 supernatant analytical results for select inorganic analytes.

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 2-8 reflects this data.

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

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

**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 $\mu$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$^3$.
- 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.

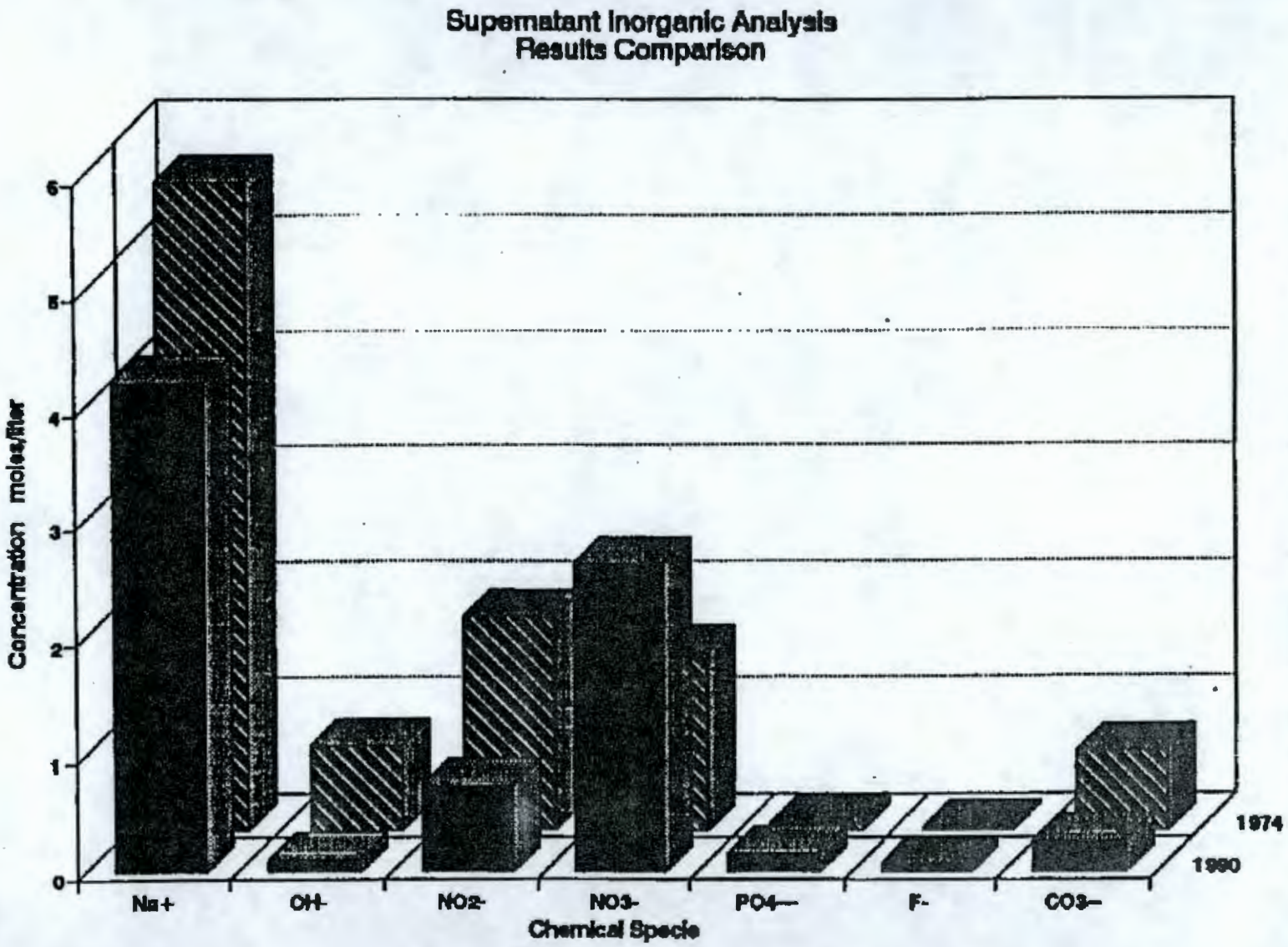


Figure 2-8. Supernatant Inorganic Analysis Results Comparison.

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 |

[a]Possibly due to unit reporting error.
[b]Possibly 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: $NO_3^-$ and $SO_4^{-2}$ 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 $\mu$Ci/g.

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

Figures 2-9 and 2-10 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 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 $\mu$Ci/L, respectively. TOC data suggests that organic compounds present are water soluble.

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

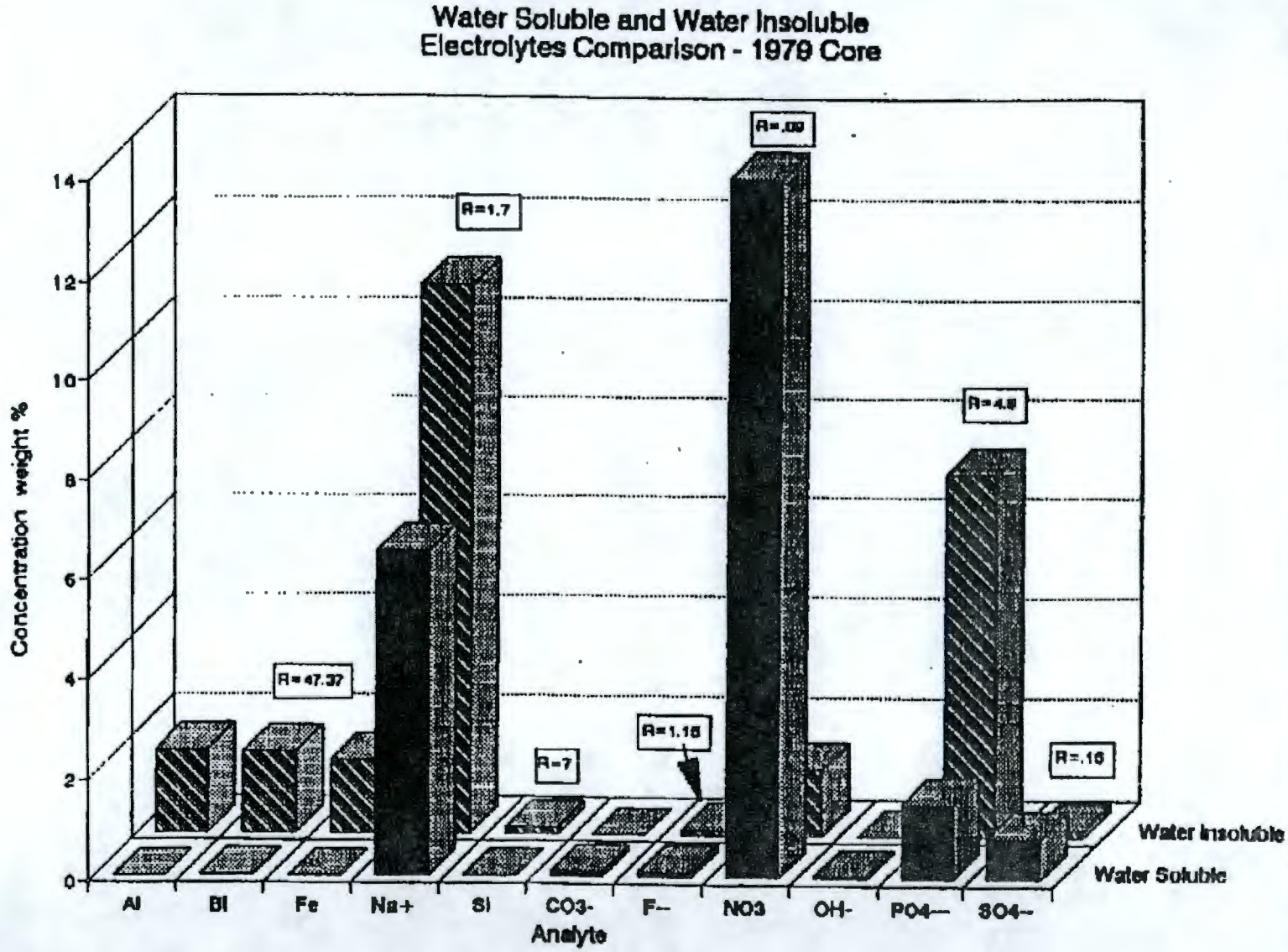


Figure 2-9. Water Soluble and Water Insoluble Electrolytes Comparison - 1979 Core.

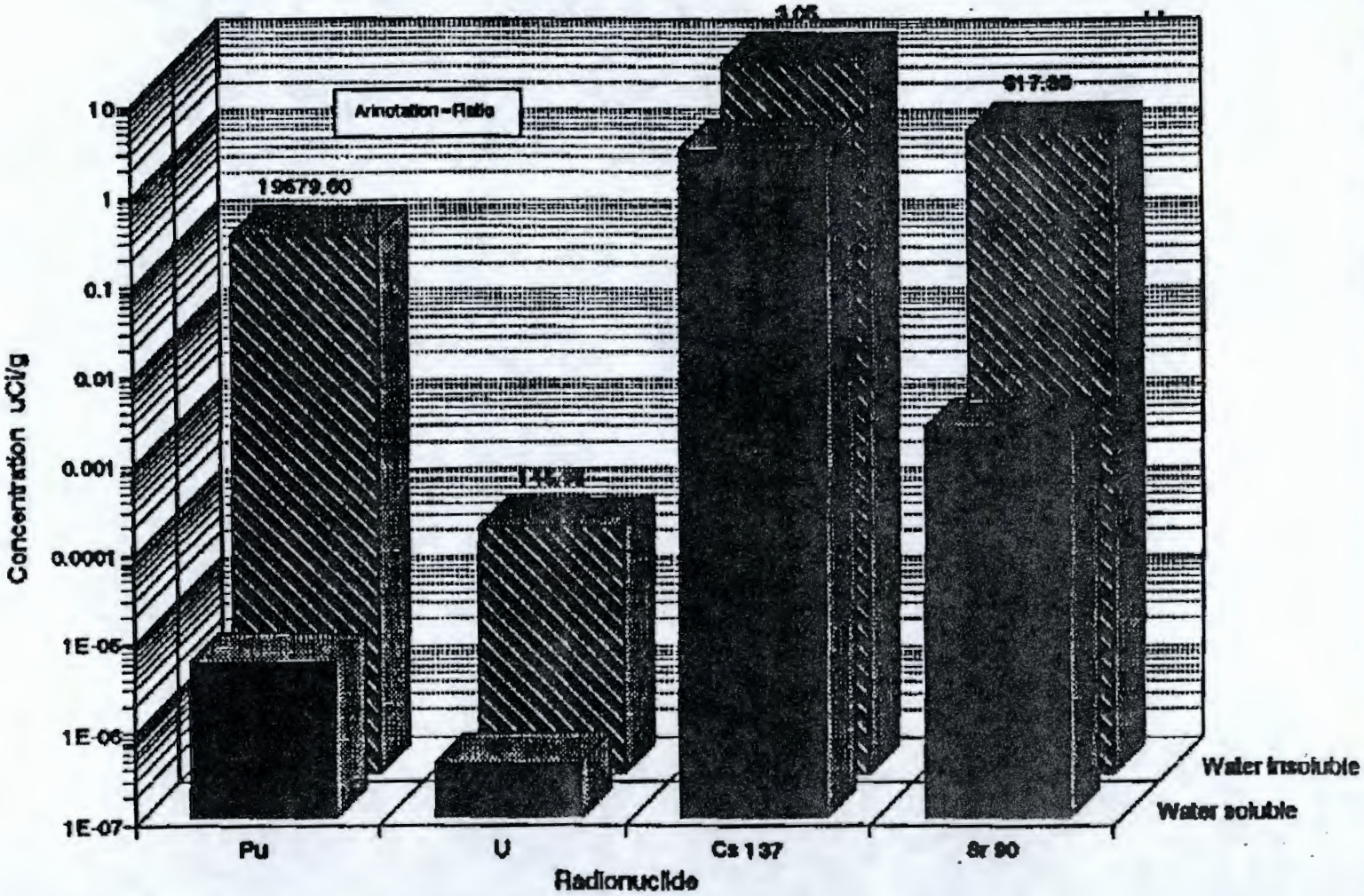


Figure 2-10. Water Soluble and Water Insoluble Radionuclides Comparison - 1979 Core.

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 $\mu$Ci/g | | | |
| $^{239/240}Pu$ | 67E-8 | 2.76E-2 | 0.103 |
| $^{238}U$ | 6.27E-4 | 4.02E-4 | 5.67E-5 |
| $^{93}Zr$ | NR | 1E-3 | NR |
| $^{241}Am$ | NR | 1.51E-3 | NR |
| $^{137}Cs$ | NR | NR | 11.11 |
| $^{137}Ba$ | NR | 10.05 | NR |
| $^{90}Sr$ | NR | 25.13 | 14.2 |
| $^{99}Tc$ | NR | 2.51E-3 | NR |

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

**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 ($H_2SiF_6.2H_2O$ or $(NH_4)_2SiF_6.xH_2O$) 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.

- 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.

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 |
| $Na_2CO_3.10H_2O$ | 1.46 | Transparent | 1:3 |
| NaOH | 2.13 dry | white | 1:1 |
| $Na_2SO_4.10H_2O$ | 1.46 | | 1:1.5 |
| $Na_2HPO_4.7H_2O$ | 1.7 | Hygroscopic powder | 1:8 |
| $NaHPO_4.2H_2O$ | 1.9 | Colorless crystals | 1:1 |
| $Na_3PO_4.12H_2O$ | 1.6 | Colorless to white crystals | 1:1.35 |

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.

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

| Major compounds | Other equilibrium species |
|---|---|
| $Al(OH)_3.xH_2O$, $AlPO_4$ | $Al(OH)_3.xSiO_2$, plus other aluminum salts and oxides |
| $BiPO_4$ | $Bi(OH)_3$ plus other bismuth salts |
| $Fe(OH)_2.xH_2O$, $Fe(SO_4)$ | $FeF_3$, $Fe_3(PO_4)$, other ferrous and ferric salts, other sulfates and hydroxides |
| $Na_4SiO_4$, Metal silicate | $H_2SiF_6.2H_2O$, $(NH_4)_2SiF_6$ |
| $NaNO_3$ | NaOH, other sodium salts |
| $U(PO_4)$, $(UO_2)_3(PO_4)_2$, $UO_2(OH)$ | Other uranium salts and hydroxides, $UO_2$ embedded in a phosphate matrix |
| Metal nitrates, carbonates and sulfates | Metal phosphate and hydroxides |

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.

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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.-) diameter 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 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.

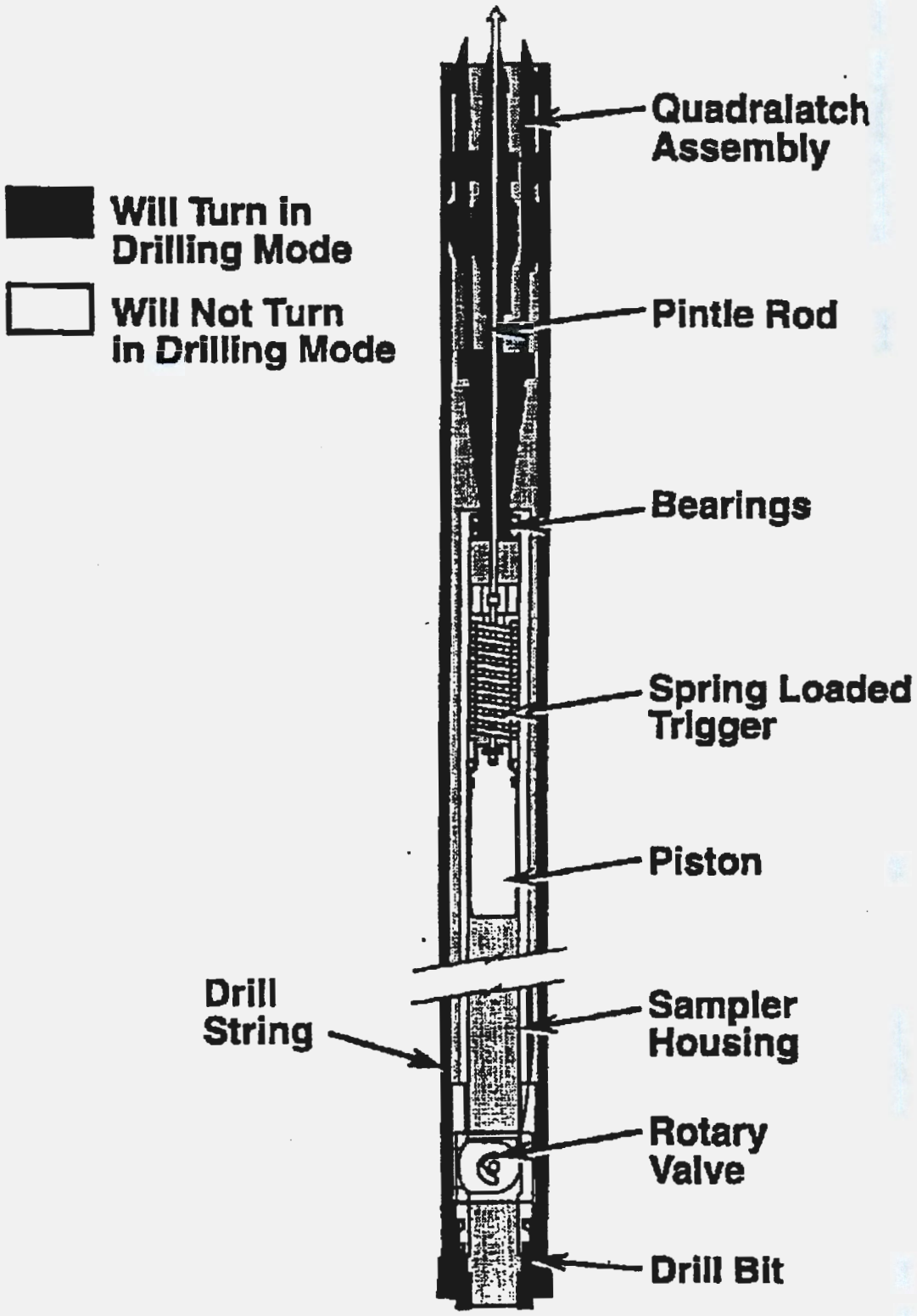
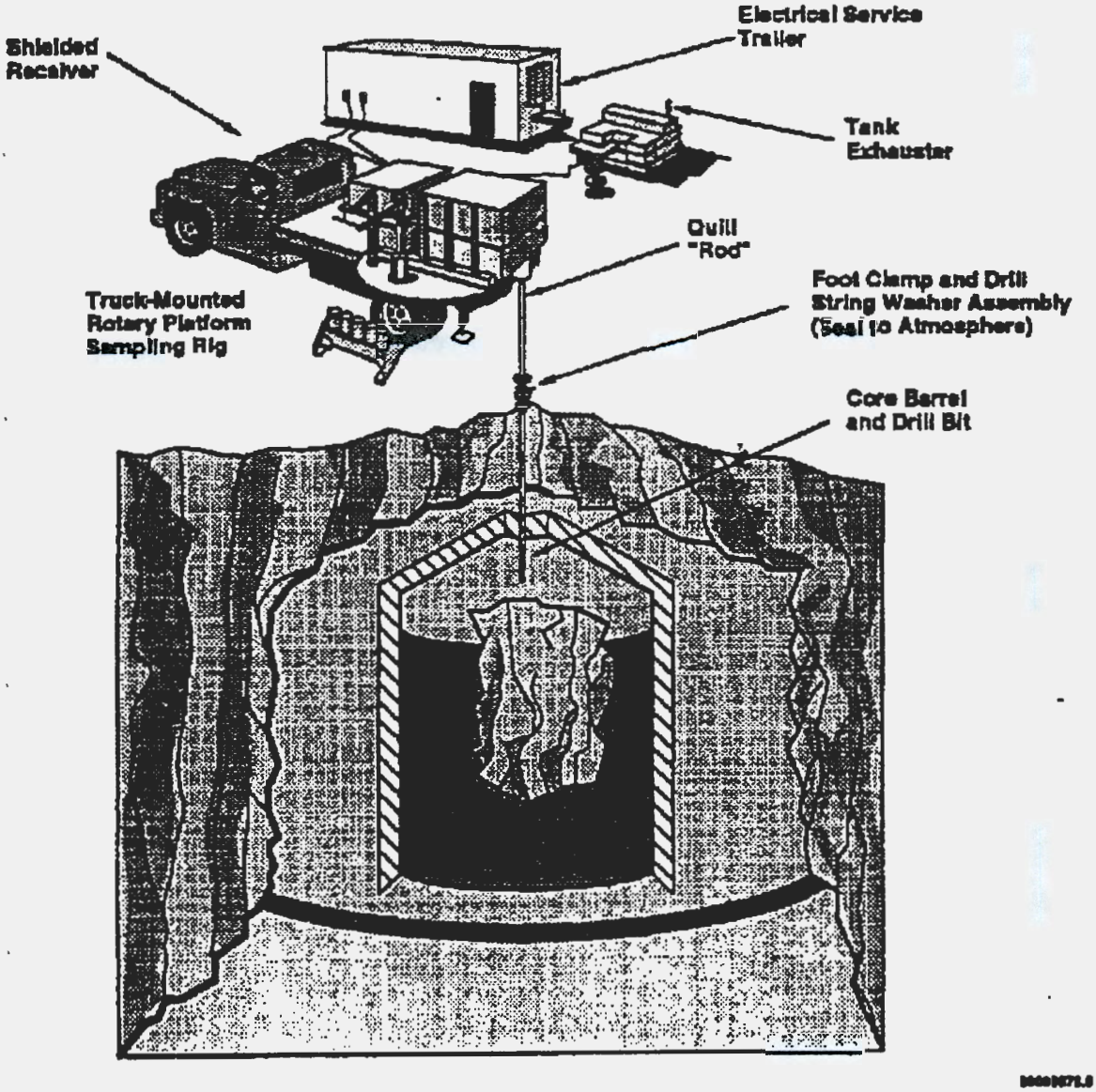


Figure 3-2. Mobile Sampling Truck.

## Waste Characterization



Core sampling of SSTs is performed in accordance with approved Westinghouse Hanford Company (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, Section A.1) 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/m$^2$ (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.

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%.

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).

## 3.3 SAMPLE TRANSFER TO LABORATORY HOT CELL

### 3.3.1 Sample Packaging for Transportation

Sample loaded casks were sealed and transported in 4 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 U.S. Department of Energy (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.

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.

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 |

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

# 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 volatile organic analysis (VOA), percent water, and differential scanning calorimetry (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, identification, 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.

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 |

### 4.1.2 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 VOA 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.

### 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.

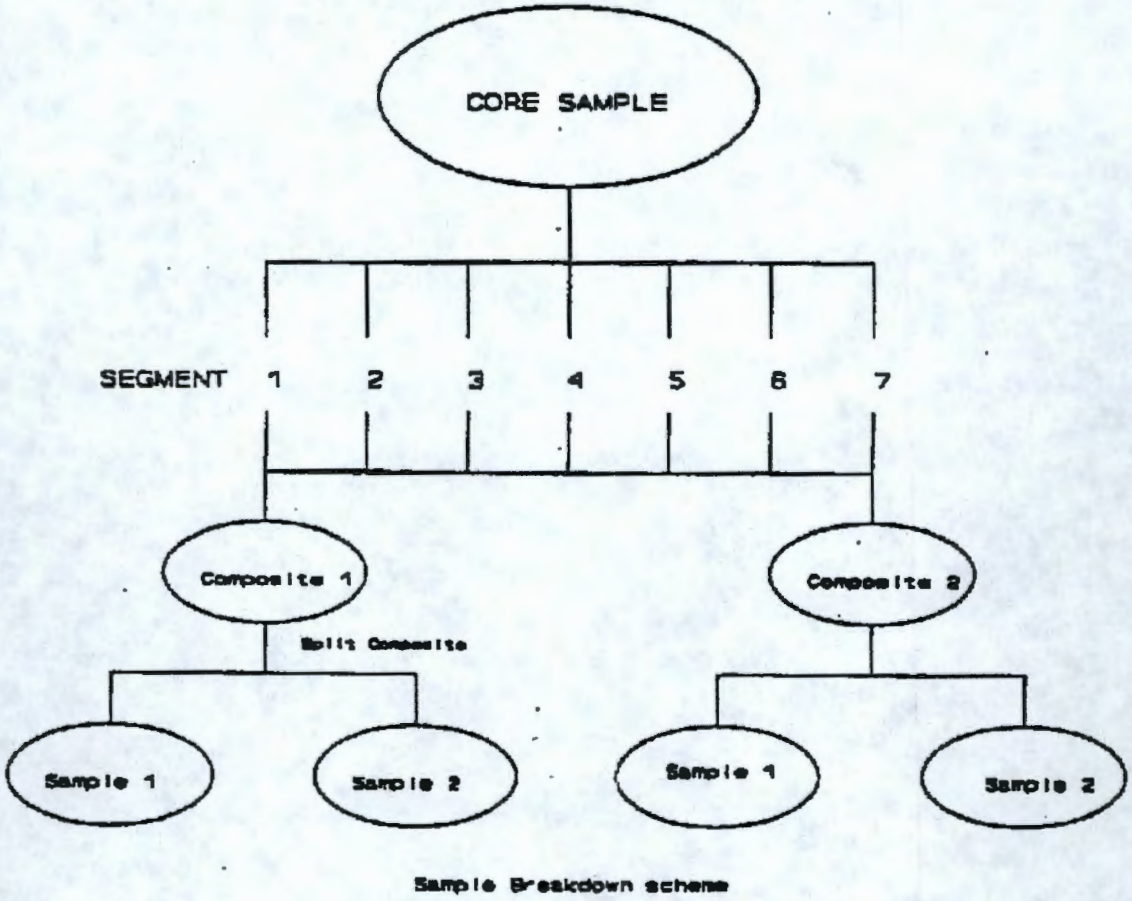
### 4.1.5 Sample Archiving

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 Hanford Analytical Services (HAS).

## 4.2 SAMPLES TRANSFER SCHEME

Sub-samples of the composites and of the core segments were shipped to the Pacific Northwest National Laboratory (PNNL) 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.

Figure 4-1. Sample Breakdown Scheme.



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.

Core 40 (segments 7 and 5) and core 41 (segmients 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 PNNL 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, thermal-gravimetric analysis (TGA), DSC, and particle size analysis remained in the 222-S Laboratory.

# 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 PNNL 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, extractable organic 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 identifications. Appendix A contains tabulated segment identification number information and laboratory procedure numbers corresponding to the analytical tests applied (see tables A-10 through A-16). 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

Samples were prepared for analysis in PNNL-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.

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

| | |
|---|---|
| VOAs (PNNL) | Core 40, segments 5 and 7<br>Core 41 segments 2, 3, 5, 6, 7 |
| Density, % solids, rheology (PNNL) | Core 40, segments 2, 4, 6 |
| SVOA, EOX (PNNL), TOC (222-S) | Core 40, composites 1 and 2<br>Core 41, composites 1 and 2 |
| Solids, metals, anions, radionuclides (222-S) | |
| TGA/DSC (222-S) | All core segments and composites |

## 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 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.

Particle size analysis was performed at the 222-S Laboratory using scattering laser technology following sonication suspension of the sample aliquots in a deionized 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 (Figures A-3 through A-5).

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 (PNNL).

| | 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 (PNNL).

| 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 | W%ight % | 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 - 1μm | 1.5 - 1.5μm | 1.5 - 2μm | 2 - 5μm | 5 - 10μm | 10 - 20μm | 20 - 150μm |
| 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 PNNL.

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

At the PNNL, 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 $\mu g/g$, 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 4E-7 $\mu Ci/g$, which is approximately three orders of magnitude lower than the samples and indicates that there was little contamination of the samples during preparation (PNNL).

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 $^{137}Cs$ shows good results agreement. Cesium-137 beta activity was based on results reported from gamma spectroscopy.

There are also indications that $^{129}I$ 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 recovery 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.

$^{238}U$ and $^{239}Pu$ are the dominant isotopes present with lesser quantities of $^{235}U$ and $^{240}Pu$, 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 package.

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

## 5.5 THERMAL-GRAVIMETRIC ANALYSIS RESULTS

### 5.5.1 Methods

TGA and 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).

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.

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

| Core | Composite | Uranium µg/g | Plutonium µCi/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 (PNNL).

| Isotope | Mass percent range core 40 | Mass percent range core 41 |
|---|---|---|
| $^{234}U$ | 0.0051 - 0.0068 | 0.0055 - 0.0077 |
| $^{235}U$ | 0.6753 - 0.7003 | 0.6738 - 0.6834 |
| $^{236}U$ | 0.0055 - 0.103 | 0.0054 - 0.0077 |
| $^{238}U$ | 99.2843 - 99.3133 | 99.3034 - 99.3154 |
| $^{238}Pu$ | 0.0036 - 0.0043 | 0.004 - 0.0042 |
| $^{239}Pu$ | 96.8142 - 96.9950 | 97.0820 - 97.1357 |
| $^{240}Pu$ | 2.9457 - 2.9916 | 2.8367 - 2.8514 |
| $^{241}Pu$ | 0.0347 - 0.1301 | 0.0193 - 0.0442 |
| $^{242}Pu$ | 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 |
|---|---|---|---|---|---|
| $^{238}U$ | μg/g | 1.82E+3 | 2.02E+3 | 2.82E+3 | 2.41E+4[a] |
| $^{238}U$ | μCi/g | 6.09E-4 | 6.79E-4 | 9.48E-4 | (8.09E-4) |
| AT | μCi/g | 8.68E-2 | 7.23E-2 | 1.81E-1 | 1.08E-1 |
| TB | μCi/g | 5.03E+1 | 3.58E+1 | 5.24E+1 | 4.85E+1 |
| $^{137}Cs$ | μCi/g | 1.34E+1 | 1.13E+1 | 2.02E+1 | 2.47E+1 |
| $^{90}Sr$ | μCi/g | 9.97E00 | 8.25E00 | 1.19E+1 | 9.35E00 |
| $^{241}Am$ | μCi/g | 1.02E-2 | 1.40E-2 | 1.45E-2 | 1.33E-2 |
| $^{239/240}Pu$ | μCi/g | 4.40E-2 | 7.96E-2 | 5.56E-2 | 4.95E-2 |
| $^{99}Tc$ | μCi/g | 3.81E-2 | 2.90E-2 | 4.57E-2 | 3.49E-2 |
| $^{3}H$ | μCi/g | 3.96E-4 | 6.31E-2 | 3.93E-4 | 8.89E-4 |
| $^{14}C$ | μCi/g | 2.20E-4 | 3.02E-4 | 3.19E-4 | NR |

[a]Could be due to data transcription error.

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 relative percent differences (RPD) 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.

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 |

## 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 (ICP) spectroscopy. For analytes with low ICP sensitivity, atomic absorption spectroscopy (AAS) was used. Hydride atomic absorption (HYAA) reduction method was used for arsenic and selenium, and cold vapor atomic absorption (CVAA) technique 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 AAS analysis for arsenic and selenium.

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 detection limit, the laboratory software program returns an RPD of 200%.
- 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 $\mu g/g$, for cores 40 and 41 composite samples.

The preparation blanks concentrations, which mostly read below 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 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 PNNL and 222-S Laboratory manuals, or the tank BX-107 cores 40 and 41 data package.

Appropriate quality assurance/quality control 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.

IDL 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 $\mu g/g^*$ (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[a] | 183[a] | 182[a] | 183[a] |
| 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 $\mu g/g$* (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 $\mu g/g$* (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 |

[a]Biased due to interference. Fusion is not suitable for most trace metals because of larger dilution required.
[b]Not valid results due to method contamination (nickel crucibles).
*Wet basis.

### 5.7.2 Results

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

## 5.8 ORGANIC ANALYSIS RESULTS

### 5.8.1 Methods

TOC, total organic/extractable organic halides (TOX/EOX), SVOA and 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 used 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 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 PNNL 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 $\mu g/g$* (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.

SVOAs were extracted on November 11, 1992, and analyzed on December 12, 1992. No dilutions were necessary for SVOAs. 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 $\mu g/g$ for cores 40 and 41 composites.

No volatile EPA target compounds in concentration above the Contract Required Quantification Limits (CRQL) 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.

Table 5-11. Total Organic Carbon Results for Cores 40 and 41 Composites in μg/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 μg/g (PNNL).

| 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.

Water-soluble organic halides contents were < 10 μg/g for sample and blanks, based on the total wet weight of the sample in core 40. Core 41 EOXs were below MDL.

SVOA 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.

Table 5-13. Semi-Volatile Organics Results for Core 40 Composites in μg/g (PNNL).

| 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 μg/g (PNNL).

| 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.

Table 5-15. Overall Data Summary and Inventory Estimates (Wet Basis). (3 pages)

| Group | Mol. wt. | Process history | TRAC | 1979 core total | 1992 cores 40 and 41 average total | Tank inventory based on 1992 core data |
|---|---|---|---|---|---|---|
| Metals | | µg/g | µg/g | µg/g | µg/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). (3 pages)

| 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 | | μCi/g | μCi/g | μCi/g | μCi/g | KCi |
| AT | | NR | NR | NR | 0.0963 | 0.1811 |
| $^{241}$Am | | NR | 1.51E-3 | | 0.013 | 0.0244 |
| $^{239/240}$Pu | | 6.7E-7 | 2.76E-2 | 0.1027 | 0.0572 | 0.1076 |
| $^{238}$U | | 6.27E-4 | 4.02E-4 | 5.64E-5 | 7.467E-4 | 0.0014 |
| TB | | NR | NR | NR | 46.7 | 87.81 |
| $^{137}$Cs | | NR | 9.7437 | 11.11 | 17.4 | 32.72 |
| $^{99}$Tc | | NR | 2.51E-3 | NR | 0.0369 | 0.0693 |
| $^{90}$Sr | | NR | 25.13 | 14.2 | 9.87 | 18.56 |
| $^{14}$C | | NR | 9.744E-5 | NR | 2.61E-4 | 4.91E-4 |
| $^{3}$H | | NR | NR | NR | 3.77E-4 | 7.09E-4 |
| Total radionuclides inventory | | | | 25.41 | 46.8 | ~88 |
| Anions | | μg/g | μg/g | μg/g | μg/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). (3 pages)

| 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 (Table A-7).

## 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), 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
- 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 PNNL. Those blanks were prepared and analyzed separately from the tank BX-107 cores 40 and 41 samples for volatile and semi-volatile organic compounds.

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

Acetone was the only volatile compound observed above the CRQL. Phtalates 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 Toxic Characteristics Leaching Procedure (TCLP) extraction in the 222-S Laboratory. 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.

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 |

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.

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# 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 (see Figures A-3 through A-5). 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 $\mu$m 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 $\mu$m while approximately 77% of the volume is occupied by particles from 5 to 150 $\mu$m. Table 6-6 is an overall summary of the data. Figure 6-1 is a reflection of the data in Table 6-6.

## 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 (see Table A-6). 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 that 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.

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

| Particle size interval microns | Approximate average number distribution (± 2%) | Approximate number distribution range (± 20%) | Approximate average volume distribution (± 2%) | Approximate volume distribution range (± 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 |

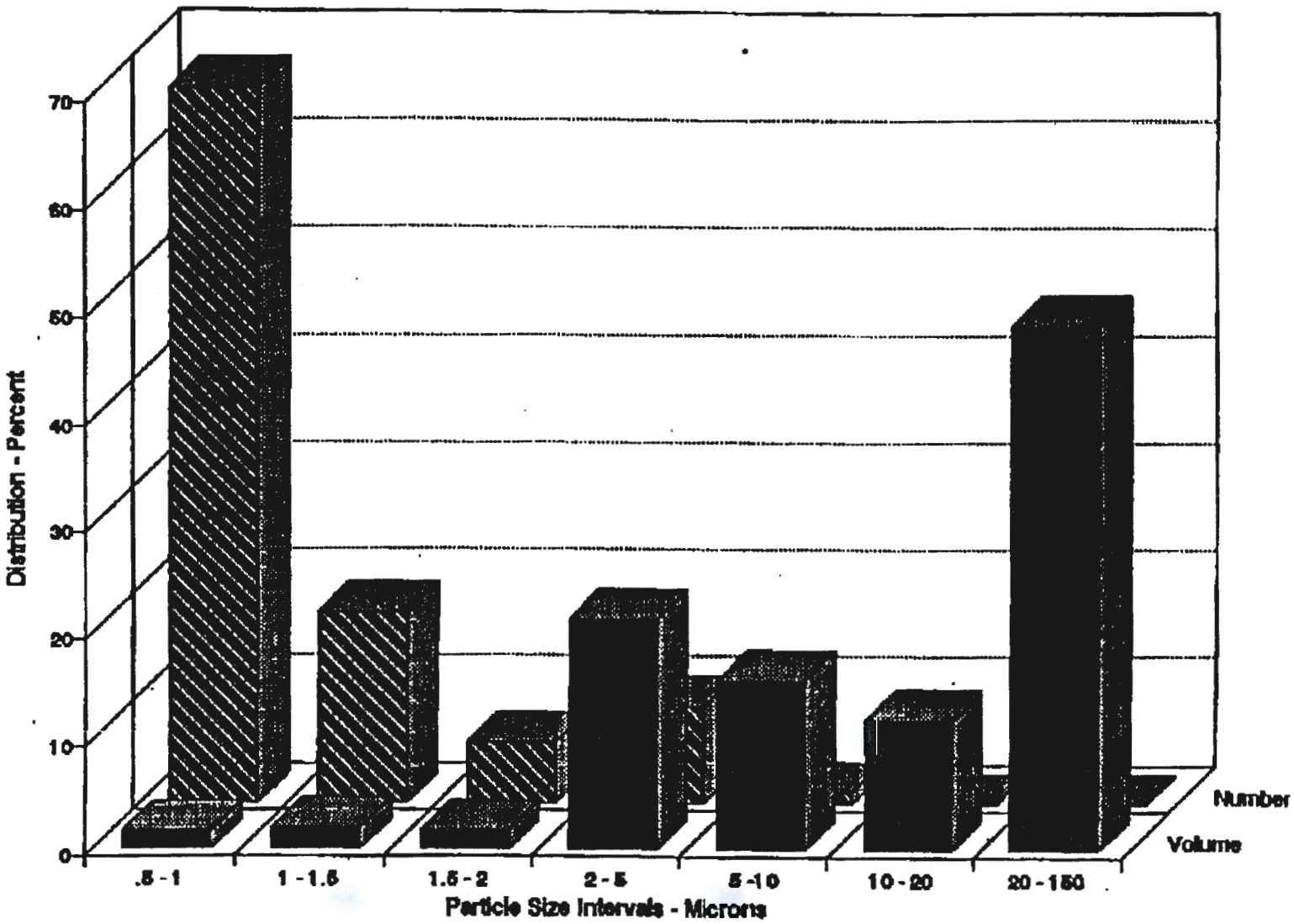


Figure 6-1. Particle Size Distribution Summary.

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

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

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

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

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

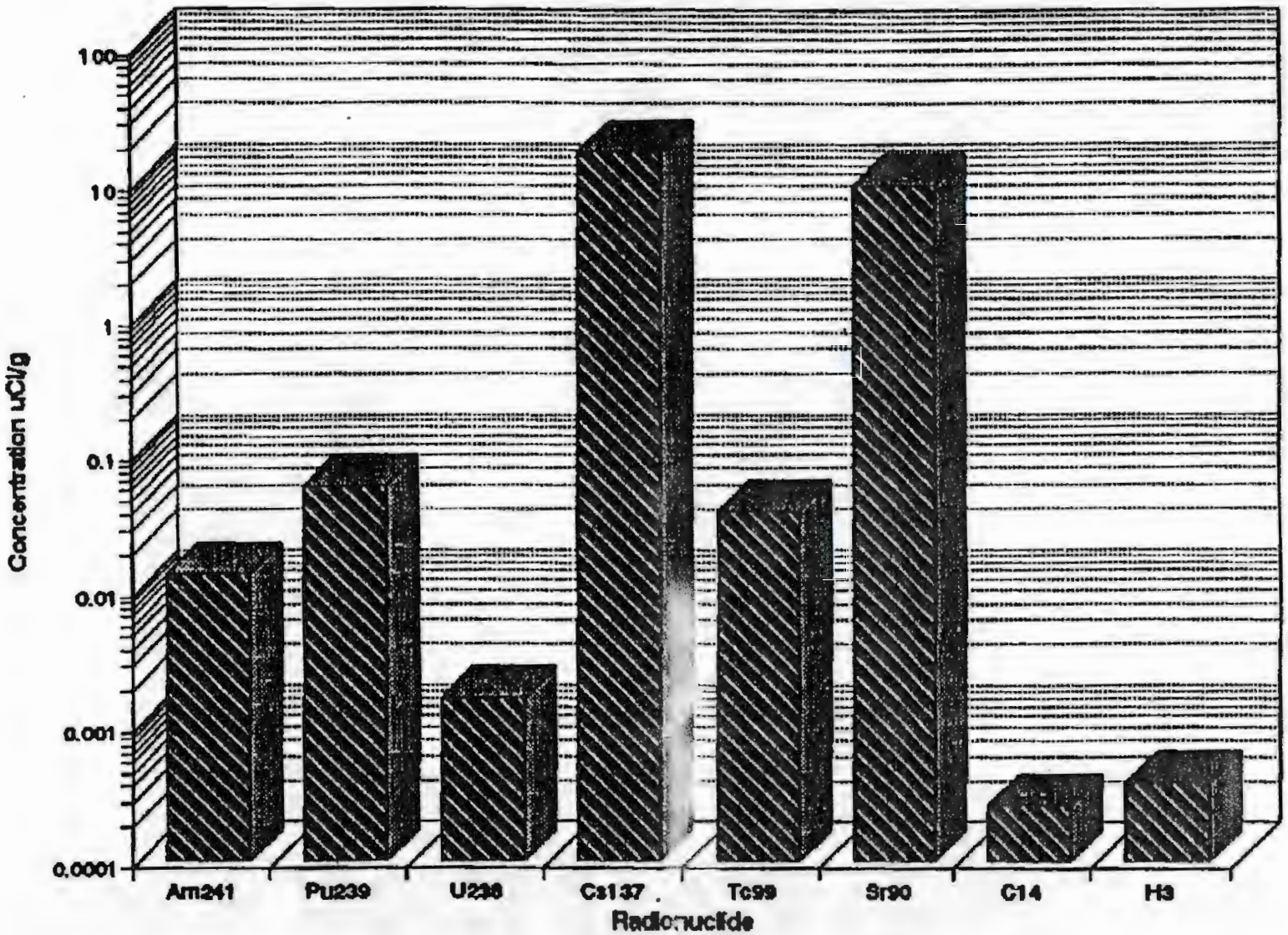


Figure 6-2. Radionuclides Profile - 1992 Data.

TB results were also higher that 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 PNNL. 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 $\mu$Ci/g for core 41 samples. Total cesium plus strontium concentration calculated from the total beta reported value exceeded the postulated limit of 100 $\mu$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.

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

| Isotope | Units | Concentration range |
|---|---|---|
| $^{234}U$ | μg/g | 0.102 - 0.154 |
| $^{235}U$ | μg/g | 13.476 - 14.006 |
| $^{236}U$ | μg/g | 0.108 - 2.06 |
| $^{238}U$ | μg/g | 1986.07 - 1986.3 |
| $^{238}Pu$ | μCi/g | 2.059E-6 - 2.46E-6 |
| $^{239}Pu$ | μCi/g | 0.055 - 0.56 |
| $^{240}Pu$ | μCi/g | 0.0016 - 0.0017 |
| $^{241}Pu$ | μCi/g | 1.104E-5 - 7.442E-5 |
| $^{242}Pu$ | 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 |
|---|---|---|
| $^{238}U$ | 5.64E-5 | 7.467E-4 |
| $^{239/240}Pu$ | 1.027E-1 | 5.72E-2 |
| $^{241}Am$ | NR | 1.30E-2 |
| $^{137}Cs$ | 11.11 | 17.4 |
| $^{90}Sr$ | 14.2 | 9.87 |

*Not compensated for radioactive decay.

## 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 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.

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

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*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 (see Table A-8).

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

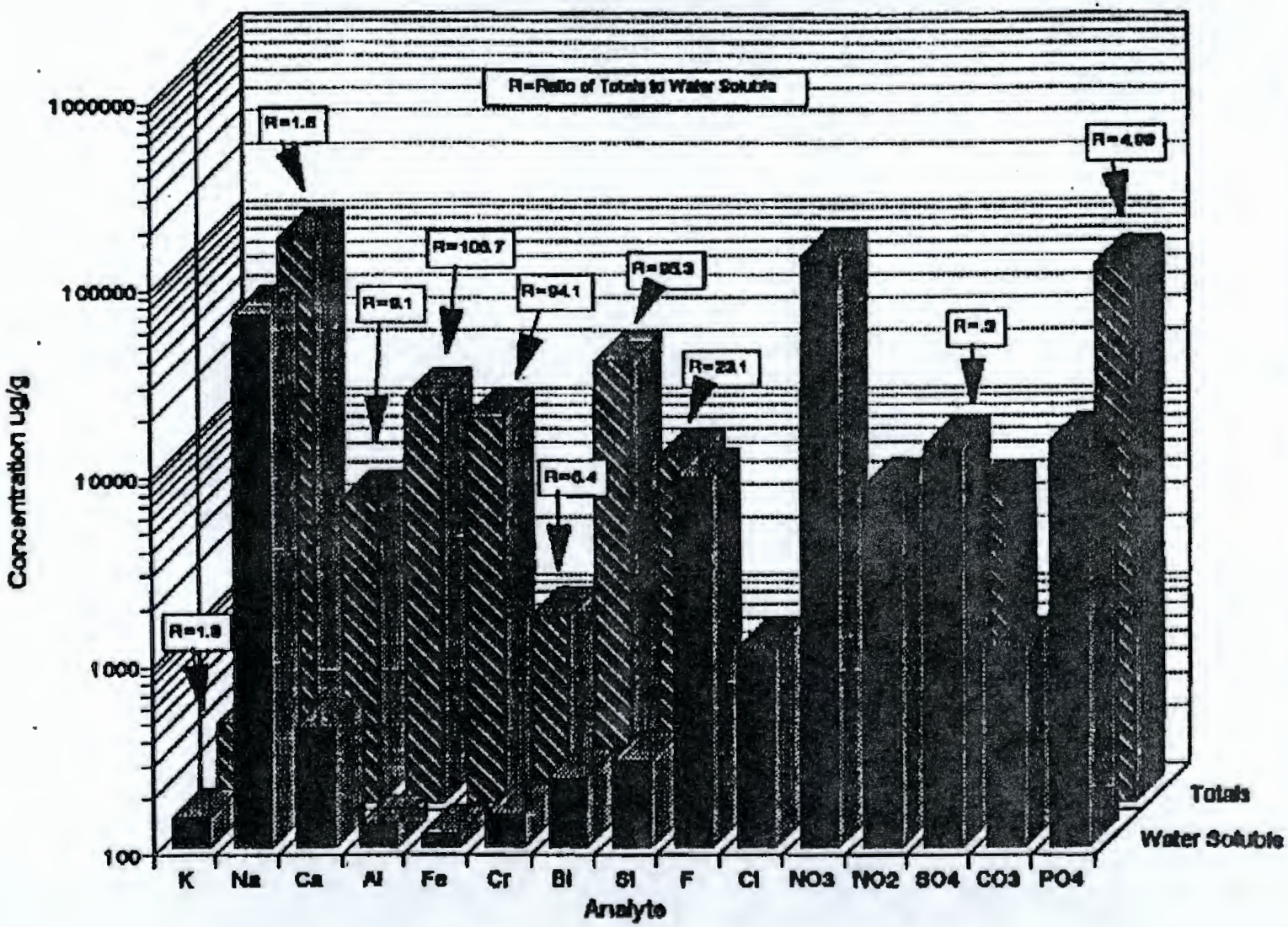



Figure 6-3. Water Soluble and Totals Comparison for 1992 Inorganic Data.

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 |

### 6.4.2 Comparison with 1979 Core Data

Core data from 1979 for metallic elements and electrolytes was appropriately converted to $\mu g/g$. 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 6-13. Comparison of 1979 and 1992 Data for Metallic Elements and Anions in $\mu g/g$.

| 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 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).

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 |

### 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.

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.

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, $H_2SiF_6.XH_2O$, aluminufluorosilcates, fluorophosphates |
| $FeSO_4.7H_2O$ | 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 |
| $Na_2SO_4$ | 0.19797 | Other sodium salts and hydroxides |
| $Na_2CO_3$ | 0.02324 | Other sodium salts and hydroxides |
| $Na_3PO_4$ | 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 |
| $FeO.\ x\ H_2O$ | 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 |
| $Na_3PO_4.12H_2O$ | 0.46944 | Other sodium salts, hydroxides |
| $NaNO_3$ | 5.91665 | Other sodium salts and hydroxides, $NaNO_2$ |
| $ySiO_2.xH_2O$ | 0.34884 | Silica, aluminofluorosilicates, $H_2SiF_6.xH_2O$ |
| $Al(OH)_3$ | 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 (see Table A-9).

## 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

Tables 6-18 and 6-19 summarize TOC, volatile, and semi-volatile organic average results.

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

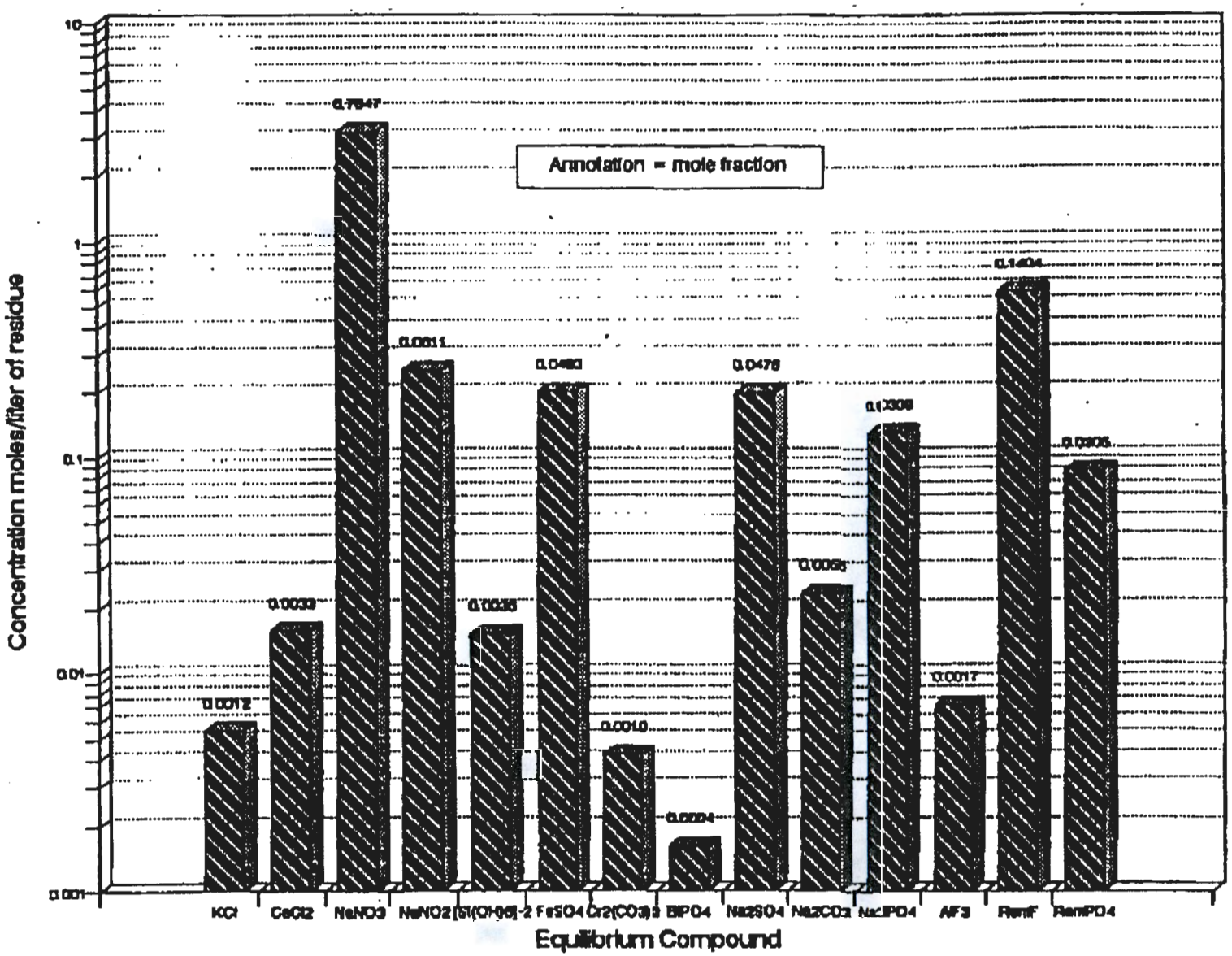



Figure 6-4. Postulated Water Soluble Chemical Profile.

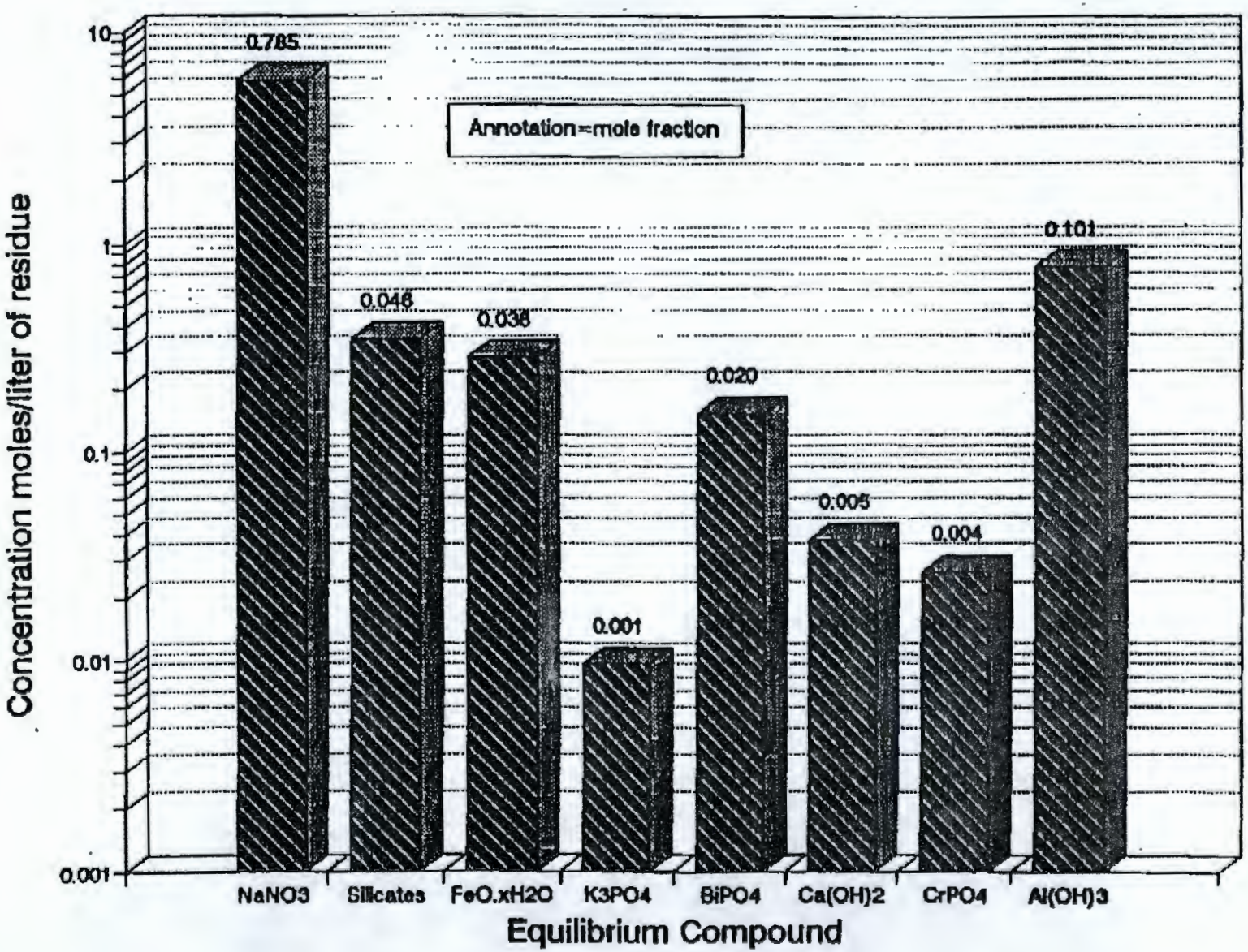



Figure 6-5. Postulated Sludge Equilibrium Profile.

Table 6-17. Average CN and $NH_3$ Results in $\mu g/g$. (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 $\mu g/g$ |

Table 6-19. Average Volatile and Semi-volatile Organics in $\mu g/g$. (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.

## 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).

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.

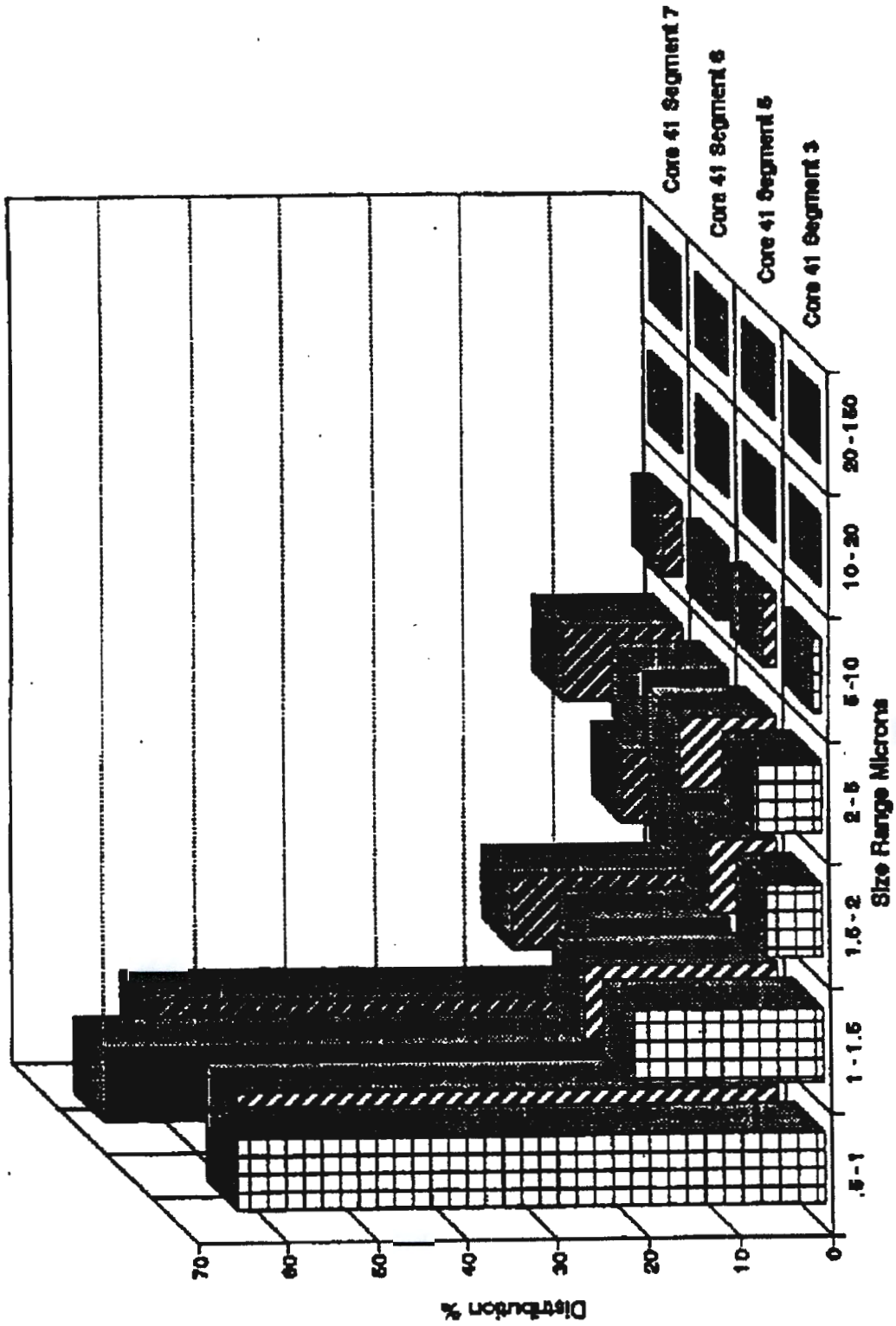
Table 5-4 presented particle size analysis data for cores 40 and 41 segments. The focus will be on number distribution at this point. Figure 6-6 is a plot of this data relative to cores 41. It shows that particle size distributions along riser 7 is consistently homogeneous. The same conclusion is reached 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 (see Tables A-1 through A-5). 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.

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

| 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 |

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



### 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.

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

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

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.

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

| 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.

## 6.8 TWRS PROGRAM ELEMENTS INFORMATION

TWRS elements (i.e., Tank Safety Screening, Waste Tank Operations and Maintenance, the Hanford Waste Vitrification Plant (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.

Table 6-23. Tank Safety Screening Summary.

| Primary decision variable | Primary decision criterion (Babad 1993) | 1992 Core data results |
|---|---|---|
| Energetics | $\leq$ 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 |

### 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 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 $\mu g/g$. 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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## 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 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 Function 1 {Pump Capacity}/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/m$^2$ (100 lb/in$^2$ [psi]).

A strip chart records the hydraulic ram pressure during sampling. Tables A-1 and A-2 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-3, A-4, and A-5 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.).

Figures A-1 and A-2 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-weighed resistant force average was calculated as follows.

$$\text{Time-weighed resistant force} =$$

$$1/\text{Total segment sampling time} * \sum_{1}^{n} \text{time} * \text{resistant force}$$

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.

Table A-1. 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.<br>275 for 55 sec. |

Table A-2. 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<br>997 12 sec<br>1141 30 sec | 852 18 sec<br>1285 37 sec | 852 5 sec<br>1285 30 sec<br>>4027 1 sec<br>3450 10 sec | 564 5 sec<br>1141 55 sec |

Table A-3. 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-4. 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-5. 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 | |

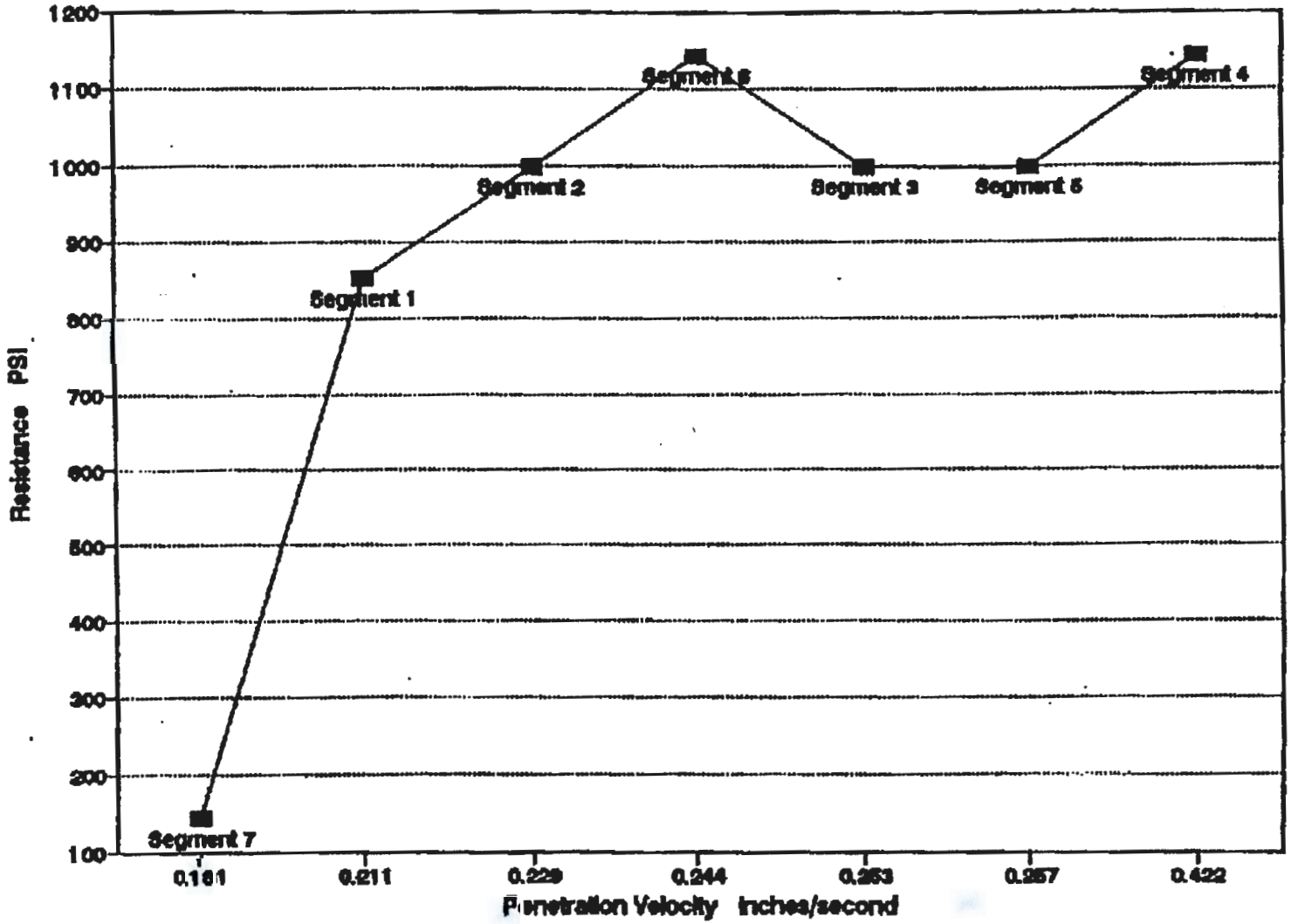



Figure A-1. Time-Weighed Resistant Force Versus Penetration Velocity for Core 40.

Figure A-2. Time-Weighed Resistant Force Versus Penetration Velocity for Core 41.

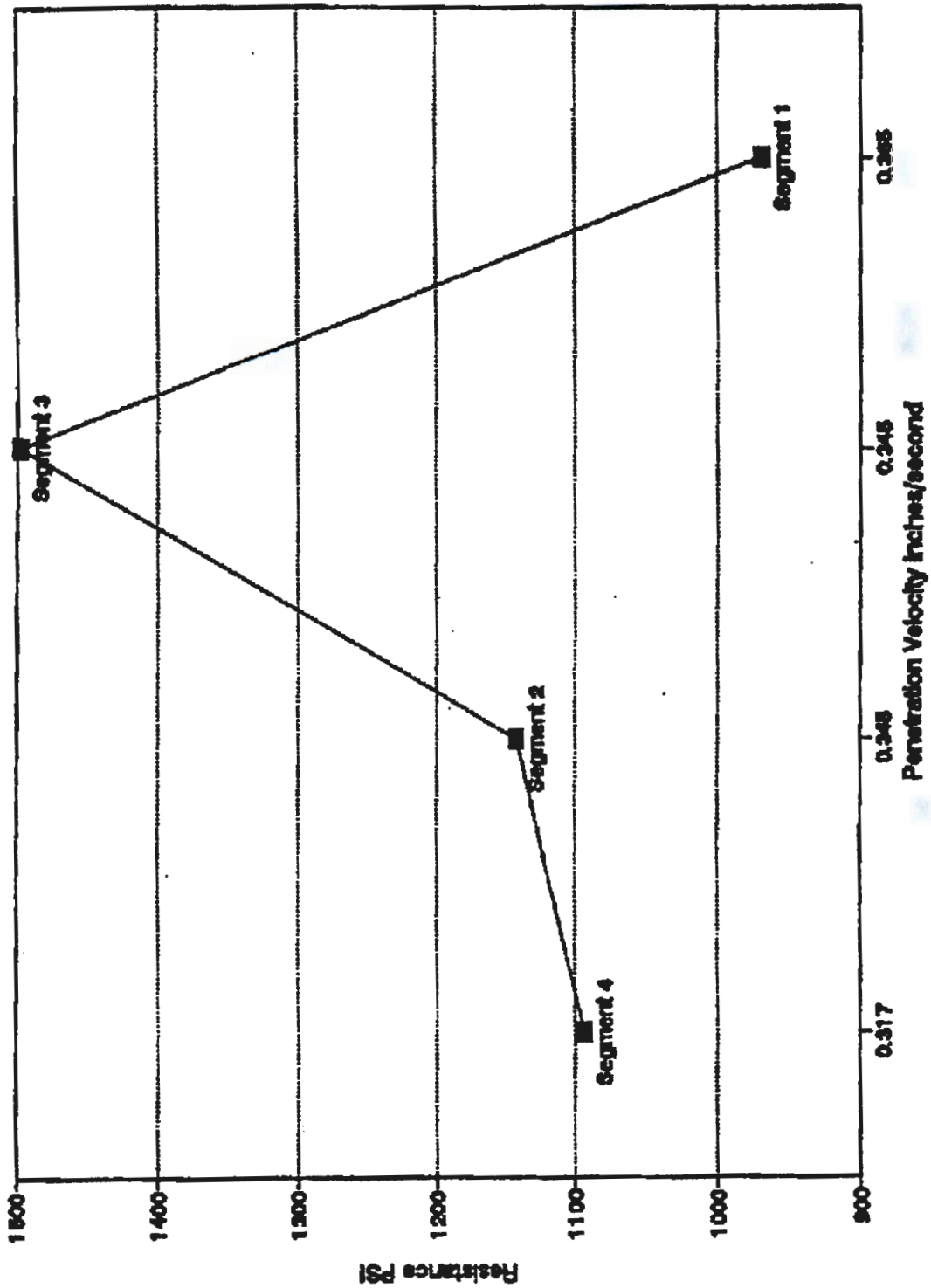
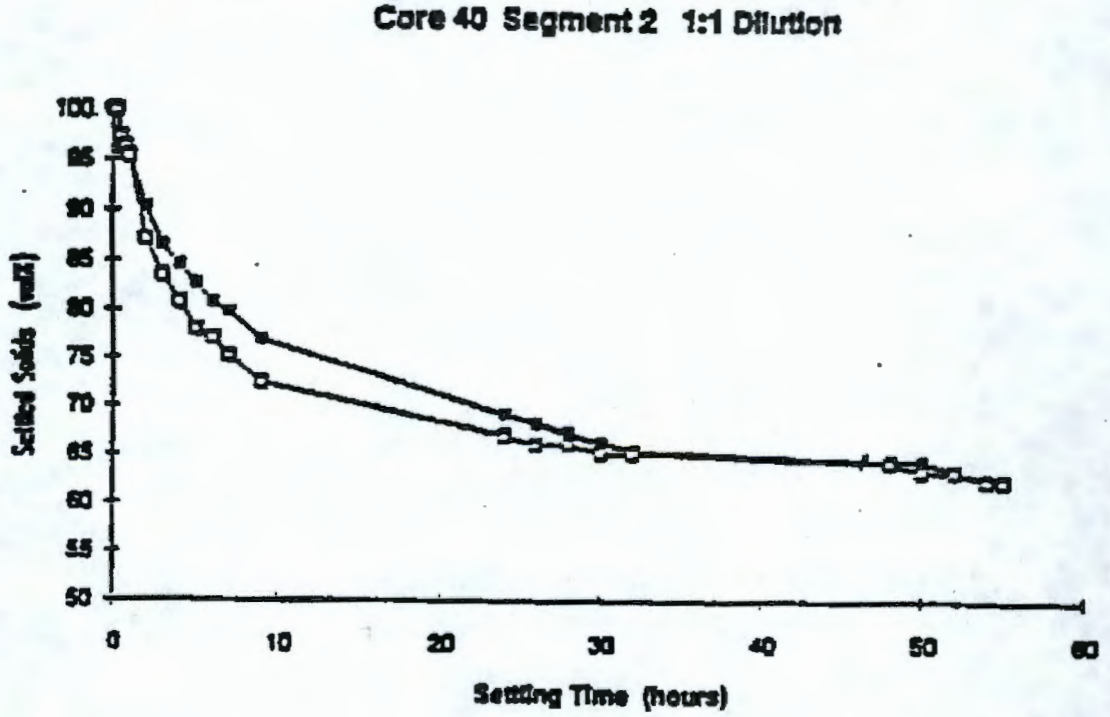


Figure A-3. Settling Rate Curves: Core 40, Segment 2.



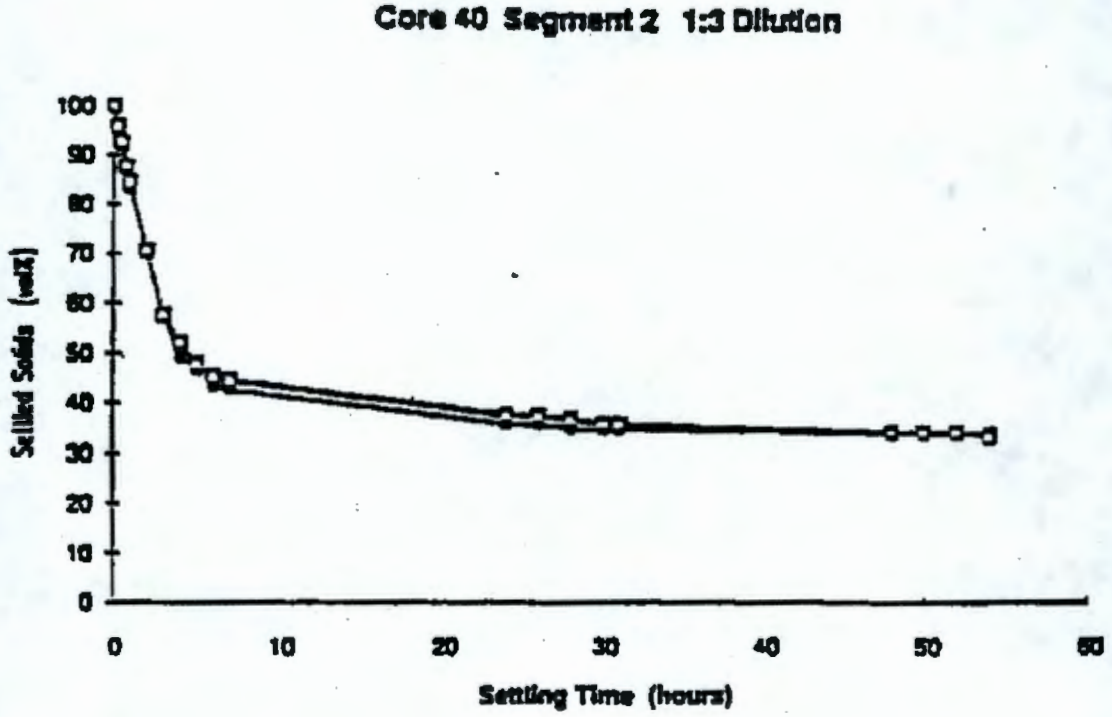
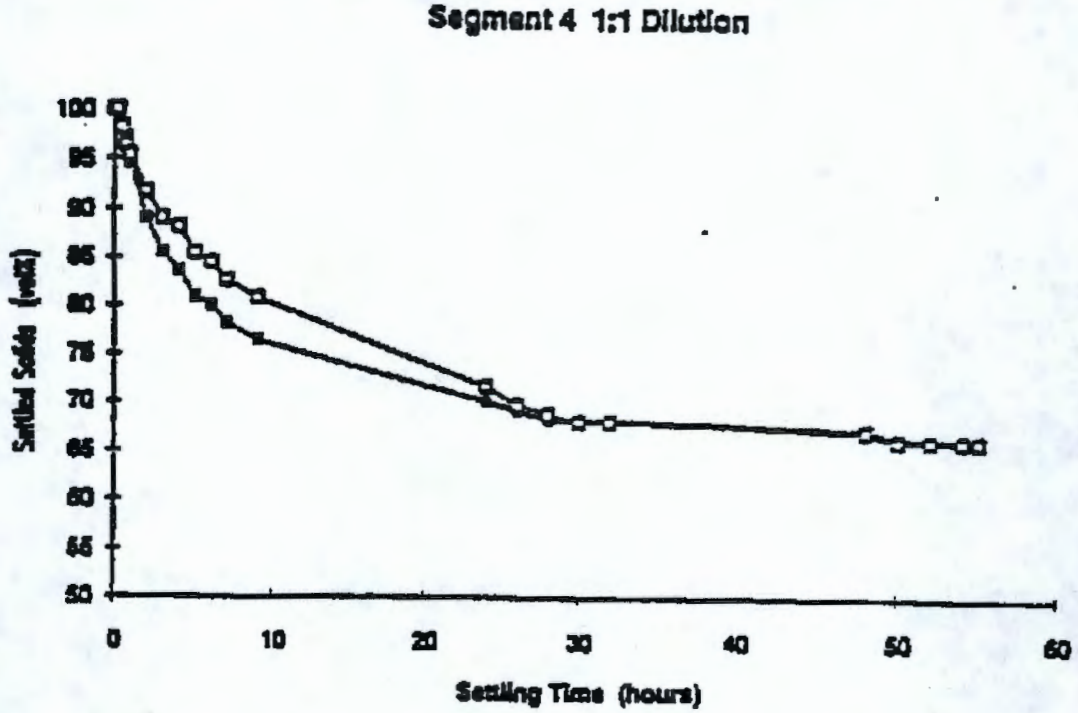



Figure A-4. Settling Rate Curves: Core 40, Segment 4.



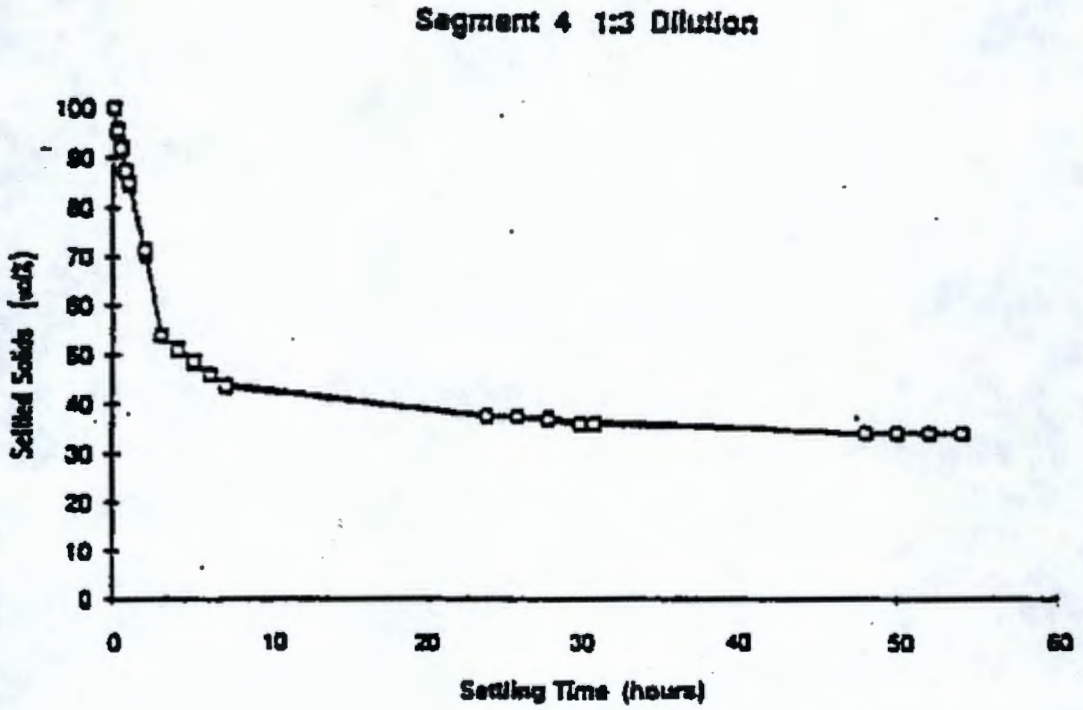
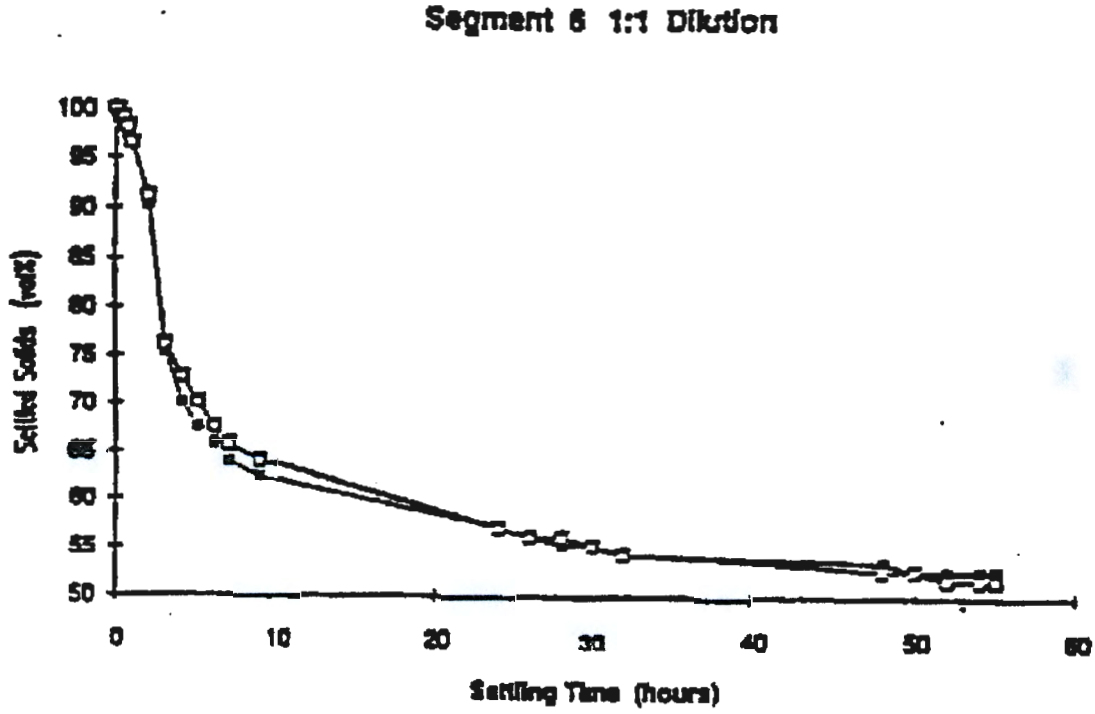



Figure A-5. Settling Rate Curves: Core 40, Segment 6.



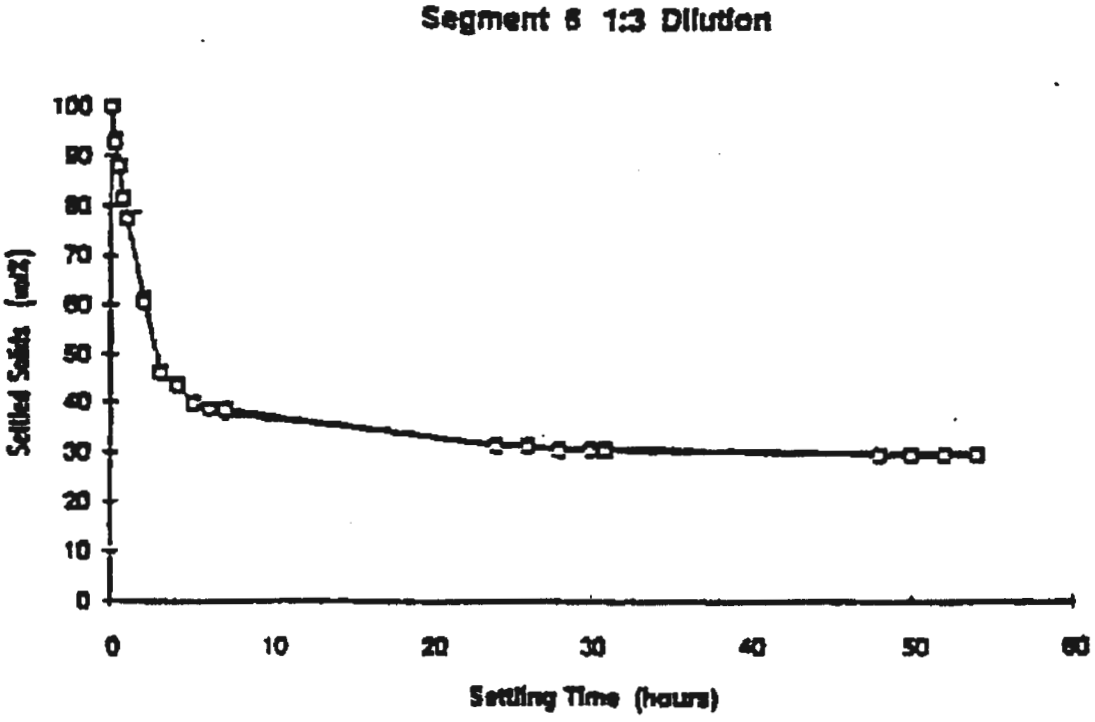



Table A-6. Composite Level Radiochemical Analysis Results.

| Radio-chemical | Core 40 composite 1 | Core 40 composite 2 | Core 41 composite 1 | Core 41 composite 2 |
|---|---|---|---|---|
| $^{238}U$ µg/g | 1.56E3 - 2.07E3 | 1.86E3 - 2.18E3 | 2.82E3 | 2.29E4 - 2.52E4* |
| $^{239/240}Pu$ µCi/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 µCi/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 µCi/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 |
| $^{137}Cs$ µCi/g | 1.34E+1 - 1.35E+1 | 1.09E+1 - 1.16E+1 | 2E+1 - 2.04E+1 | 2.43E+1 - 2.51E+1 |
| $^{90}Sr$ µCi/g | 9.76 - 10.2 | 7.71 - 8.97 | 11.8 - 12 | 8.74 - 9.96 |
| $^{241}Am$ µCi/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 |
| $^{99}Tc$ µCi/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 |
| $^{3}H$ µCi/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 |
| $^{14}C$ µCi/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-7. Cores 40 and 41 (1992) Analytical Data Summary. (4 pages)

| 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 | µg/g | µg/g | µg/g | µg/g | µg/g | µg/g | µg/g | µg/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 | |
| 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 | |
| ICP.w.V | 0.82 | 0.82 | 0.00 | 0.00 | 0.41 | 0.00 | 0.82 | 0.41 | |

Table A-7. Cores 40 and 41 (1992) Analytical Data Summary. (4 pages)

| 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 | µg/g | µg/g | µg/g | µg/g | µg/g | µg/g | µg/g | µg/g | Kgs |
| | | | | | | | | | |
| 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 |
| 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 |
| 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 | |

Table A-7. Cores 40 and 41 (1992) Analytical Data Summary. (4 pages)

| 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 | µg/g | µg/g | µg/g | µg/g | µg/g | µg/g | µg/g | µg/g | Kgs |
| | | | | | | | | | |
| 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.w.Sb | 37.30 | 37.30 | 36.25 | 37.20 | 0.51 | 36.25 | 37.20 | 37.01 | |
| 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 | 5.22 | 10.50 | 8.18 | |
| ICP.w.Ba | 0.38 | 0.53 | 0.31 | 0.55 | 0.14 | 0.31 | 0.67 | 0.44 | |
| 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 | 8.65 | 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 | |
| Spec.f.U238 µg/g | 1815 | 2020 | 2820 | 2405 | 416.53 | 1560 | 2820 | 2265 | 4370 kgs |

Table A-7. Cores 40 and 41 (1992) Analytical Data Summary. (4 pages)

| 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 | | | | | |
| Radionuclides | | | | | | | | | |
| Units | µCi/g | µCi/g | µCi/g | µCi/g | µCi/g | µCi/g | µCi/g | µCi/g | KCi |
| | | | | | | | | | |
| 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 |
| $^{241}$Am | 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 |
| $^{239/240}$Pu | 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 |
| $^{238}$U | 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 |
| $^{99}$Tc | 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 |
| $^{90}$Sr | 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 |
| $^{14}$C | 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 |
| $^{3}$H | 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 |
| 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-8. BX-107 Charge Balance.

| Analyte | Atomic/ Molecular Weight | Prep Type | Oxidation State Used for Calc's | Oxidation Number | meq Wt. (g/meq) | milleqiuvalents Core 40 Comp 1 | milleqiuvalents Core 40 Comp 2 | milleqiuvalents Core 41 Comp 1 | milleqiuvalents 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 | 4.550 | 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 | | | | |
| Ti | 47.9 | ICP Acid | Ti+4 | 4 | 0.01198 | | | | |
| Zr | 91.22 | ICP Acid | Zr | 4 | 0.02281 | | | | |
| Hg | 200.59 | AA | Hg+2 | 2 | 0.10030 | | | | |
| U ug/g | 238.02 | Fluroimetry | 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 | 0.578 |
| Cl | 35.45 | Direct IC | Cl- | -1 | 0.03545 | 0.035 | 0.033 | 0.029 | 0.033 |
| NO3 | 62 | Direct IC | NO3- | -1 | 0.06200 | 2.290 | 2.411 | 2.052 | 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 | Fusion 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-9. BX-107 Material Balance.

| Analyte | Atomic/ Molecular Weight | Oxide Form | Method | No. Main Atoms | No. Oxygen Atoms | 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 | 1.089 | 2.25 | 2.82 | 3.06 | 3.14 |
| Sb | 121.75 | Sb2O5 | ICP Acid | 2 | 5 | 1.329 | | | | |
| 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 | | | | |
| Ca | 40.08 | CaO | ICP Acid | 1 | 1 | 1.399 | 0.30 | | 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 | 1.342 | 1.31 | 1.30 | 1.78 | 1.70 |
| Pb | 207.2 | PbO | ICP Acid | 1 | 1 | 1.077 | | | | |
| Mg | 24.31 | MgO | ICP Acid | 1 | 1 | 1.658 | | | | |
| 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 | 10.30 | 10.00 |
| Zn | 65.37 | ZnO | ICP Acid | 1 | 1 | 1.245 | | | | |
| Bi | 208.98 | Bi2O3 | ICP Acid | 2 | 3 | 1.115 | 2.36 | 2.46 | 5.02 | 2.92 |
| B | 10.81 | B2O3 | ICP Acid | 2 | 3 | 3.220 | | | | |
| Ce | 140.12 | Ce2O3 | ICP Acid | 2 | 3 | 1.171 | | | | |
| La | 138.91 | La2O3 | ICP Acid | 2 | 3 | 1.273 | | | | |
| Si | 28.09 | SiO2 | ICP Fusion | 1 | 2 | 2.139 | 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 | 4.32 | 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 | Acetate | 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 | 56.35 | 55.95 |
| U | 238.029 | UO2 | Fluorimetry | 1 | 2 | 1.134 | 0.21 | 0.23 | 0.32 | 2.73 |
| | | | | | | Total % | 101.02 | 101.33 | 100.34 | 101.01 |

Table A-10. Laboratory Identification Numbers Core Composite Samples. (222-S Laboratory)

| Sample point | STD | BLK | Samples |
|---|---|---|---|
| BX107-C40-C1<br><br>BX107-C40CC1* | J922, J930<br><br>J984, J991 | J923<br><br>J985, J993 | J924, J931, J932, J939, J943, J944, J947, J951,<br>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 A-11. 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 A-12. 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 A-13. Laboratory Identification Numbers for Core 40 and 41 Segments and Composites-PNNL.

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

Table A-14. Analytical Methods for Physical and Rheological Testing.

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

Table A-15. 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<br>Alpha energy analysis | LA-925-106<br>PNL-MA-597 |
| Total alpha<br>Total beta | Proportional counting | LA-508-101 |
| $^{238}Pu$, $^{239,240}Pu$, $^{241}Am$, $^{237}Np$ | Alpha spectrometry | PNL-ALO-423/<br>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 |
| $^{14}C$<br>$^{3}H$ | Liquid scintillation | LA-348-104<br>LA-218-113 |
| $^{137}Cs$ | Gamma energy analysis | LA-548-121 |
| $H^+$ | pH | LA-212-103 |
| As<br>Se | Atomic absorption | PNL-ALO-214<br>PNL-ALO-215 |
| Pu isotopic<br>Ur isotopic | Mass spectrometry | PNL-ALO-455<br>PNL-MA-697 |
| TOC/TIC | Furnace oxidation<br>Persulfate oxidation | LA-622-102<br>PNL-ALO-380/381 |

Table A-16. Analytical Methods For Organic Analyses.

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

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